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Effects of the Synthesis Coating Parameters for Metal Bipolar Plates

(Kesan Parameter Salutan Sintesis untuk Plat Dwikutub Logam)

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ABSTRACT

Metallic bipolar plates tendency to have high contact resistance, but also very susceptible to corrosion. This may decrease in the performance of fuel cells after several times of usage in fuel cell applications. Research has shown that after a metal plate was coated, the characteristic of materials dependent on the type, composition of the coating materials and the method. This study determines design of coating parameters including gas flow rate, DC power, and deposition time of coating for metal bipolar plates, which can be an indicator of the suitability of these plates for use as bipolar plates in proton exchange membrane fuel cell (PEMFC) applications. The aim of this research was to obtain a limitation range value of parameters that can be used as a standard for the use of metal plates as bipolar plates. The optimization parameters designed by Taguchi are used to determine the characteristics of interfacial contact resistance (ICR) and corrosion current density (I_{corr}). The integration of the Taguchi method with simulation can show the optimal design parameters of the coating on the various materials use. The optimization feature based on Taguchi is applied to ICR and I_{corr} values, to determine the feasibility of metal plates as potential bipolar plates in PEMFC.

Keywords: Coating bipolar plate; Cr–C; metallic bipolar plates; Nb–C; Taguchi

ABSTRAK

Kecenderungan plat dwikutub logam mempunyai rintangan sentuh dan kadar kakisan tinggi, namun mudah terdedah kepada kakisan. Penyelidikan ini telah menunjukkan bahawa setelah plat logam disalut, sifat elektrik dipengaruhi oleh jenis dan komposisi salutan yang digunakan, dan kaedah rawatan salutan yang dijalankan. Kajian ini menentukan parameter salutan merangkumi kadar aliran gas, kuasa DC dan masa pemendapan lapisan untuk plat dwikutub logam, untuk mencapai kesesuaian plat digunakan sebagai plat dwikutub dalam aplikasi PEMFC. Pengoptimuman melalui Taguchi dapat digunakan sebagai penentuan pencirian rintangan sentuh antara muka (ICR) dan ketumpatan arus kakisan (I_{corr}). Fokus kajian terhadap kesinambungan kaedah Taguchi dan simulasi menunjukkan reka bentuk parameter optimum salutan Nb–C dan Cr–C. Penyatuan kaedah Taguchi dengan simulasi rintangan kakisan dan ICR dapat menunjukkan parameter reka bentuk salutan yang optimum pada pelbagai bahan yang digunakan. Ciri pengoptimuman berdasarkan Taguchi diterapkan pada nilai ICR dan I_{corr}, untuk menentukan kemungkinan plat logam sebagai plat dwikutub berpotensi dalam PEMFC.

Kata kunci: Cr–C; Nb–C; plat dwikutub logam; plat dwikutub salutan; Taguchi

Introduction

Metallic bipolar plates tendency to have high contact resistance and corrosion rate after several times of usage in proton exchange membrane fuel cell (PEMFC) conditions, that affected performance of a cell in PEMFC stack. High electrical conductivity, good corrosion resistance, thin and lightweight are bipolar plates characteristics in PEMFC (Baroutaji et al. 2015). Few years of difficulty in making bipolar plates by many researcher using graphite materials, that was brittle especially in manufactured graphite flow field channels (Hartnig et al. 2011). To keep alligned with current fuel cell technologies in terms of cost as well as high efficiency, metallic bipolar plates were introduced to improve PEMFC performance. Metal such as carbon steel, titanium, and stainless steel were an alternative material for metallic bipolar plates that can survive within the PEMFC (Mani & Rajendran 2017). However, the corrosion resistance of metal bipolar plates will be reduced for a certain period which leads to decrease in the performance of bipolar plates (Li et al. 2018). Therefore, surface treatment was performed for related problems (Madadi et al. 2019).

The bipolar plate manufacturing needs to achieve the standard set by U.S. Department of Energy (DOE) (Taherian 2014). For metal bipolar plates, the important target value is to achieve electrical conductivity up to 100 S/cm and corrosion current density is <1 μA/cm². Extensive studies of researchers on the application of bipolar plates are also conducted to obtain good conductivity and mechanical properties aligned with U.S. DOE targets. Huang et al. (2016) studied Cr–C is the ideal coating material on 316 L stainless steel because it gives corrosion current density up to 0.04 μA cm^{-2} with low ICR value of 4.27 mΩ cm^2. In addition, good chemical stability of protective layer on stainless steel bipolar plates for up to 30 days in potentiostatic polarization gives low corrosion density current up to 0.13 μA cm^{-2}, by using niobium (Nb) deposited of TiO_2 through sol–gel techniques (Wang et al. 2018). Therefore, stainless steel can reduce the machining process to form into thin sheets, corrosion resistance and high material strength, as well as low price to be considered a good material candidate for metallic bipolar plate in PEMFC (Wang et al. 2003). Hence, these types of metal are superior with the high content of weight percentages of Cr and Ni, hence increase electrical conductivity as well in the PEMFC (Arwati et al. 2019). Herewith, the study showed that metal plated promise a choice with a variety of power into becoming bipolar plate in PEMFC applications (Yang et al. 2017).

The study also used Taguchi method for optimization of coating parameter using PVD to obtain suitable coating formation parameters on SUS 316 L substrate. In between, screening of coating parameters produced on SUS 316 L plates will go through a series of tests, to ensure the substrate after coatings produced meet U.S. DOE targets towards electrical characteristics. The electrical characteristics tests included interfacial contact resistance (ICR) that were performed to determine the effect of the optimization coating parameters after surface treatment. The best parameter was optimized through the tests performed that were analyzed by using the lowest S/N ratio. ANOVA is performed to investigate the influence of gas flow rate, DC power and deposition time of coatings. The focus of this research is to achieve low interfacial contact resistance (ICR) so that high electrical conductivity in the PEMFC system, thereby increasing the corrosion resistance in the cathode cell. This will encourage the performance of the PEMFC stack is stable for both electrical conductivity and corrosion resistance based on U.S. DOE throughout the PEMFC operation cell.

Experimental and Materials

This study using SUS 316 L plates, in validation with the selected parameters (gas flow rate of 8 to 10 sccm, DC power of 80 to 100 W and deposition time of 60 to 80 min) by applied coatings of Nb–C and Cr–C on the surface plate. The materials used was SUS 316 L with the chemical composition; Cr: 16%; Ni: 10%; Mo: 2%; C: < 0.03%; Mn: 1%; Si: < 0.75%; P: < 0.045%; S: < 0.03%; and Fe: Balance (in mass %). It was chosen as metal plates preparation for the bipolar plates because it has been widely used in the commercial industry of PEMFC. SUS 316 L were cut into disk shaped (coupon) with a thickness of 3 mm were recommended. A lesser thickness will provide lightweight of material to sustain with reasonable strength for the bipolar plates (Yuan et al. 2005).

TF450 PVD magnetron sputtering system (SG Co. Ltd, Singapore) was equipped with commercially available 4 inches diameter by 3 mm thickness coating targets. The substrates were ultrasonically cleaned in acetone for 30 min to remove any contaminants from the surface metals, dried at room temperature before placing on the substrate holder in the PVD chamber. Inside the chamber, the substrate holder was placed onto a rotating stage to 20 rpm that was connected to a resistive heater up to 180 °C. The PVD chamber is vacuum to 4.6×10^{-6} Torr and pre-sputtering for 12 h using argon mixed with nitrogen before deposition process takes place. The heater onto the substrate begin during deposition and the working pressure is maintained to 1.4×10^{-2} Torr.

Experimental Design and Data Analysis

The coating optimization parameters was conducted using Taguchi method for robust design without high amount of experimentation to be analysed (Bushroa et al. 2011). The control factors in experiment are gas flowrate (sccm), DC power (W) and deposition time (t) as shown in Table 1, whereas each having two levels, high and low based on the previous experimentation and literature review (Wang et al. 2014; Zhao et al. 2016). In this work, an orthogonal array of L_8 was utilized to study the effect of control factors (gas flow rate, DC power and deposition time) on the response to ICR and corrosion rate value in optimize the experimental error (S/N ratio) by the experimental design setting. Table 2 presents the observations table of L_8 array obtained from parameters of each coating (Nb-C and Cr-C). The experiment conducted considers three factors (gas flow rate, DC power and deposition time) that will be varied to get the optimum parameters before starting the coating process. From the L_8 orthogonal array, eight different designs of coating parameters are then simulated and go through for actual experimental by PVD coating. Each of the designed parameters will go through for the interfacial contact resistance test after coating.

Researcher had claimed combination of statistical and engineering methods by applied Taguchi method was to determine the level of efficiency, validate experiments and help in reducing the cost of experiments conducted (Radzuan et al. 2016). In the meantime, calculating the signal to noise (S/N) ratio and a statistical analysis of variance (ANOVA) were carried out. Each parameter of control factors will be studied and determined as in prediction and actual experiment in validating the significant factors involved. In addition, contribution percentage of each parameter were studied via ANOVA accordance with interfacial contact resistance and corrosion resistance values. Variance analysis includes degree of freedom (DF), variance (V), ratio (F), sum of square, pure sum and P–value (Oktiawati et al. 2017). In this work, the higher efficiency of interfacial contact resistance (ICR) represents better performance of SUS 316 L plates. Therefore, ICR studied with the concept of 'the smaller–the–better' was considered as the good quality characteristics SUS 316 L plates. The S/N ratio for the smaller–the–better is as shown in (1);

$$S/N = -10 \log \left| \frac{1}{n} \dots \sum_{i=1}^{n} y_i^{\,2} \right| \quad (1)$$

By considering (1), the S/N ratio values in this work are calculated, where n is the number of measurements in an experiment, whereas in this case, n=1 and y is the measured efficiency value in an experiment.

TABLE 1. Factors sampling range

Factors	Level 1	Level 2
Gas flow rate (sccm)	8	10
DC power (W)	80	100
Deposition time (t)	60	80

TABLE 2. Observations of Taguchi's model L_8 orthogonal array of Nb-C and Cr-C

Exp. No.	A	B	C
	Gas flow rate (sccm)	DC power (W)	Deposition time (t)
1	1	1	1
2	1	1	2
3	1	2	1
4	1	2	2
5	2	1	1
6	2	1	2
7	2	2	1
8	2	2	2

MEASUREMENT OF CORROSION RESISTANCE AND INTERFACIAL CONTACT RESISTANCE

The corrosion resistance (CR) of SUS 316 L was analyzed using Tafel extrapolation by potentiostat PGZ100 (Voltalab 10). The procedure was controlled using Nova 1.10 electrochemical software. A flat cell of SUS 316 L was used as a coupon substrate at room temperature in a solution of 0.5 M sulphuric acid (H_2SO_4). The SUS 316 L substrate was used as the working electrode, the electrode of Ag/AgCl was used as reference electrode, and platinum electrode acted as the counter electrode. All potentials were referred to the SCE during the experiment. The substrate was stabilized at open circuit (OCP) for 1 h and the data scan from the Tafel plotted were obtained under potential scan rate of 1 mV/s. The corrosion current density (Icorr) aimed less than 1 µA cm^{-2}.

The surface ICR of SUS 316 L plate after coating was measured by placing the plate between the carbon paper (as GDL) under the real simulation condition and by measuring the voltage drop as shown in (2), whereas R is the electrical contact resistance, V is the voltage drop through the setup, I is the applied current and A is the surface area.

$$(R=VA/I) \quad (2)$$

$$(R1 + R2 /2) \quad (3)$$

The ICR was equal to the total resistance in the test assembly as in (3), whereas R1 refers to the resistance of two copper plates, and R2 referred to the resistance of GDL with coated sample (R1 + R2/2). Requirements and testing method were applied based on through plane studied on ICR measurement (Jendras et al. 2017). It was performed using a customized through-plane electric flow device with 1 A of current supplied, at a pressure of 140 N cm^{-2}, at operating temperature of 80 °C.

RESULTS AND DISCUSSION

The orthogonal array of L_8 obtained from the Taguchi method for two types of control factors, in order to study parameters of Nb–C and Cr–C coatings in each run of experimental. As in Table 3, the lowest ICR value (1.1259 mΩ. cm^2) and corrosion rate (137.71 × 10^{-12} A) by Nb–C coating were recorded on samples 7 and 4, while sample 1 represents as the lowest ICR values (1.5595 mΩ. cm^2) and corrosion rates (70.1740 × 10^{-12} A) as for Cr–C coating. Hence, Table 4 only shows ICR values converted to S/N ratio as the minimum response to the control factors. The smallest S/N ratios for ICR of Nb–C (-4.86275 dB) and Cr–C (-7.9647 dB) coating were found in samples 2 and 3. Based on the SN ratio response, overall measures values for ICR gives minimize disturbances (<10 mΩ. cm^2) with low S/N ratio as the purpose of experimental design is to have less interference between parameters response (gas flow rate, DC power, and deposition time) (Utami et al. 2016).

TABLE 3. Measured values for ICR and corrosion rates of Cr–C and N –C in L8 array

Control Factors		Nb-C		Cr-C	
Exp. No.	Level	ICR (mΩ. cm^2)	CR (A/cm^2)	ICR (mΩ. cm^2)	CR (A/cm^2)
1	A1 B1 C1	1.5478	137.71 × 10^{-12}	1.5595	70.1740 × 10^{-12}
2	A1 B1 C2	1.7614	2.757 × 10^{-9}	2.0981	7.575 × 10^{-9}
3	A1 B2 C1	1.3766	1.982 × 10^{-9}	2.5017	2.513 × 10^{-9}
4	A1 B2 C2	1.2223	107.550 × 10^{-12}	2.2795	78.2870 × 10^{-12}
5	A2 B1 C1	1.6211	1.281 × 10^{-9}	1.7805	3.091 × 10^{-9}
6	A2 B1 C2	1.5248	1.736 × 10^{-9}	1.9591	86.431 × 10^{-12}
7	A2 B2 C1	1.1259	1.694 × 10^{-9}	1.8900	1.149 × 10^{-9}
8	A2 B2 C2	1.2559	1.597 × 10^{-9}	1.8805	3.928 × 10^{-9}

TABLE 4. Orthogonal array of Taguchi L_8 indicates the value of ICR

Factor		ICR of Nb–C	ICR of Cr–C
Exp. No.	Parameter	S/N (dB)	
1	A1 B1 C1	-4.3037	-4.6943
2	A1 B1 C2	-4.8627	-7.1499
3	A1 B2 C1	-2.5076	-7.9647
4	A1 B2 C2	-1.9117	-7.6967
5	A2 B1 C1	-3.9604	-5.9892
6	A2 B1 C2	-4.0818	-6.5952
7	A2 B2 C1	-1.0638	-5.4368
8	A2 B2 C2	-1.4398	-6.4058

The interaction of Nb–C between parameter of A, B, and C were analyzed with ANOVA, as in Table 5. The CR value does not affect the layer parameters used, because the contribution is less than 50%. Therefore, only the ICR results were gone through the ANOVA, thus to determine the contribution percentages by each parameter used. In Table 5, B factor (DC power) contributes 89.54% and recorded as highest contribution towards ICR. However, A factor (gas flow rate) contributes 69.01% recorded by Cr–C as shown in Table 6. Toward a better understanding, DC power absorption along with temperature helps to react quickly in line with molecular enlargement and the pressure in the PVD chamber gives slight collision between molecule and gaseous (Yeldose et al. 2008). In addition, influenced of the parameter contribution on coating parameters to the ICR value are high, accordance with the optimal results obtained through the Taguchi method that has been carried out.

The ANOVA results also shows the relations impact of all factors by comparing the mean square with an approximation of the experimental errors at specific levels. The influence of P–value have a significant influence on the represents parameters. These impacts will statistically use to determine the implication of a factor. The same method used by many researchers in studies varied parameters with residual errors at specific levels (Oktiawati et al. 2017; Radzuan et al. 2016). The studied showed that the P–value obtained must be in between 0.05 to confirm each optimization parameter carried out were significant (Ibrahim et al. 2010). From Tables 5 and 6, it observed that factors A and B influenced the most to Nb–C coatings, while Cr–C coatings only influenced by factor A. It has been explained that Nb (niobium) element have low resistance material which can absorb the DC power along with temperature in molecular expansion, while C (carbon) atom provides rapid diffusion between substrate and Nb (Cai et al. 2016). The study also examined the mixture of nitrogen and argon gas in the PVD chamber will induces forming a coating and store more electrical energy as Cr (chromium) element known as high electrical conductivity (Yeldose et al. 2008).

TABLE 5. Orthogonal array of Taguchi L_8 indicates the ICR value of Nb–C

Factor	ICR value of Nb–C					
	DF	S	V	F	P	Contribution (%)
A	1	0.029330	0.029330	10.98	0.03	7.46
B	1	0.351877	0.351877	131.76	0.00	89.54
C	1	0.001076	0.001076	0.40	0.56	0.27
Error	4	0.010682	0.002671			2.72
Total	7	0.392966				100

TABLE 6. Orthogonal array of Taguchi L_8 indicates the ICR value of Cr–C

Factor	ICR value of Cr–C					
	DF	S	V	F	P	Contribution (%)
A	1	0.349281	0.349281	16.67	0.02	69.01
B	1	0.072962	0.072962	3.48	0.14	14.42
C	1	0.000012	0.000012	0.00	0.98	0.002
Error	4	0.083813	0.020953			16.56
Total	7	0.506067				100

Figure 1 shows the main effects plot of S/N ratio with optimization parameter based on the lowest peak for each parameter A, B and C. The S/N ratios in Table 3 were used to develop the main effects plot. Based on Figure 1(a) and 1(b), factor A and B are the most influenced parameters by the ICR in optimizing the coating parameters of Nb–C and Cr–C. it has been seen in Figure 1(a), factor A (DC power) is the most influence parameter in optimizing the ICR values of the Nb–C coating. It can be seen the graph pattern at 80 W of DC power and 8 sccm of gas flow rate aligned parallel to the DC power to form coating onto 316 L substrate. Although the Nb (niobium) has poor electrical conductivity but it absorbs DC power along high temperature to react quickly in molecular expansion, while C (carbon) atoms give rapid infiltration of the substrate between the atoms Nb (Cai et al. 2016). In contrast, Cr–C coating parameters further influenced by factor B (gas flow rate). Indeed, Cr (chromium) element has high electrical conductivity, but the mixture of nitrogen and argon gas induced to form the coating aligned in parallel with the deposition time as shown in Figure 1(b).

Nowadays, high corrosion resistance and good material performance of bipolar plates are very important in PEMFC aqueous acidic environment. Modification on the surface metal is needed to overcome this problem since it has certain life, which is prone to corrodes before physical damage occurs (Asri et al. 2017; Taherian 2014). It was an alternative method to slow down the corrosion process as Nb–C (niobium carbide) and Cr–C (chromium carbide) were suggested as coating on SUS 316 L plates. The best four performing parameters are defined in Table 3 for CR and ICR values of Nb–C and Cr–C. According to Figure 2(a) and 2(b), the shape and position of the polarization curves affects by corrosion potential (E_{corr}) and samples had shown anodic reactions after been coated. Extrapolation of cathodic current density shows good in corrosion resistance. Table 7 presents the values of the corrosion potential (E_{corr}) and corrosion current density (I_{corr}) for each coating parameters selected in polarization conditions. The highest E_{corr} for Nb–C and Cr–C are –11 and – 106 V, that presented by samples 8 and 1. It can be observed that by increasing the E_{corr} value, the I_{corr} value will decrease and potentially reduce the corrosion attack after coatings. However, Nb–C 4 is chosen based on the lowest I_{corr} presents with good ICR among other parameters.

TABLE 7. The values of and E_{corr} and I_{corr} for SUS 316 L plates with Nb-C and Cr-C coatings in polarization conditions

Material	Nb-C		Material	Cr-C	
Sample	E_{corr} (mV)	I_{corr} (A/cm^2)	Sample	E_{corr} (V)	I_{corr} (A/cm^2)
316 L	– 709	1.75430×10^{-9}	316 L	– 709	1.75430×10^{-9}
1	– 31	137.71×10^{-12}	1	– 106	70.1740×10^{-12}
4	– 78	107.550×10^{-12}	4	– 139	78.2870×10^{-12}
5	– 232	1.281×10^{-9}	6	– 387	86.431×10^{-12}
8	– 11	1.597×10^{-9}	7	– 300	1.149×10^{-9}

Figure 3 exhibits the ICR values measured at temperatures of 40, 60, 80, and 100 ºC for the four best parameters. It can be seen the ICR values of SUS 316 L (4.6307 mΩ cm² to 2.6358 mΩ cm² at 40 to 100 °C) decreased with the increase of temperature from 40 to 100 °C after Nb–C and Cr–C coatings. As in Figure 3(a), the ICR results after Nb–C coatings showed the performance of the samples from the lowest value in order of Nb–C 7 < Nb–C 4 < Nb–C 8 < Nb–C 3 < SUS 316 L. Whereas, the ICR for Cr–C coatings are Cr–C 1 < Cr–C 5 < Cr–C 8 < Cr–C 6 < SUS 316 L as seen in Figure 3(b). Overall, the ICR values for SUS 316 L plates after coatings meets the requirement of U.S DOE, less than 10 mΩ cm².

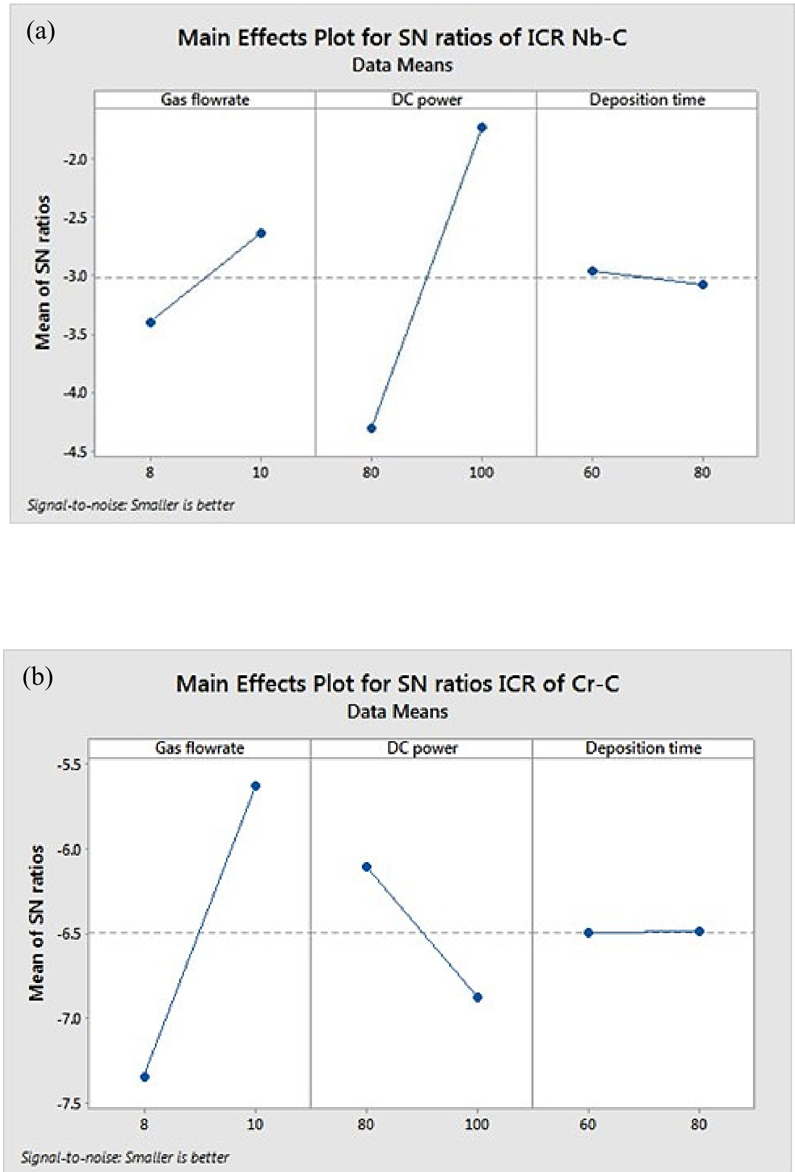
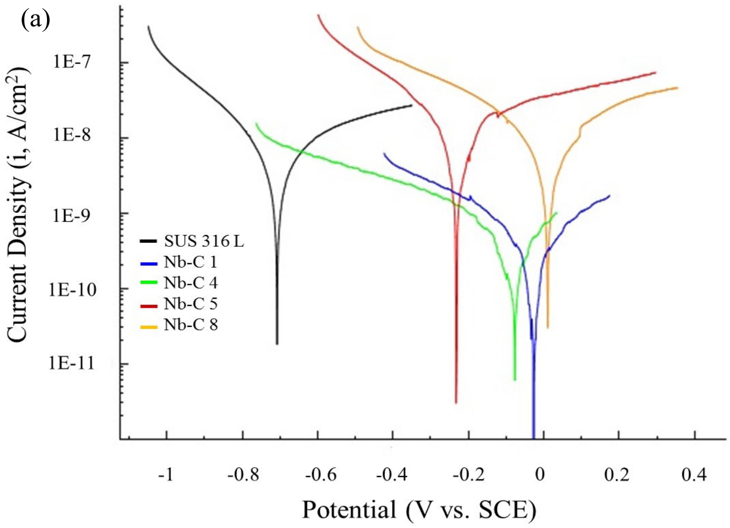


FIGURE 1. The main effects plot for S/N ratios of (a) ICR for Nb–C coating and (b) ICR for Cr–C coating



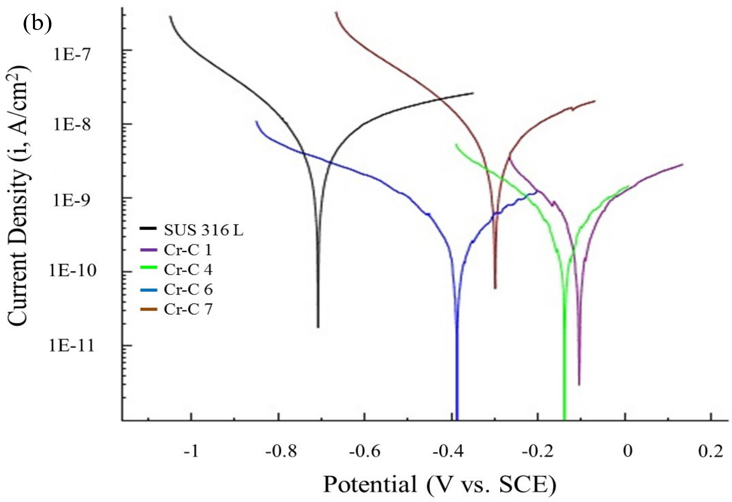
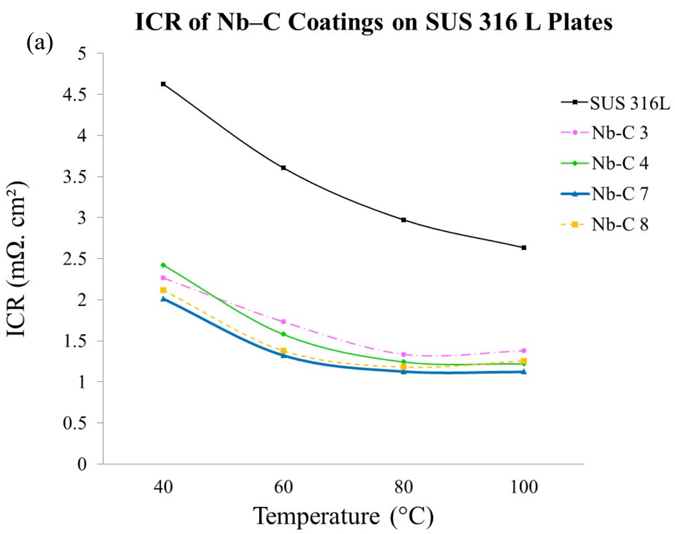


FIGURE 2. Polarization curves of Tafel plots of (a) Nb–C and (b) Cr–C coatings with different parameters



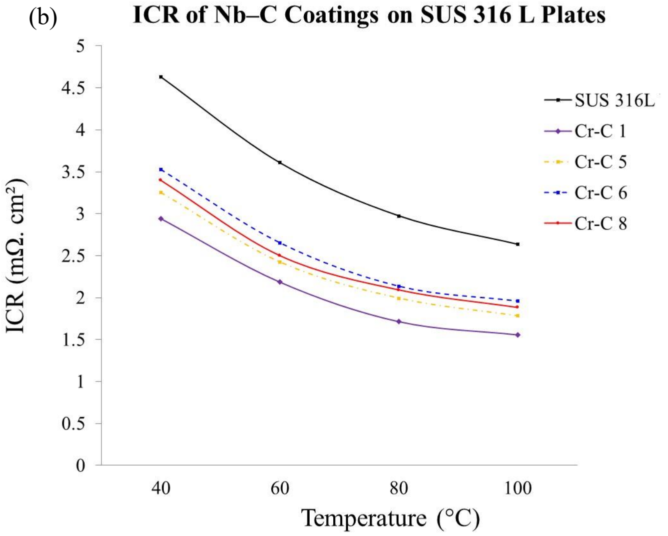


FIGURE 3. ICR values before and after coatings of (a) Nb–C and (b) Cr–C with different parameters

CONCLUSION

In this study, an orthogonal Taguchi L_8 array was used to investigate the effects of the synthesis parameters on Nb–C and Cr–C coatings using PVD coating method. The parameters studied is used to optimize the coating process as well as to improve corrosion behavior and interfacial contact resistance on SUS 316 L plates. Optimization of coating parameters are selected for sample Nb–C 4 (8 sccm, 100 W and 80 min) and Cr–C 1 (8 sccm, 80 W and 60 min), in relation with lowest current density (I_{corr}) and ICR values. In addition, the study found the DC power (89.54%) and the gas flow rate (69.01%) gives high percentages contribution to the ICR values of Nb–C and Cr–C after coating. The test results are statistically significantly with the P–value ≤ 0.05. Moreover, SUS 316 L had achieved the standard of U.S. DOE technical target in I_{corr} (≤ 1 μA/cm^2) and ICR (≤ 10 mΩ. cm^2) as a bipolar plate potential in PEMFC.

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