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Interfacial Contact Resistance for Ti-6Al-4V and SUS 316L Plates as Bipolar Plates in PEMFC

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Abstract: This research mainly presents the performance of Ti-6Al-4V and SUS 316L plates for PEMFC under interfacial contact resistance (ICR). In approaching a lightweight and ease of manufacture bipolar plates, both selected materials were chosen based on their good and relatively low cost compared to graphite. The main aim of this study is to investigate the ICR of the current flowing between two plates in the PEMFC. Metallic bipolar plates tendency to have high contact resistance and corrosion after several times of usage. Therefore, coating is a method in preventing the bipolar plates surface material from damage in the acidic environment of PEMFC. The experiment was carried out on Ti-6Al-4V and SUS 316L plates before coating at different temperature at 40 °C to 80 °C and measured from 0.1 A to 1 A for the ICR values. The ICR results of Ti-6Al-4V after coated Au was 0.01 mΩ cm² at 60 °C while SUS 316L was 0.04 mΩ cm² at 40 °C, respectively. Coating materials using metal nitrides and carbides (CrN, TiN and NbC) are propose to develop different coating materials at anode and cathode bipolar plates using air brushing spray (conventional method) and PVD (high end technology method, to provide high corrosion resistance at cathode as well as increasing electrical conductivity at anode in PEMFC components.

Key words: Interfacial contact resistance (ICR) · PEMFC · Bipolar plates · SUS 316L · Ti-6Al-4V

INTRODUCTION

Fuel cell power system using hydrogen energy has good potential for clean atmosphere and efficient for transportation. This is supported by Davis *et al.* [1] that fuel cell technology economically sustainable in development of hybrid electric transportation as consumer needs. Though the assembly pressure influencing the contact resistance of PEMFC that may lead to leakage of the fuels and create a high contact resistance to the system [2]. Extensive studied by Zhang *et al.* [2] and Choe *et al.* [3] in fuel cell technology has been made to improve the PEMFC.

According to Yuan *et al*. [4] have states that metallic bipolar plates consumed 60% to 80% total components in proton exchange membrane fuel cells (PEMFC) [3-4]. An electrochemical occurred when air and hydrogen gases flow into the channels of the plates as shown in Figure 1. Membrane electrode assembly (MEA) as a barrier which gas are diffused by gas diffusion layer

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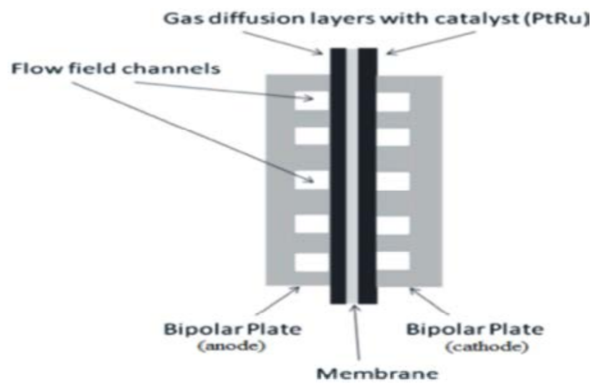

Fig. 1: Schematic view of PEMFC structure between anode and cathode bipolar plates [5]

(GDL) and split into proton and electrons to the current collector. The hydrogen ions will flow to the cathode through the membrane and produce molecule H_2O. Water is removed by the air flow in the fuel cell [5].

It has been reported in the literature review by experimental research on the contact resistance. Reviews such as Tawfik *et al*. [6] discussed in methods and techniques for metallic bipolar plates to strive between graphite composites in maintaining low contact resistance. Wang *et al*. [7] studied the contact resistance between carbon paper and stainless steel due to its low cost, high strength, ease of machining as well as its corrosion resistance. Mishra *et al*. [8] investigated the effects of different gas diffusion layer materials and contact pressures on the contact resistance. Nevertheless, theoretical and prediction models of the contact resistance in PEMFC is feasible and still under improvement.

This paper work focused on the properties of Ti-6Al-4V and SUS 316L as a bipolar plate. Both metallic materials provide a good alternative to graphite bipolar plates. Since the bipolar plate requires fabrication of channels in the plate surfaces, titanium and SUS 316L have high ductility in order ease of manufacturing processes especially for mass production compared to graphite [9-10]. It also readily made materials, as well as not complex material preparation needed as ceramic polymers bipolar plates [11]. Although the titanium plates was expensive than SUS 316L, by used proper surface treatment and design of bipolar plate, titanium and SUS 316L can be used to produce strong bipolar plates with inexpensive process fabrication. A major concern by using metallic bipolar plate is the corrosion process. Therefore, titanium and SUS 316L have high corrosion resistance which the corrosion rates were less than 100 mm/year [12].

To avoid corrosion, titanium and SUS 316L have to be coated with a protective layer. Coating should be conductive and the bipolar plates surface must improve from the formation of micro pores and micro cracks after coatings. In the PEMFC system of bipolar plates, anode cell interact as current collector which the hydrogen gas produced hydrogen ion before went through in the cathode cell. In the PEMFC, oxidation exposed more on cathode cell because of oxygen reduction and high water concentration. Therefore, different coating proposed at anode and cathode to maintain the electrical conductivity as well as high corrosion resistance as the temperature increase in the system.

CrN (chromium nitride) and NbC were suggested as coating on cathode cell. High Cr content greatly improves the corrosion resistance of SUS 316L in simulated PEMFC environment as well as conductive material [13]. As one of a refractory ceramic, Nb could be used to achieve thinner protective layer on surface material, minimizes concentration of process defect after coating, high melting point up to 2204 °C, possess in chemical stability and high resistance to corrosion [14-15]. Transition metal nitrides and carbides possess prominent physical and chemical properties such as superconductivity, high hardness, high melting points, high electrical conductivity and good corrosion resistance. These properties of materials were ideal materials for use as wear resistance and corrosion resistance coatings, field emitters and diffusion barriers [15]. Whereas the C (carbon) was recommend as coating at anode cell. As for the C, it exhibited the best conductivity and better interfacial contact resistance [16]. Therefore, high conductivity and corrosion resistance will attain the bipolar plates endure for long terms operation in PEMFC environment.

In this study, Ti-6Al-4V and SUS 316L were suggested as a new candidate for a metallic bipolar plate due to ease of machining process instead of good mechanical properties.

MATERIALS AND METHOD

The samples of disk shaped with a thickness of 0.7 mm were recommended. A lesser thickness will provide lightweight of material to sustain with reasonable strength for the bipolar plates. Yuan *et al*. [4] had reviewed the design criteria bipolar plate for low weight and volume that can withstand with high temperature in PEMFC operating system.

Fig. 2: A disk shaped sample was prepared for Ti-6Al-4V and SUS 316L with 0.7 mm thickness

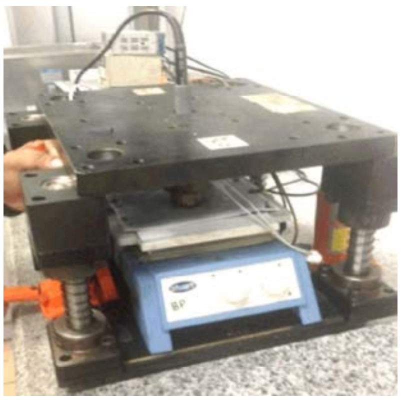

Fig. 3: Interfacial contact resistance (ICR) apparatus

The Ti-6Al-4V and SUS 316L plates were prepared as a substrate. Two samples from each material were cut into diameter of 20 mm. Each sample was prepared without coating before tested. The samples surfaces were ground with 400, 600, 800 and 1200 SiC paper before tested to remove the scratches and oxide layers.

Figure 2 displays the sample of disk shaped prepared for titanium and SUS 316L which are ready to be tested for ICR. The ICR was measured followed by the setup shown in Figure 3. Current (I) was sent through in a complete setup whereas the potential (V) was measured through a circuit in different preheating temperature (40 °C, 60 °C and 80 °C).

This setup was inspired by Wang *et al*. [17] and also has been practically used by Laedre [18] in measured interfacial contact resistance between two bipolar plates. Figure 3 shows the apparatus of interfacial contact resistance based on the ICR setup. Contact resistance is measured using pressure of 150 N/cm^2 from the piston.

The ICR values were measured from potential (V) and current (A) resistance using the Equation 1 to Equation 5 as in Table 1 and Table 2 Ti-6Al-4V and SUS 316L plates. The current 0.1 A to 1 A was employed to identify the ICR behaviour from temperature 40 °C to 80 °C before and after coating with Au. Observation was made for the ICR for the preferred parameter.

Areas and applied current:

$$A_{piston} = \pi r2 \qquad \text{(Eq. 1)}$$

$$A_{sample} = \pi r2 \qquad \text{(Eq. 2)}$$

Force excited on plate:

$$F = P/A \qquad \text{(Eq. 3)}$$

Table 1: The potential (V) and current (A) resistance before and after coating with gold (Au) for Ti-6Al-4V between 40 °C to 80 °

Material		Ti-6Al-4V			
		Uncoated		Coated with Au	
T (°C)	I(A)	V (V)	ICR(mΩ cm²)	V (V)	ICR (mΩ cm²)
40	1	0.2153	0.3382	0.05006	0.0319
60		0.1957	0.3074	0.01812	0.0115
80		0.1633	0.2565	0.03185	0.0203

Table 2: The potential (V) and current (A) resistance before and after coating with gold (Au) for SUS 316L between 40 °C to 80 °

Material		SUS 316L			
		Uncoated		Coated with Au	
T (°C)	I(A)	V (V)	ICR(mΩ cm²)	V (V)	ICR (mΩ cm²)
40	1	0.2948	0.4631	0.06236	0.0397
60		0.2297	0.3608	0.13184	0.0839
80		0.1894	0.2975	0.18516	0.1179

Contact resistance at 15 bar:

$$R = V/I \qquad \text{(Eq. 4)}$$

$$ICR = R\,(A_{sample}) \qquad \text{(Eq. 5)}$$

RESULTS AND DISCUSSION

The ICR with the current ranging from 0.1 A to 1 A for each sample has been observed. The ICR values of Ti-6Al-4V before coating ranged between 0.40 mΩ cm² to 0.26 mΩ cm² while SUS 316L plates ranged between 0.65 mΩ cm² to 0.30 mΩ cm², respectively. Parameter with the current 1 A were chosen as the best graph pattern and lowest ICR amongst others.

Figure 4 presents the ICR of Ti-6Al-4V before and after coating with Au from 40 °C to 80 °C at 1 A. The graph pattern indicates stable performance ICR of uncoated samples towards temperature. The ICR values decreased as the temperature increased. However, the ICR values after coated with Au increased from 0.01 mΩ cm² to 0.02 mΩ cm² due increment temperature at 80 °C.

Meanwhile, Figure 5 shows a result of SUS 316L before and after coating with Au from 40 °C to 80 °C at 1 A. The ICR values of uncoated SUS 316L decreased as the temperature increased. Nonetheless, the performance of ICR values after coated Au was proportional to the increment of temperature. As the temperatures increase the ICR values increased for the SUS 316L coated Au from 0.04 mΩ cm² to 0.12 mΩ cm², respectively.

Overall results, the Ti-6Al-4V obtained the lowest ICR values compared SUS 316L after modification surface plates by Au. The lowest ICR value for Ti-6Al-4V was 0.01 mΩ cm² at 60 °C. While SUS 316L ICR value recorded was 0.04 mΩ cm² at 40 °C. Though, both metallic bipolar plates meet the U.S. Department of Energy (DOE) target for ICR value less than 10 mΩ cm^2.

Both metals exhibit excellent mechanical properties but attributed to the passivation in the presence of oxygen and formed oxide metal film on the surface metal. Karimi *et al*. [11] claimed that it is necessary to remove or reduce oxide films and thickness to an acceptable level in order to increase conductivity as well as decrease the ICR. Therefore, the metallic bipolar plates must be coated to avoid effect the membrane.

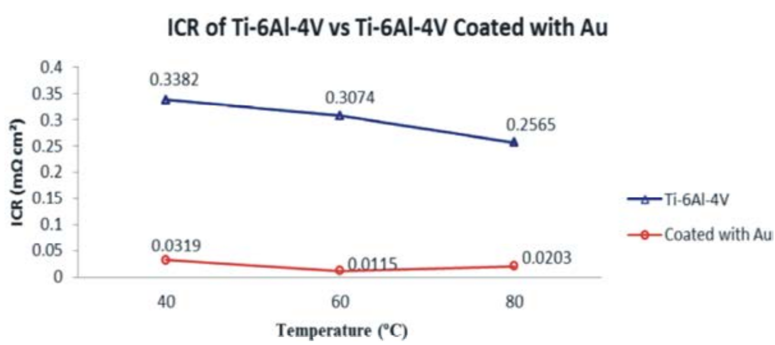

Fig. 4: Interfacial contact resistance (ICR) of Ti-6Al-4V plates before and after coating with Au over temperature (0 °C - 80 °C) at 1 A

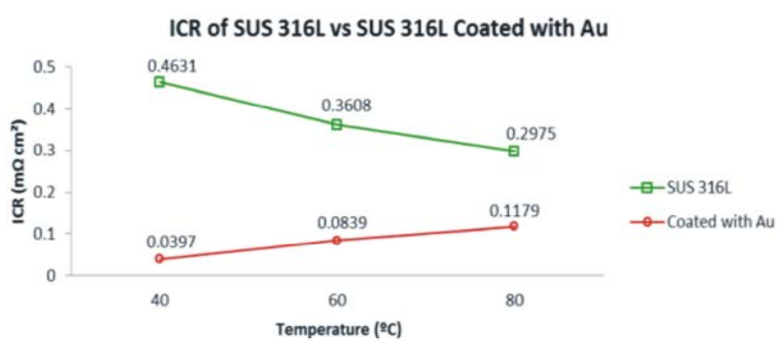

Fig. 5: Interfacial contact resistance (ICR) of SUS 316L plates before and after coating with Au over temperature (0 °C - 80 °C) at 1 A

Table 3: 2020 US Department of Energy (DOE) targets for bipolar plates [19]

Property	Target	Description
Cost	< 3$ kW^{-1}	500 000 units stacks per year
Corrosion resistance (anode)	< 1 μA cm^{-2}	pH 3, 0.1 ppm HF, 80 °C, Ar purge,
Potentiodynamic test Corrosion resistance (cathode)	< 1 μA cm^{-2}	-0.4 V - 0.6 V (Ag/AgCl), 0.1 mV/s, pH 3, 0.1 ppm HF, 80 °C, aerated Potentiostatic test (>24 h) 0.6 V (Ag/AgCl), $i_{passive}$ < 50 nA cm^{-2}

Table 4: Types of coating and method proposed to Ti-6Al-4V and SUS 316L

	Coating	
Method	Anode BP	Cathode BP
Air brushing spray (conventional)	Carbon	CrN TiN NbC
PVD (high end technology)	Carbon	CrN TiN NbC

COATING MATERIALS AND METHODS

The benchmark for commercialization for bipolar plate material properties by 2020 US DOE targets are summarized in Table 3. The DOE requirements subjected to corrosion in the PEMFC environment which the electrochemical test have to immerse more than 24 hours in the solvent. The electrochemical coating follow the benchmark by DOE which act as self-healing for the metal bipolar plates from corrodes to reduce the contact resistance of the bipolar plate [20], increasing the available power from the fuel cell and increase its electrical conductivity [19,21]. Thus, the electrochemical coating can use as a method in order to get positive results towards bipolar plates coating with low cost of low mass produced. By using this method, the results are not constant for each parameter and need long period (at least > 24 hours) to have good surface coating.

Coatings were applied using several methods by conventional or high end technology method for corrosion prevention as in Table 4. Two types of method propose are air brush spray and PVD coating. The coating materials chosen based on the previous study of surface medication for stainless steel and titanium bipolar plates. Modification process more concentrates at the cathode side of bipolar plates because tendency of corrosion occur more in this region because of oxygen continuously flows to the cathode and combined with H+ ion. Therefore, negative electrons from water (H2O) are released to the air at the cathode side [11, 22]

In order to fabricate subsurface layer on the surface of 304 SS bipolar plate, Wang *et al*. suggested niobium carbide (NbC) using plasma surface diffusion alloying. Hence, the NbC coating on 304 SS bipolar plate provided dense diffusion layer thickness of 6-7 μm and remained low current densities (I_{corr}) within the ICR of 8.47 mΩ cm^2 [15]. Overall performance of niobium carbide modified 304 SS bipolar plate developed excellent performance in PEMFCs. Dissimilar method and coating material, Fukutsuka *et al*. investigated SUS 304 stainless steel bipolar plate by using plasma assisted chemical vapour deposition (CVD), which used carbon as coated material to produce high corrosion resistance application in PEMFCs [23]. The author reported high electrical conductivity and strength material produced after deposited. The electrochemical analyses were tested for anodic and cathodic under PEMFCs operating conditions. The result presents the ICR values of 8.9 mΩ cm2 was still in the DOE ranged with less 1 μA cm-² current density.

Liu *et al*. [24] experimental on PVD coated 316L stainless steel in order to determine the corrosion behaviour in 0.5 M NaCl aqueous solution using EIS. From the SEM micrographs, CrN coatings expected to be high corrosion resistance by limiting the oxygen diffusion to the coating or steel substrate interface that produce excellent corrosion resistance due to dense equiaxed crystallites. In contrast, TiN coatings susceptible to have a high density of defects during growth and provided the straight boundaries for the diffusion of oxygen due to the fine columnar crystallites. The result was under investigation meanwhile to meet the DOE target.

Omrani *et al*. [10] examined the influence of titanium nitride (TiN) nanoparticle implantation on 316L SS bipolar plates in terms of its corrosion and electrical conductivity. The resulting TiN implantation was deposited on polished 316L SS plates using a plasma focus technique filled with nitrogen gas within 10, 20 and 30 shots. The I_{corr} of the coated 316L SS plate (1.0 μA cm-2) was lower than the bare 316L SS plate (8.3 μA cm-2) in a simulated environment. Although increasing 30 shots of TiN focus implantation, the corrosion rate per year decrease to 0.0146 μm per year. It has been reviewed for the metal nitrides (chromium nitride, titanium aluminum nitride or titanium nitride) reveal good electrochemical stability with acceptable electrical conductivity [25]. Titanium nitride has been extensively used for good corrosion resistance and low interfacial contact resistance [10,17,25]. In this studied, the ICR value of the coated 316L SS plate of 5 Ω cm^2 was reported achieving the DOE target.

The coating method chosen are air brushing spray [26] and PVD [27-28]. The device of spraying method is similar to the development of the spray gun. The coating material may be in the form of powder, ceramic rod, wire or molten materials. Husby [26] had using air brushing techniques and the I_{corr} values ranged from 0.11 µA cm-² to 0.54 cm-² for stainless steel bipolar plates. The result record the best I_{corr} after coating which meet the DOE target of < 1 µA cm-². The air brushing method is feasible as coating method to have self-clean capability, promising method for low cost, high volume and large area production and maintaining sufficient transmittance [29-30] for clear enough for bipolar plates in PEMFC applications. This conventional air brush method has the potential for fabricating multilayer coating onto substrates.

As for physical vapor deposition (PVD) uses physical process as heating or sputtering to produce a vapor of material in depositing a layer on metal substrate. PVD coatings are sometimes harder [28] and more corrosion resistant than coatings applied by the electroplating process In comparison, the I_{corr} by using PVD coating is 0.00029 µA cm^{-2}, which is more corrosion resistant [27] than electroplating process, 1.9 µA cm-2. It has been observed that PVD process is more environmental friendly than conventional process as electroplating method. Most coatings have high temperature and good impact strength, excellent abrasion resistance and are so durable that protective topcoats are almost never necessary [28,31].

CONCLUSION

In the current study, the results of ICR indicated that the alternative material of and SUS 316L plates meet the requirement of DOE (U. S. Department of Energy) that to be achieved lower than 10 mΩ cm^2. To sustain the electrical conductivity and provide high corrosion resistance, as well as in high temperature in acidic environment, SUS 316L and Ti-6Al-4V promising low ICR value as bipolar plate material, as well as high electrical conductivity. These studies are in progress in order to develop different coating materials at anode and cathode bipolar plates using air brushing spray (conventional method) and PVD (high end technology method), using metal nitrides and carbides (CrN, TiN and NbC) at Fuel Cell Institute, National University of Malaysia.

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