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ARTICLE

# Effect of Growth Rate on Microstructure and Corrosion Resistance of Micro-arc Oxidation Coatings on Magnesium Alloy

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**Abstract:** AZ91D magnesium alloys were processed by micro-arc oxidation (MAO) in silicate-containing electrolyte. The key factor of this research is a unique experimental design to fabricate coatings with the same thickness but by different power voltages which represents different growth rate. The coatings with the same thickness correspond to different coating growth rates of 1, 5, 15 and 25 μm/min, which makes the comparison and analysis-targeted microstructure and corrosion resistance of the coatings academic and practical. The coating growth rate demonstrates considerable influence on surface porosity, size and amount of micro-pores, mass and mass to thickness ratio, and anti-corrosion property of the coatings based on both qualitative and quantitative analysis, but it is not the case for the composition and element distribution of the coatings. An industrial application-oriented selection of appropriate coating growth rate, which demands for both productive efficiency and good performance, has to be considered together with anti-corrosion property of the coatings. The case of the coating fabricated with the growth rate of 15 μm/min supports this point strongly.

**Key words:** magnesium alloy; micro-arc oxidation; coating growth rate; microstructure; corrosion resistance

A surface treatment technique, named micro-arc oxidation (MAO) or plasma electrolytic oxidation (PEO)，is usually used to produce a protective coating with ceramic phases on the surface of magnesium, aluminium and titanium alloys[1-3]. Compared with other existing surface treatment techniques, MAO treatment has many advantages, such as simple pre-treatment and processing, environmental friendly electrolytes and formation of relatively thick coatings. Much research on composition, microstructure and performance of the MAO coatings has been focused on magnesium alloys in recent years[4-8].

The formation of MAO coatings on magnesium alloys is complex because of existence of the plasma-chemical, electrochemical reaction and anodic oxidation processes during micro-arc oxidation[9-11]. So influences of the process parameters, such as power voltage, current density, and composition and concentration of the electrolyte on the coating performance have attracted attention from many researchers[12-17]. However, very little research was focused on the effect of the coating growth rate on the microstructure and performance of MAO coatings. And in fact, the coating growth rate exhibited considerable influence during industrial applications of MAO coating fabrication.

In the present study, AZ91D magnesium alloys widely used in industrial applications were processed by micro-arc oxidation in silicate-containing electrolyte. The coatings with the same thickness but corresponding to different growth rates were prepared, while the coating thicknesses were different in published literatures as mentioned above when discussing the coating growth rate. The influence of the coating growth rate on coatings' characteristics, such as surface porosity, size and amount of micro-pores and corrosion resistance of the coatings were investigated quantitatively. In addition, an industrial application-oriented selection of appropriate coating growth rate, which demands for both productive efficiency and good

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performance, had to be considered together with anti-corrosion property, or other desired properties, of the coatings.

## 1 Experiment

The chemical composition of AZ91D magnesium alloy used for substrate material in this study are as follows (wt%): Al 8.3~9.7, Zn 0.35~1.0, Mn 0.17~0.27, Si 0.1, Cu＜0.03, Ni＜0.002, Fe＜0.005, Mg balance. The specimens were machined to the rectangular pieces, with the dimensions of 30 mm×20 mm×10 mm. In order to remove the oxide skin formed during machining, the surface of the specimens was ground on 400 grit silicon carbide paper. And then the specimens were rinsed in water and dried in warm air before MAO process.

According to the results of earlier experiments, the MAO coatings with the same thickness but different growth rates (as shown in Table 1) were prepared, using a home-made power supply by controlling the power voltage and treatment time. The silicate electrolyte prepared with high purity chemicals and deionised water was continuously stirred and cooled to ensure the temperature in the range of 20~40 °C during the MAO treatment. The specimens connected with an aluminium wire of 3 mm in diameter were used as anode and a piece of stainless steel plate was employed as cathode.

Surface morphologies of PEO coatings were researched by a JSM-6700F scanning electron microscopy (SEM) and cross-section morphologies of coatings were detected by a JSM-5600LV scanning electron microscopy. The size and amount of micro-pores on coating surface were counted quantitatively by Image J software. Phases of coatings were investigated by X-ray diffraction (XRD), employing a Ricoh D/MAX-2400 instrument with a scan range from 10° to 80° (in $2\theta$), a step length of 0.02° and a Cu Kα radiation source. An EPMA-1600 electron probe micro-analyzer was used to analyze the distribution of key elements on the cross-section of coatings. The mass of specimens before and after PEO treatment was measured by a BP211D analytical balance. A TT260 coating thickness gauge was used to measure the thickness of PEO coatings and the mean of 10 measured values was calculated and used as the thickness of PEO coatings to ensure accuracy of the results.

A CHI660C electrochemical workstation and a three- electrode cell were used to obtain potentiodynamic polarization curves of the bare metal and the MAO coated samples corresponding to different coating growth rates. The specimens were used as the working electrode, accompanied with a platinum counter electrode and a saturated calomel reference electrode (SCE). After the specimens with an exposed surface area of 1 $cm^2$ were immersed in 3.5wt% NaCl solution for 30 min at room temperature, the potentiodynamic polarization test proceeded at a scan speed of 5 $mV \cdot s^{-1}$ and a scan range of –1.8 to –1.3 V.

**Table 1 Growth rate and thickness of MAO coatings on different specimens**

| Specimen | Growth rate/μm·$min^{-1}$ | Coating thickness/μm |
|---|---|---|
| 1# | 1 | 25 |
| 2# | 5 | 25 |
| 3# | 15 | 25 |
| 4# | 25 | 25 |

## 2 Results and Discussion

### 2.1 Coating morphologies and micro-pores on the coating surface

Fig.1 shows surface morphologies and distributions of the micro-pores on the surface of the coatings with the same thickness but corresponding to different growth rates. Surface morphologies reveal obvious qualitative changes in the shape and size of the micro-pores when the growth rate of the coating is increased. For the coating with the growth rate of 1 μm/min, micro-pores are small and some of them are close to each other and even connect together. Increasing the coating growth rate, the micro-pores grow constantly, accompanied with a rise in the

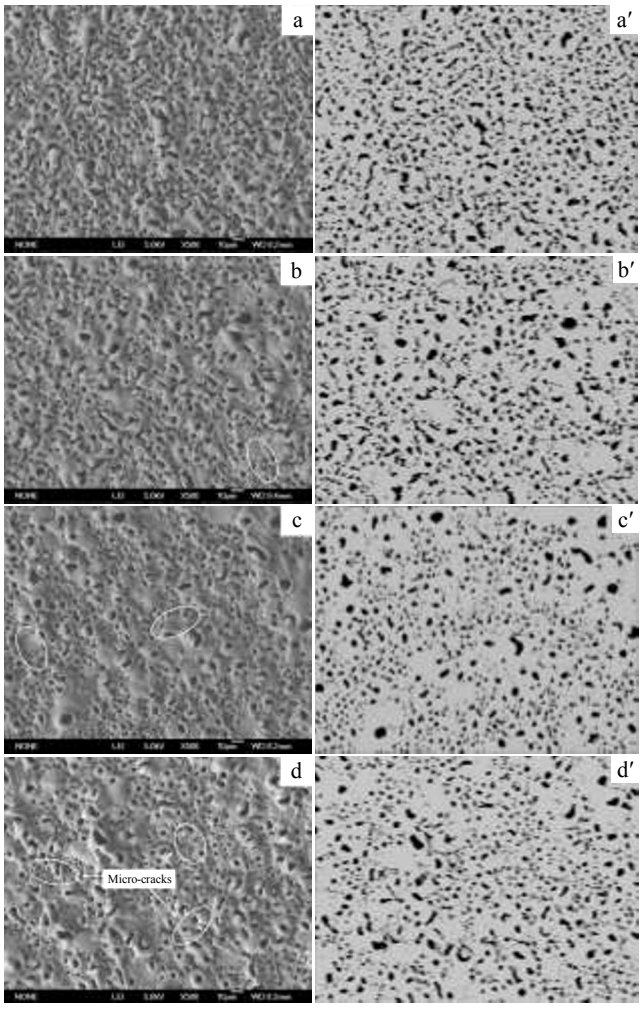


Fig.1 Surface morphologies and distributions of micro-pores on coatings with different growth rates of 1 μm/min (a, a′), 5 μm/min (b, b′), 15 μm/min (c, c′), and 25 μm/min (d, d′)