

Research on the optimal design of surface treatment process for Al-B₄C composite by the method of orthogonal experiment

Qingfang Zeng^{1, a}, Qiulin Li^{1, b, *}, Wei Liu^{2, c}

¹Advanced Materials Institute, Graduate School at Shenzhen, Tsinghua University, Shenzhen 518055, China

²School of Materials Science and Engineering, Tsinghua University, Beijing 100084, China

^aemail: cqf13@mails.tsinghua.edu.cn, ^bemail: liql@sz.tsinghua.edu.cn, ^cemail: liuw@mail.tsinghua.edu.cn

Keywords: Metal Matrix Composite; Surface Treatment; Optimal Design

Abstract. Anodizing is a surface treatment method and this is the first to report research on anodizing on Al-B₄C composite. The growth of anodic alumina oxide was conducted on Al-B₄C composite in cost-effective sulfuric acid electrolyte. In this paper, we have discussed the effect order of anodizing voltage, electrolyte temperature, and concentration of sulfuric acids, which affect thickness and growth process of the anodic oxide film. The results indicate that anodizing voltage is the main factor that affects thickness and growth process, and affects Al-B₄C composite's corrosion resistance; the minor factor is sulfuric acid concentration. And the optimum experiment condition is concluded to be 20 wt. % H₂SO₄, 25V and 12°C.

1. Introduction

Al-B₄C composite is an ideal neutron absorption material for spent nuclear fuel storage racks. B₄C is a ceramic material which has good corrosion resistance even in aggressive environment and aluminum. Aluminum and its alloys have excellent physical and mechanical properties such as low density, good electromagnetism and high strength/weight ratio [1] [2] [3]. Although aluminum has a huge advantage when compared with other metals, it is not always completely impervious to corrosion because of the existence of magnesium and ferrum elements [4]. The self-protective oxide layer on aluminum can become unstable when it is exposed to extreme pH levels or for a long time. So, it is high demanded to develop highly effective and selective method to resolve this problem. Ceramic coating is a promising means to combine advantages of the metal substrate and the ceramic materials, which can simultaneously meet environmental service demands such as corrosion resistance, and mechanical properties [5] [6] [7] [8]. Many techniques like micro-arc oxidation (MAO) [9], chemical vapor deposition (CVD), physical vapor deposition (PVD), plasma spraying and gas-flame spraying [10] have been put forward to prepare those excellent candidates. The coatings produced by MAO, CVD or PVD techniques are harder and have more corrosion resistance but these techniques also have some disadvantages, for instance, they need complicated equipment and large energy. The coatings produced by plasma spraying and other techniques have insufficient adhesion to the base material. Among various synthesis methods, electrochemical anodization has been demonstrated as a facile and excellent approach to increase the life time of aluminum by fabricating ceramic coating on aluminum sheet. It is superior over other techniques due to the following advantages: (i) relatively short process time, (ii) effective control of the size and shape of the grains, (iii) easy to anchor firmly to the substrate and (iv) less cost consumption [11].

To the best of people's knowledge, this is the primary priority to discuss about effect order of sulfuric acid concentration, anodizing voltage and electrolyte temperature on thickness of anodic oxide film formed on Al-B₄C composite in sulfuric acid solutions.

2. Experimental details

2.1. Material

1060Al-31 wt. %B₄C composite specimens (with compositions provided in Table 1 and prepared by our own) of dimensions 50 × 25 × 3mm, have been used as the substrate material.

Table 1 Chemical composition (wt. %) of Al alloy in the Al-B₄C composite

Ti	Fe	Si	Al
5.08	1.02	0.88	Allowance

2.2. Anodizing procedure and sealing

The preparation process principally involves three successive stages: surface pretreatment, anodization process and sealing procedure. Firstly, all samples were dry-polished using abrasive glass sand (grid #180) to obtain the surface with roughness of about Ra 2.4μm.

Then the specimens were degreased in acid aluminum degreasing agent and alkali cleaned with 5% NaOH at ambient temperature for 2 min followed by desmutting in 5% HNO₃ at room temperature for 3 min.

Between each step, rinsing in distilled water was performed.

Finally, the samples were rinsed with ethanol and dried. Anodic oxidation of composite was performed in an electrochemical cell, where the composite sheets were used as anode and aluminum sheets as a counter-electrode. In order to investigate the influence of electrolytes, anodizing voltage and electrolyte temperature on anodic coating, an orthogonal experiment with four levels and three factors was designed (as Table 2). Fifteen minutes after anodizing, the anodic film was sealed in 5.5g/L nickel acetate solution with 8.2g/L boric acid, at 70°C for a quarter hour.

Table 2 Factors and its values of Taguchi orthogonal experiment

Level	H ₂ SO ₄ concentration/ wt. %	Anodizing voltage / V	Electrolyte temperature /°C
1	10	10	8
2	15	16	12
3	20	20	16
4	25	25	20

3. Results and discussion

Most of the researchers had only focused on investing respectively the longitudinal influence of anodizing voltage (or current density), the concentration of sulfuric acid and electrolyte temperature on anodic oxidation films. The transverse effect of these factors, namely the effect level of these factors on the coatings was seldom concerned. So in the present work, a special attention has been given to the variation of the coating thickness by using the Taguchi orthogonal L₁₆ (4⁵) array.

The variation of the anodic thickness with three factors of the orthogonal experiment is presented in Table 3. Table 4 shows film thickness ranges of the experiment factors when the thickness of anodic oxide film is used as the index. From table 4, it presents that the range of the anodizing voltage is 32.04μm, which is the biggest value among ranges of all experiment factors, indicating that the anodizing voltage is the main factor affects thickness of anodic oxide film according to the property of Taguchi orthogonal experiment design. The minor effect factor is the concentration of sulfuric acids because its range is 25.52μm. Electrolyte temperature has minimal effect on thickness of anodic oxide film, compared with the other two experiment factors.

Table 3 The Design and Result of Orthogonal experiment

No	H ₂ SO ₄ concentration/ wt. %	Anodizing voltage/V	Electrolyte temperature/°C	Thickness of anodic film/μm
1	10	10	8	5.35
2	10	16	12	20.70
3	10	20	16	15.20
4	10	25	20	30.41
5	15	10	12	6.20
6	15	16	8	22.56
7	15	20	20	38.23
8	15	25	16	41.83
9	20	10	16	23.49
10	20	16	20	51.58
11	20	20	8	25.43
12	20	25	12	46.60
13	25	10	20	25.10
14	25	16	16	28.83
15	25	20	12	39.36
16	25	25	8	60.47

Table 4 Range of Anodic Oxide film for experiment elements (μm)

Item	H ₂ SO ₄ concentration/ wt. %	Anodizing voltage/V	Electrolyte temperature/°C
M ₁	14.9025	14.5225	24.19
M ₂	24.455	24.6675	25.715
M ₃	36.775	29.555	28.3375
M ₄	38.44	45.8275	36.33
R	23.5375	31.305	10.615

M₁ - M₄: the average of sum of films' thickness at different levels for each factor

R: the difference between the maximum and minimum of m₁ - m₄

Every curve for each factor in Fig.1 was obtained from every level and its data M_i, the average of sum of films' thickness at level i. Fig.1 indicates the variation tendency of the anodic oxide thickness with concentration of experiment factors-sulfuric acid, anodizing voltage and electrolyte temperature. At low concentration or low anodizing voltage, a relatively thin anodic oxide film is obtained. As the concentration and anodizing voltage increase, the film thickness is augmented. From Fig.1, an optimum condition, in which a comparatively thick layer forms, can be concluded that the concentration of sulfuric acid is 20 wt. %, the anodizing voltage is 25V and electrolyte temperature is 12°C. Thickness of layers on Al-B₄C composite affects corrosion resistance of the composite.

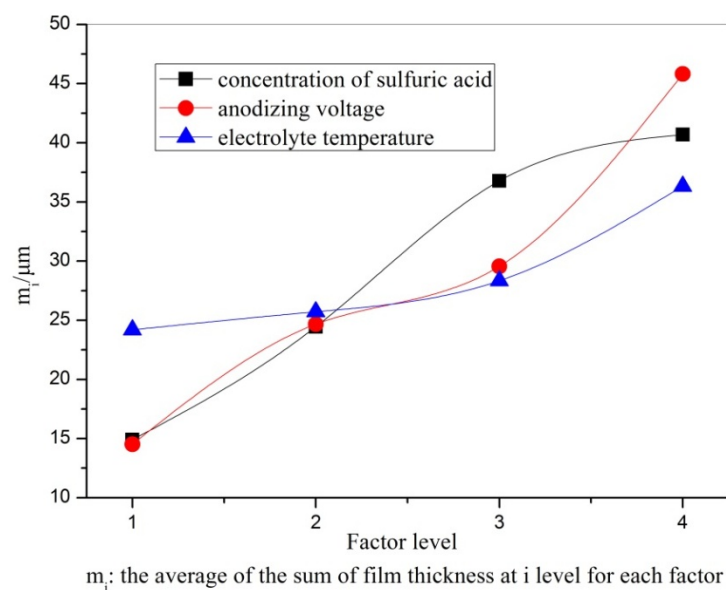


Fig.1. Trend analysis graph for orthogonal experiment factors

4. Conclusions

This paper reports on the fabrication of anodic coatings by anodization of Al-B₄C composite and discusses effect order on alumina film thickness of the three experiment factors, electrolyte concentration, anodizing voltage and electrolyte temperature, using Taguchi orthogonal experimental method.

(1) The results demonstrate that anodizing voltage is the main experiment factor affecting the alumina thickness and growth rate. The minor experiment factor is the concentration of sulfuric acids, and the electrolyte temperature has the minimal impact among the three experiment factors.

(2) Thick layers can be formed on Al-B₄C composite at experiment condition- 25wt. % H₂SO₄, 25V and 8°C. This is the optimal oxidation parameters of Al-B₄C composite to obtain thick layers on composite. But thickness is only one of factors that affect composite's corrosion resistance. It needs more research on layers' surface morphology and cross-section morphology between the layers and Al-B₄C composite matrix. When the layers are dense and compact with Al-B₄C composite matrix, composite's corrosion resistance is better with thicker lays.

Acknowledgement

This work was financially supported by China Nuclear Power Engineering Co. Ltd. (No. 20139660030).

References

- [1] M. García-Rubio, P. Ocón, M. Curioni, G.E. Thompson, P. Skeldon, A. Lavía, I. García, Degradation of the corrosion resistance of anodic oxide films through immersion in the anodizing electrolyte, *Corros. Sci.* 52 (2010) 2219-2227.
- [2] V. Moutarlier, M.P. Gigandet, B. Normand, J. Pagetti, EIS characterization of anodic films formed on 2024 aluminum alloy, in sulfuric acid containing molybdate or permanganate species, *Corros. Sci.* 47 (2005) 937-951.
- [3] J.j. Suay, E. Gimenez, T. Rodriguez, K. Habib, J.J. Saura, Characterization of anodized and sealed aluminum by EIS, *Corros. Sci.* 45 (2003) 611-624.

- [4] Jian-Min Shi, Lin Zhang, Jing Chen, Study on corrosion behavior of Al-B₄C in Spent fuel storage environment, Chinese Society for Corrosion and Protection, 33 (2013) 419-424.
- [5] V. Raj, M. Mumjitha, Comparative study of formation and corrosion performance of porous alumina and ceramic nanorods formed in different electrolytes by anodization, Mater. Sci. and Eng. B 179 (2014) 25-35.
- [6] Tzung -Ta Kao, Yao -Chung Chang, Influence of anodization parameters on the volume expansion of anodic aluminum oxide formed in mixed solution of phosphoric and oxalic acids, Appl. Surf. Sci. 288 (2014) 654-659.
- [7] Song-Mei Li, Bin Li, Jian-Hua Liu, Corrosion property of Super hydrophobic membrane layer made by chemical etching and anodization in Al alloy surface application, Chinese Journal of Inorganic Chemistry 28 (2012) 1755-1762.
- [8] L. Bouchama, N. Azzouz, N. Boukmouche, Enhancing aluminum corrosion resistance by two-step anodizing process, Surf. & Coat. Technol. 235 (2013) 676-684.
- [9] X. Yang, X. Zhu, H. Jia, T. Han, Oxygen Evolution: the Mechanism of Formation of PAA, Monatsh. Chem. 140 (2009) 595.
- [10] Hossein Fadaee, Mehdi Javidi, Investigation on the corrosion behavior and microstructure of 2024-T3 Al alloy treated via plasma electrolytic oxidation, Journal of Alloys and Compounds 604 (2014) 36-42.
- [11] Zu-Fang Zhu, Aluminum anodization and surface treatment technology, Chemical Industry Press, Beijing, 2004.