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Role of Al₁₈B₄O₃₃ Whisker in MAO Process of Mg Matrix Composite and Protective Properties of the Oxidation Coating

Yanqiu Wang^{1)*}, Xiaojun Wang²⁾, Kun Wu²⁾, Fuhui Wang^{1,3)}

- 1) Corrosion and Protection Laboratory, Education Ministry Key Laboratory of Superlight Materials and Surface Technology, Harbin Engineering University, Harbin 150001, China
- 2) School of Materials Science and Engineering, Harbin Institute of Technology, Harbin 150001, China
- State Key Laboratory for Corrosion and Protection, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China

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Selective growth of oxidation coating was observed on Mg matrix composite Al $_{18}B_4O_{33w}$ /AZ91 (a composite with Al $_{18}B_4O_{33}$ crystal whisker as reinforced phase) when this composite was treated by microarc oxidation (MAO) technique, and then the role of Al $_{18}B_4O_{33}$ whisker in the process of MAO was analyzed. The protective properties of MAO coating also were investigated. Scanning electron microscopy (SEM) was used to characterize the existing state of Al $_{18}B_4O_{33}$ whisker in MAO process and the microstructure of MAO coating. Corrosion resistance of the bare and coated composite was evaluated by immersion corrosion test and potentiodynamic polarizing test. Wear resistance of MAO coating was investigated by a ball-on-disc friction and wear tester. The results showed that sparking discharge did not occur on Al $_{18}B_4O_{33}$ whisker and the whisker existed in the coating as a heterogeneous phase when MAO coating grew on the composite; then the whisker would be covered gradually with growing thick of the coating. Corrosion current density of the coated composite was decreased by 4 orders of magnitude compared with that of the uncoated composite; excellent corrosion resistance was closely related to the compact whisker-coating interface since Al $_{18}B_4O_{33}$ whisker did not induce structural defects. The coating also exhibited high wear resistance and a slight adhesive wear tendency with bearing steel as its counterpart material.

KEY WORDS: Mg matrix composite; Whisker; Microarc oxidation (MAO); Coating; Corrosion resistance

1. Introduction

In recent years, there has been an increasing interest in researching and developing Mg matrix composites for applications in the region of aerospace because of their low density, high specific strength, high specific stiffness and low coefficient of thermal expansion *etc.*^[1-4]. Many discontinuous reinforced phases, such as short fibers, particles, or crystal whiskers, have been applied to fabricate Mg matrix composites^[1-4]. Among these reinforced phases, aluminum borate whisker (Al₁₈B₄O₃₃) shows potential applications in Mg matrix composites because of its low cost and excellent properties^[5].

While at the same time, Mg matrix composites including those reinforced by aluminum borate whisker are very susceptible to

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corrosion due to two reasons. On the one hand, Mg matrix is usually susceptible to corrosion because of its intrinsic chemical activity^[6]; on the other hand, structural flaws and/or galvanic coupling within metal matrix composites could result in increased localized corrosion of the matrix^[7–10]. In addition, Mg matrix composites generally have poor anti-wear performance due to the poor wear resistance of Mg alloys, particularly during sliding in oxidizing environments, even if the ceramic reinforced phase in composites may improve their tribological properties to some extent^[11–13]. Consequently Mg matrix composites could hardly be used without appropriate surface treatment; however, the information about surface treatment of Mg matrix composites is very limited till now. Yue et al. [14,15] have investigated laser surface treatment and laser cladding of Mg matrix composite reinforced by SiC particle, and corrosion resistance of the composite could be improved. In addition, chemical conversion coating technology has also been applied to Mg matrix composite for protection^[16,17].

Microarc oxidation (MAO), as an effective method to prepare protective coating on valve metals, has become an important surface treatment technology for Mg alloys and Mg matrix

^{*} Corresponding author. Ph.D.; Tel./Fax: +86 451 82519190; E-mail address: qiuorwang@hrbeu.edu.cn (Y. Wang).

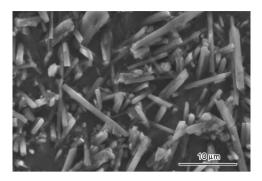


Fig. 1 SEM image of Al₁₈B₄O_{33w}/AZ91 composite.

composites^[18–26]. For metal matrix composites, reinforced phases within valve metals might affect sparking discharge, consequently affect formation and growth of MAO coating since reinforced phases generally are ceramic and cannot be microarc-oxidized^[23–26]; therefore, the effects of reinforced phase on MAO process of metal matrix composites are deserved to be investigated to obtain MAO coatings with excellent properties.

In fact, the previous study of authors has shown that MAO coating could be successfully prepared on Mg matrix composite reinforced by Al₁₈B₄O₃₃ whisker and that the effects of this whisker on microarc discharge behavior and coating characteristics have been observed and analyzed roughly^[27]; however, further study showed that the discussion about the effects of $Al_{18}B_4O_{33}$ whisker in literature^[27] was not exact because the possible effects of structural defects in the composite, such as flaws resulting from composite preparation, on voltage evolution trend and MAO process were neglected. Unfortunately, the factor in respect of composite preparation was not realized and was not excluded when analyzing and discussing the effect mechanism of Al₁₈B₄O₃₃ whisker in the previous study. That is, study about effects of Al₁₈B₄O₃₃ whisker on MAO process was interfered by other factors, so that the speculation about effect mechanism of Al₁₈B₄O₃₃ whisker in literature^[27] was inexact.

Metal matrix composites are a kind of artificially designed materials; structural defects are very easy to form when the composites are prepared. In the present study, structural defects in Mg matrix composite Al₁₈B₄O_{33w}/AZ91 were excluded as possible to ensure the effects of Al₁₈B₄O₃₃ whisker can be studied separately and then some different results are obtained. The role of Al₁₈B₄O₃₃ whisker in MAO process was observed; its effect mechanism on MAO was re-discussed. At the same time, corrosion resistance and wear resistance of the MAO coatings prepared on this composite were evaluated.

2. Experimental

The material used in this study was Mg matrix composite Al₁₈B₄O_{33w}/AZ91 which had about 20% volume fraction of Al₁₈B₄O₃₃ whisker. The matrix of Mg alloy AZ91 has a nominal chemical composition of Mg–(8.5–9.5)Al–(0.45–0.9)Zn in mass%. The composite was prepared by a squeeze casting method. The properties of Al₁₈B₄O₃₃ whisker and the details of composite preparation had been described elsewhere^[28]. The original morphology of Al₁₈B₄O_{33w}/AZ91 composite shows that Al₁₈B₄O₃₃ whiskers with diameter in the range of about 0.1–1.0 µm are distributed on the composite surface relatively homogeneously (as shown in Fig. 1).

Disk specimens, 2.5 mm in thickness and 20 mm in diameter, were polished with 1000# abrasive paper, degreased ultrasonically in acetone, and then immersed in electrolyte for MAO treatment. The electrolyte was prepared from a solution of sodium silicate and potassium fluoride in distilled water. The specimen and stainless steel sheet were used as the anode and the cathode, respectively. An MAO device with AC pulse power supply was adopted. A constant current density of 60 mA/cm² was applied. At the control mode of constant current, a current was preset and kept constant during MAO; the cell voltage varied with oxidation time and the response of voltage to time was recorded. For comparison, Mg alloy AZ91 was also microarc oxidized using the identical parameters and then its voltage response was recorded.

Surface morphology evolution of the composite at the early stage of MAO was observed by scanning electron microscopy (SEM, Hitachi S-570 and S-4700). Surface morphology and cross-sectional microstructure of the coating formed at the prolonged stage of MAO were also observed by SEM.

Corrosion resistance of the bare and coated composites was evaluated by salt immersion test and electrochemical test in 3.5% NaCl solution. Potentiodynamic polarizing curves were measured by using a potentiostat (M273). A three-electrode cell, with sample as working electrode, saturated calomel electrode (SCE) as reference electrode and platinum sheet as counter electrode, was employed in this test. After 10 min initial delay, scan was conducted at a rate of 0.5 mV/s from -250 mV vs open circuit potential (OCP) toward more positive direction until breakdown of coating occurred.

Tribological behaviors of the coated $Al_{18}B_4O_{33w}/AZ91$ composite were investigated by using a ball-on-disc friction and wear tester (CJS111A, developed by Harbin Institute of Technology) under conditions of low load and low sliding speed. The test was carried out using a GCr15 bearing steel sphere of 6 mm diameter as the counterpart material under a load of 0.5 N in air without lubrication, with test conditions of 100 r/min and 3.5 mm radius of wear track. The morphology and the elemental compositions of the worn coating were observed and determined by SEM and energy dispersive X-ray spectrometry (EDS).



Fig. 2 Surface morphology evolution of Al₁₈B₄O_{33w}/AZ91 composite at early different stages of MAO: (a) 25 s, (b) 30 s, (c) 60 s.

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