Optimisation of Sintering Factors of Titanium Foams Using Taguchi Method

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Abstract

Metal foams have the potential to be used in the production of bipolar plates in Polymer Electron Membrane Fuel Cells (PEMFC). In this paper, pure titanium was used to prepare titanium foam using the slurry method. The electrical conductivity is the most important parameter to be considered in the production of good bipolar plates. To achieve a high conductivity of the titanium foam, the effects of various parameters including temperature, time profile and composition have to be characterised and optimised. This paper reports the use of the Taguchi method in optimising the processing parameters of pure titanium foams. The effects of four sintering factors, namely, composition, sintering temperature, heating rate and soaking time on the electrical conductivity has been studied. The titanium slurry was prepared by mixing titanium alloy powder, polyethylene glycol (PEG), methylcellulose and water. Polyurethane (PU) foams were then impregnated into the slurry and later dried at room temperature. These were next sintered in a high temperature vacuum furnace. The various factors were assigned to an L_9 orthogonal array. From the Analysis of Variance (ANOVA), the composition of titanium powder has the highest percentage of contribution (24.51) to the electrical conductivity followed by the heating rate (10.29). The optimum electrical conductivity was found to be 1336.227 ± 240.61 S/cm^{-1} for this titanium foam. It was achieved with a 70% composition of titanium, sintering temperature of 1200°C, a heating rate of 0.5°C/min and 2 hours soaking time. Confirmatory experiments have produced results that lay within the 90% confidence interval.

Keywords: PEMFC, Slurry Method, Metal Foam, electrical conductivity.

1. INTRODUCTION

The polymer electrolyte membrane fuel cell (PEMFC) is a cell of choice for future automotive propulsion applications, in part because of its modestly low operation temperature (< 100°C) [1]. Hydrogen and oxygen were used in PEMFC system to generate electricity with water as the only byproduct. The advantage of this system is very environmental friendly. The components in this system are MEAs (membrane electrode assemblies), anode and cathode. We can also call anode and cathode as bipolar plate. This bipolar plate are designed to accomplish many functions, such as to distribute reactants uniformly over the active areas, remove heat from the active areas, carry current from cell to cell and prevent leakage of reactants and coolant [2].

Conventionally, carbon based materials have been selected to make the bipolar plate. These carbons are chemically steady in a fuel cell environment and produce the highest electrochemical power output. However, the lack of mechanical strength with is natural with carbon, limits the size of the bipolar plate can be produced, as well same as the volumetric power density [3]. Because of these disadvantages, many researchers have studied the alternative material to replace carbon as a bipolar plate. The alternative materials are carbon-carbon composite, carbon-polymer composite and metals [4]. This study, focused mainly on the possibility of adopting titanium (Ti) foam as bipolar plate.

The traditional approach to experimental work is to vary one factor at a time, holding all other factors fixed. This method does not produce satisfactory result in a wide range of experiment settings [5]. In this study, the Design Of Experiment (DOE) method which is called Taguchi method is adopted to determine and optimize the sintering parameters. In recent years, the Taguchi method has become a powerful tool for improving productivity during research and development [6]. The effect of four factors: composition, sintering temperature, heating rate and soaking time on the electrical conductivity were investigated. The optimum sintering condition was proposed and confirmation experiments were conducted.

2. METHODOLOGY

2.1 Sample Preparation

Titanium powder was purchased from Sumitomo Titanium Corporation of Japan. The particles are spherical in shape with the diameters less than 45μm. The density of the titanium powder was 4540 kg/m^3 and the melting point was 1670^{o}C. Polyethylene glycol (PEG) and methylcellulose (CMC) were used as binders. PEG and methylcellulose are water soluble materials.

Firstly, PEG and CMC were stirred in distilled water for one hour. Pure titanium powder was subsequently added to the solution and stirred for two hours. The titanium slurry was used to impregnate polyurethane (PU) foam. The PU foams were dipped into the slurry and the dipping and drying processes were repeated until the struts of the foam were completely coated with titanium slurry. The excess slurry was then removed by pressing the foam under a roller. Lastly sintering process will be carried out for these samples followed the orthogonal array of Taguchi method.

2.2 Design of Experiment (DOE)

There are many sintering parameters that can affect the mechanical and physical properties of titanium foam. A design of experiment (DOE) methods is necessary for the experimental work which involving many inputs to minimise the number of experiments need to be performed. The most frequently used methods are partial or full factorial and the Taguchi approach. The Taguchi approach is mostly used for scientific research. The method is based on balanced orthogonal arrays [7]. In this work, L_9 (3^4) orthogonal array consisting of 9 experiment trials and 4 column is used followed by ANOVA (ANalysis Of VAriance) to determine the significant level and contribution of each variables to the electrical conductivity. The main variables involved in this study are shown in Table 1. Three levels for each variable refer to the maximum and minimum limit that influences electrical conductivity.

Table 1: Factor level in the experiment and Orthogonal array

Experiment no.	Factor					Experimental value		
	A	B	C	D	Composition	sintering Temperature (^oC)	Heating rate (^oC min^{-1})	Soaking time (min)
1	1	1	1	1	60	1200	1.5	60
2	1	2	2	2	60	1250	1	90
3	1	3	3	3	60	1300	0.5	120
4	2	1	2	3	65	1200	1	120
5	2	2	3	1	65	1250	0.5	60
6	2	3	1	2	65	1300	1.5	90
7	3	1	3	2	70	1200	0.5	90
8	3	2	1	3	70	1250	1.5	120
9	3	3	2	1	70	1300	1	60

2.3 Characterisation of Electrical Conductivity

From the ohm's law, the current (I) to the applied voltage (V) as follows;

$$V = IR \quad (1)$$

where R is the resistance of the material through which the current is passing. The value of R is influenced by sample configuration, and for many materials is independent of current. The resistivity (ρ) is independent of sample geometri but related to R through the expression where l is the distance between the two points at which the voltage is measured, and A is the cross-sectional area perpendicular to the direction of the current.

$$\rho = R\frac{A}{l} \quad (2)$$

$$\sigma = \frac{1}{\rho} \quad (3)$$

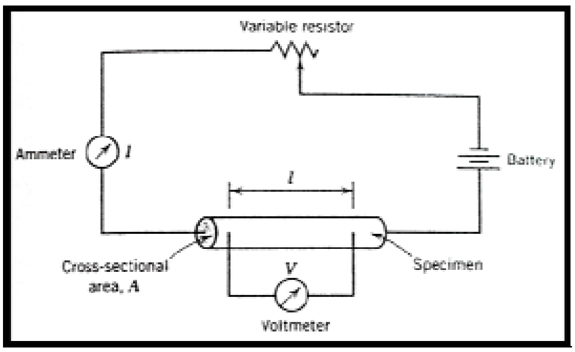

Fig. 1Schematic diagram of an experimental arrangement for measuring electrical resistivity [8]

Using equation 3 we can get the electrical conductivity using the resistivity result. Figure 2 show the experiment arrangement for measuring electrical resistivity.

3. RESULT AND ANALYSIS

The electrical conductivity was calculated using resistivity from the sample after the sintering process. Three replications were recorded for each experiment as shown in Table 2. The ANOVA technique was used to establish the relative significance of the factors. As shown in Table 2, a combination of A_3 B_1 C_3 D_2 gives a maximum electrical conductivity (1225.41 S/cm). While, a combination of A_2 B_3 C_1 D_2 produced a minimum electrical conductivity for titanium foam (526.87 S/cm). These values are much higher compare to carbon fibre epoxy composite (300 S/cm) and carbon based polypropylene composite (36.4 S/cm) [10], [11]. Overall, the value for electrical conductivity of the samples was much higher than the requirement electrical conductivity for bipolar plate PEMFC. From the previous research, the requirement for electrical conductivity should be over 10 S/cm [9].

Table 2: Result Electrical Conductivity of Titanium Foams

Variable	Degrees of Freedom, f_n	Sum squared, S_n	Variance, V_n	Pure Sum squared, S_n	Varience ratio, F_n	Critical F value	Contribution, P_n
A	2	471103.8	235551.9	392419.3	5.987	$F_{0.025,2,18}$=4.5597	24.51
B	2	116926	58463	38241.5	1.486		2.39
C	2	243445.1	121722.6	164760.6	3.094	$F_{0.1,2,18}$=2.6239	10.29
D	2	61492.2	polled				
error	18	708160.4	39342.2				62.81
Total	26	1601128					100.00

Besides that, the analysis of variance (ANOVA) was used to establish the relative significance of the factors. ANOVA is a table of information that displays relative influences of factor and interactions assigned to the column of an OA. Table 3 shows the results of the ANOVA after "pooling" with "at least 97.5% confidence". From the ANOVA table, the effects of sintering factors on the electrical conductivity were determined. The composition of the titanium powder has significant effect on the electrical conductivity at the 97.5% significance level or $\alpha = 0.025$ followed by the heating rate with a significant factor at $\alpha = 0.1$. On the other hand, the soaking time factors did not have any contribution for this experiment. For the sintering temperature factor, F ratio did not exceed 90% of significant level (2.6239) but it still gave 2.39% contribution for this experiment.

Table 3: ANOVA for electrical conductivity of titanium foam at $\alpha = 0.025$

Experiment	Replication, conductivity (S/cm^{-1})			Average
	R1	R2	R3	
1	943.16	522.47	653.68	706.44
2	1195.13	798.2	697.42	896.92
3	856.25	927.24	892.45	891.98
4	830.00	927.24	892.45	673.31
5	985.53	707.67	681.65	791.62
6	529.62	504.37	546.61	526.87
7	1432.15	909.36	1334.71	1225.41
8	1360.09	843.89	785.58	996.52
9	991.00	1056.68	735.85	927.84

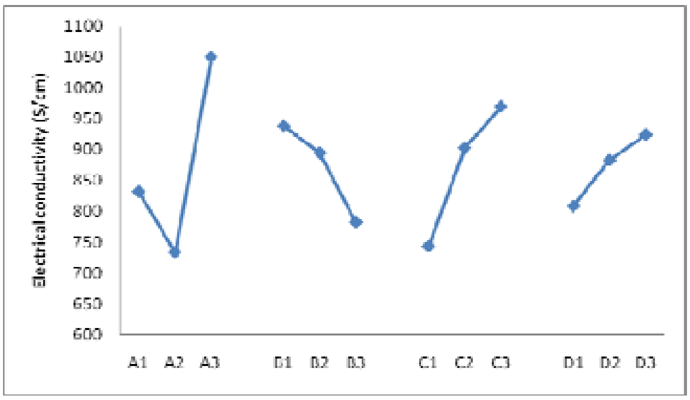

Fig. 2 Response graphs of electrical conductivity against various factors

Based on the ANOVA, the main effect of the experiments is calculated based on the highest average value as shown in Figure 2. As shown by the response plot in Figure 2, a combination of A3 B1 C3 D3 is the highest yield, i.e., composition 70% of titanium powder, sintering temperature1200°C, heating rate 0.5°C/min and soaking time 120 minutes.

The expected result at optimum performance is as shown in Table 4. The optimum performance is as high as 1336.23 S/cm while the range of the optimum performance based on 90% confidence level is 1095.62 S/cm < μ < 1576.83 S/cm of the electrical conductivity. The optimum parameter has been proven in the confirmation experiment that is conducted at the combined setting of A3 B1 C3 D3 and the result fell within the predicted 90% confidence interval as shown in Table 4.

Table 4: Optimum sintering parameter, optimum performance and confirmation experiment

Optimum parameter: A3 B1 C3 D3 (Composition, 70% of titanium powder; sintering temperature, 1200°C; Heating rate, 0.5°C/min; soaking time, 120 minute)				
Optimum performance: 1336.23 S/cm Confident interval: ± 240.6044 at 90% confident level (α = 0.1) Range of optimum performance: 1095.6226 S/cm < μ < 1576.8314 S/cm				
Confirmation experiment				
Repeat	1	2	3	Average
S/cm	1285.25	1386.68	1409.47	1359.8

4. DISCUSSION

From the analysis of the experimental results using the Taguchi method, the composition of titanium has the most significant effect on the electrical conductivity. Among the three compositions, 70% of titanium has the highest electrical conductivity. When more particle of titanium in the sample, more connection among of particle is obtained and more current (I) pass through in the sample. From the Ohm's law theory, if current (I) was increases so the resistance (R) is also increases, therefore the electrical conductivity will be increased.

The second factor which has a significant influence to the electrical conductivity is the heating rate. When the heating rate is increase, the porosity will be decreased and the grains become finer [5]. As porosity decrease, the sample will shrink further, and there will reduce the area of the sample. As the area of the sample decreases, the resistivity (ρ) will also decreases and the electrical conductivity will be increased, as state in equations 2 and 3.

5. CONCLUSION

Electrical conductivity of the titanium foams was optimised using the Taguchi method. An L_9 orthogonal array was used to vary the experiment variables. ANOVA showed that the composition of titanium powder and the heating rate as the most significant that influence the electrical conductivity of the titanium foams produced. The optimum sintering parameter were found to be A_3 B_1 C_3 D_3 corresponding to 70% of composition titanium powder, 1200°C of sintering temperature, 0.5°C/min of heating rate and 120 minutes of soaking time. Confirmation experiments indicated that when sintering titanium foam was performed at the optimum condition, an electrical conductivity of 1359.8 S/cm can be achieved.

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