

## Effect of chloride ion level on the corrosion performance of MAO modified AZ31 alloy in NaCl solutions

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### ABSTRACT

Microarc oxidation coatings were fabricated on AZ31 magnesium alloy in the electrolyte of sodium phosphate. Potentiodynamic polarization and electrochemical impedance spectroscopy tests were employed to investigate the electrochemical corrosion behavior. The corroded surface was characterized by an optical microscope and X-ray diffraction. The influence of chloride ion concentration on the corrosion resistance of microarc oxidation coated AZ31 alloy is discussed. The corrosion current density enlarged ratio and the charge transfer resistance reduced ratio indicated that the extent of the corrosion damage of microarc oxidation coated AZ31 alloy is much higher when chloride ion concentration is greater than 5%. A corrosion mechanism related to the effect of chloride ion concentrations on the corrosion behavior is proposed.

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### 1. Introduction

Many excellent characteristics for magnesium and its alloys, such as light weight, high strength to weight ratio, good dimensional stability and recyclability, make them more demand in automotive applications [1,2]. However, too high corrosion rate in aggressive environments has limited the application of Mg alloys in automotive industry [3–7]. In answer to this, coating technology has been considered to be an effective means of controlling and reducing corrosion rates, such as organic coatings [2], microarc oxidation (MAO) [8], high velocity oxy-fuel thermal spray [9] and physical vapor deposition [10]. Among them, MAO has been most commercially used on Mg and Mg alloys [11–13]. This is due to low cost, good adhesion, wide range of coating thickness, higher stability and corrosion resistance provided by MAO [14]. The corrosion performances of MAO coated Mg alloys are usually studied in NaCl solutions for industrial applications [15–18]. Corrosion damage of Mg alloys is greatly affected by Cl<sup>-</sup> level in NaCl solutions [19–21]. The influence of chloride concentration on the corrosion behavior of MAO coated AM50 has been studied [22]. However, for MAO coated AZ31 alloys, little has been reported on the effect of Cl<sup>-</sup> level on the corrosion performance of them in NaCl solu-

tions. In this study, MAO coated AZ31 Mg alloys are immersed in NaCl solutions for 7 d. Effects of various Cl<sup>-</sup> concentrations on corrosion rate and electrochemical impedance are evaluated to understand the corrosion mechanism.

### 2. Experimental details

The chemical compositions of the AZ31 alloy used in this study are as follows (in wt.%): Al 2.9, Zn 1.1, Mn 0.38 and Mg balance. The samples for MAO coating were machined to the dimensions of 20 mm × 20 mm × 1 mm. Prior to the MAO treatment, the samples were polished with various grades SiC abrasive papers (180, 280, 360, 600 up to 1000 grit). After polishing the samples, they were rinsed in deionized water for 1 min, dehydrated in ethyl alcohol for 2 min, and then dried immediately in flowing air.

The MAO coating was prepared in a MAO20 equipment (Chengdu PULSETECH Electrical Co., China). The equipment includes an adjustable DC pulse source up to 50 kW, a stainless steel container with a sample holder, a stirring system, and a cooling system with an exhaust fan. It uses the metallic sample immersed in the electrolyte as the anode, and the bath container as the cathode. The MAO coatings were prepared at aqueous solution Na<sub>3</sub>PO<sub>4</sub> with a constant pulsed DC frequency of 3000 Hz (duty cycle 0.3) for 5 min at 30–40 °C. The MAO coating samples used in this study are shown in Fig. 1.

The coated samples were immersed in 1000 ml NaCl solutions with mass ion concentrations of 0.5%, 2%, 3.5%, 5%, 6.5% and 8% for 7 d. Potentiodynamic polarization (PP) and electrochemical

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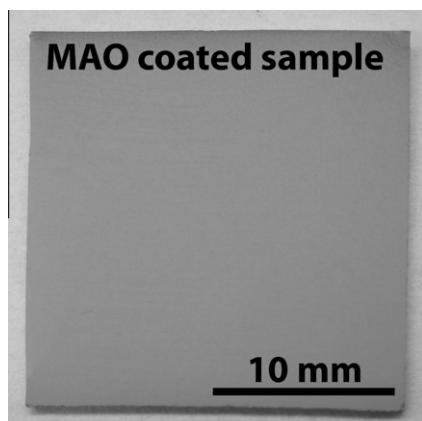


Fig. 1. Sample appearance of the MAO coating.

impedance spectroscopy (EIS) tests were carried out to evaluate the corrosion behavior of the MAO coated Mg alloys. A conventional three-electrode cell is employed in this study. In order to reach a stable potential (all potentials are given versus SCE), the open circuit potential (OCP) was measured for 10 min before the PP and EIS curves were collected. These data were recorded at various intervals of 0.5 h and 7 d. The polarization curve was measured at a scanning rate of 0.5 mV/s. The EIS tests were performed in the frequency ranging from 0.01 Hz to 100 kHz with potential amplitude of 10 mV.

The main phases in the MAO coating were detected using X-ray diffraction (XRD) experiment, in which the angle of the incident beam was fixed at  $2.5^\circ$  against the sample surface. The XRD profiles were recorded using  $\text{Co K}\alpha$  radiation at 40 kV and 45 mA. A SEM (Netherlands Quanta200) was used to examine the surface and the cross section morphologies of the MAO coatings. The changes of surface micrographs were observed by an Olympus BX60 optical microscope. The surface macro morphologies were taken by a digital camera after the total immersion in the NaCl solutions.

### 3. Results and discussion

#### 3.1. Phase and microstructure

The XRD spectrum of the MAO coating is shown in Fig. 2. The coating was mainly constituted of  $\text{Mg}_{17}\text{Al}_{12}$ , MgO,  $\text{Al}_2\text{O}_3$ ,  $\text{MgAl}_2\text{O}_4$

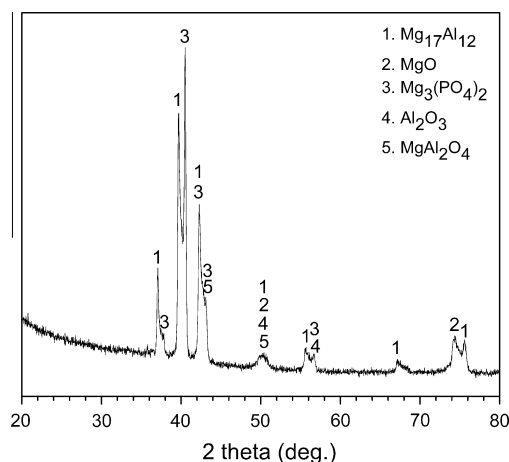


Fig. 2. XRD spectrum of the MAO coating.

and  $\text{Mg}_3(\text{PO}_4)_2$ , similar to Chang's findings on MAO coated AZ91D [23].

The SEM images shown in Fig. 3 revealed the surface and cross-sectional morphologies of the MAO coating. The micro pores and the micro cracks (Fig. 3a) are observed in the MAO coating. Good adhesion between the coating and the substrate is seen without any visible boundary from the cross-sectional morphology (Fig. 3b), which is due to the fact MAO coating is grown to the substrate by chemical conversion [24].

#### 3.2. Potentiodynamic polarization

The polarization curves for MAO coatings after immersion in NaCl solution with various ion concentrations for 7 d are shown in Fig. 4, and the corresponding Tafel data are shown in Tables 1 and 2. The corrosion potential shifted to the negative direction when the samples are immersed in 0.5%, 2%, 3.5%, 5%, 6.5% and 8% NaCl solutions irrespective of immersion time. This suggests the sample is less noble under higher chloride ion concentrations.

The corrosion current density increased with the increase of the immersion time from 0.5 h to 7 d, without regard to the chloride ion concentrations. This indicates that the corrosion rate increased with the increase of the immersion time. The corrosion current density also increased with increasing the chloride ion concentrations, consistent with the results from various authors [20,25,26]. However, the magnitude of corrosion current density seemed to be very different from each other. This may be due to the difference in the type of magnesium alloy, immersion time and PH value of NaCl solutions. More importantly, potentiodynamic polarization to some extent can only determine the corrosion rate qualitatively; however, it is not precise in quantitative analysis due to the error in the selection of linear region of Tafel plots.

Additionally, from the corrosion current density ( $I_{\text{corr}}$ ) enlarged ratio shown in Table 2, it can be observed that the ratio increased with increasing ion concentration irrespective of immersion time. However, it is noticed that the ratio increased significantly when ion concentration is more than 5%. The ratios reached 42.39% and 50.35% when ion concentrations are 6.5% and 8% after 7-day immersion. This indicates that the MAO coated AZ31 alloys deteriorated much more seriously when chloride ion concentration is greater than 5% compared to lower ion levels.

#### 3.3. Electrochemical impedance spectroscopy

EIS measurements were used to characterize the corrosion deterioration of MAO coated AZ31 Mg alloy in various ion concentrated NaCl solutions with immersion time up to 7 d. The EIS plots of MAO coatings are shown in Fig. 5. The Nyquist and Bode plots of MAO coated specimens after 0.5 h immersion are presented in Fig. 5a and b, respectively. It can be seen that at initial 0.5 h, the global impedance of the samples is very high and very close to each other when immersed in 0.5%, 2%, 3.5%, 5%, 6.5% and 8% NaCl solutions. This suggests no diffusion of electrolyte through the pores within the short immersion period. After 7-day immersion, the Nyquist and Bode plots of MAO coated specimens exhibited lower global impedance than 0.5 h immersion condition, irrespective of ion concentrations. This is due to more chemical reactions between the NaCl solution and Mg alloys with longer immersion time.

#### 3.4. Characterization of corroded surfaces

The optical surface micrographs of MAO coatings taken upon immersion in the NaCl solution for 0.5 h and 7 d are shown in

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