



## Review

## Research of growth mechanism of ceramic coatings fabricated by micro-arc oxidation on magnesium alloys at high current mode

Wei-wei Chen, Ze-xin Wang, Lei Sun, Sheng Lu \*

*School of Materials Science and Engineering, Jiangsu University of Science and Technology, Zhenjiang, 212003 Jiangsu, China*

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**Abstract**

Micro-arc oxidation (MAO) coatings of ZK60 magnesium alloys were formed in a self-developed dual electrolyte composed of sodium silicate and phosphate at the high constant current of 1.8 A (15 A/dm<sup>2</sup>). The MAO process and growth mechanism were investigated by scanning electron microscopy (SEM) coupled with an energy dispersive spectrometer (EDS), confocal laser scanning microscopy and X-ray diffraction (XRD). The results indicate that the growth process of MAO coating mainly goes through “forming → puncturing → rapid growth of micro-arc oxidation → large arc discharge → self-repairing”. The coating grows inward and outward at the same time in the initial stage, but outward growth of the coating is dominant later. Mg, Mg<sub>2</sub>SiO<sub>4</sub> and MgO are the main phases of ceramic coating.

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*Keywords:* ZK60 magnesium alloys; Micro-arc oxidation; High current; Growth mechanism

**1. Introduction**

Magnesium alloys have been paid enough attention to for many years due to the resource, performance, and price advantage, called as “the green structural materials in the 21st century”. However, the application potential and range are both limited by their susceptibility to corrosion and inferior wear performance. Therefore, the proper surface modification treatment can enhance the corrosion resistance of the existing magnesium alloy which has important practical significance [1,2].

Micro-arc oxidization (MAO) is a simple, friendly, efficient surface treatment method of forming ceramic coatings on Al, Ti, Mg and their alloys. The surface properties, such as wear and corrosion resistance, electrical insulation, adhesion to substrate, can be considerably improved by MAO [3–5]. It is an incontrovertible fact that the growth mechanism of MAO process is extremely complex, including chemical, electrochemical, high temperature plasma reaction, etc. And no reasonable model can fully and precisely describe the theory at home and abroad. Chen et al. [6,7] thought the process is a submonolayer growth, which is a cyclic process experiencing

the repeated course of “forming → breakdown → melting → liquating → sintering → reforming”. Cellular structure composed of nanoparticles is growing layer-by-layer, whose dense layer is made up of MgO mainly. Ge et al. [8] studied the MAO process of wrought magnesium alloy in the silicate system. They found that dense layer is formed in the preliminary stage, and loose layer appears later, accounting for a large percentage of the total film thickness. They also carried out quantitative analysis and measurement of the energy loss and impedance change of membrane layer in reaction. The researchers of Northwestern Polytechnical University [9–11] studied the effect from rare earth elements on the growth of membrane layer. They thought the MAO film is a double-layer structure in silicate system. The dense layer is mainly composed of MgO, and the loose layer is mainly composed of MgSiO<sub>3</sub>.

Our group has completed the research on growth mechanism at low-current earlier [12]. In this paper, combined with the testing results, the authors aim to make a further exploration at the high current in order to form a complete system on MAO process and growth mechanism at constant current mode.

**2. Experimental**

The substrate material used for investigation was wrought ZK60 magnesium alloy with a chemical composition (Zn 4.8–6.2%, Zr > 0.45%, impurities ≤ 0.30%, Mg balance). The samples with a size of 20 mm × 20 mm × 5 mm were successively ground to 1600 grit silicon carbide papers, cleaned

\* Corresponding author. School of Materials Science and Engineering, Jiangsu University of Science and Technology, Zhenjiang, 212003 Jiangsu, China. Tel.: +86 15751012979.

E-mail address: [lusheng88168@qq.com](mailto:lusheng88168@qq.com) (S. Lu).

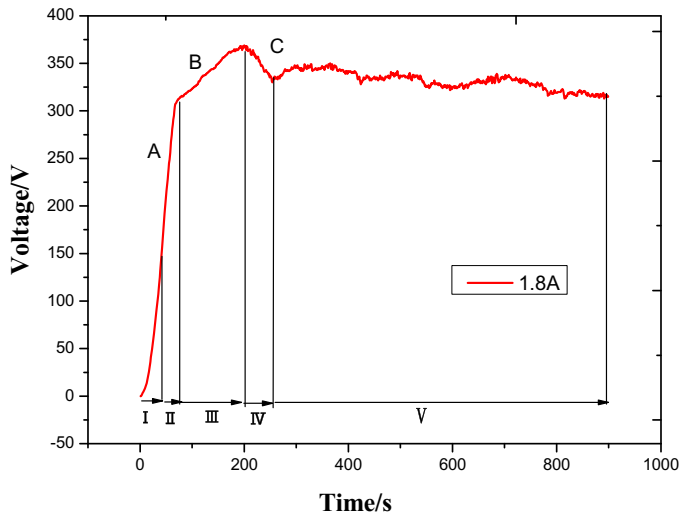


Fig. 1. Voltage-time curve at the current of 1.8 A.

by alcohol, and then dried in cold air. WHD-20 MAO system was employed for MAO treatment. The samples acted as anodes and a stainless steel container acted as cathode. The experiments were carried out in an optimized dual electrolyte composed of sodium silicate and phosphate under constant current. The temperature was kept in electrolyte under 40°C through circulating water cooling system.

The micro-structural characteristics of coating, coating elements and phase constituent were investigated by scanning electron microscopy (SEM, JSM-6480), X-ray diffraction (XRD, Shimadzu XRD-6000) and Olympus confocal laser scanning microscopy (OLS4000). The micrometer was used to measure the thickness of the specimen before and after the MAO process. The MAO film thickness was measured by the coating thickness measurement equipment (OXFORD CMI233).

### 3. Results and discussion

#### 3.1. Analysis of voltage-time curve and MAO spark discharge phenomenon

The MAO coating growth process is monitored by the voltage-time curve, which can be divided into five distinguishing stages as shown in Fig. 1. At the first stage, within 0–40 s, the substrate surface quickly formed a very thin layer, named anodic oxidation stage. The substrate surface appeared the first spark (Fig. 2(a)), which meant that the MAO process reached the striking voltage about 140 V at around 40 s. The second stage lasted from 40 s to 70 s; the anodic oxidation coating got punctured and MAO coating was formed. The third stage (70 s–200 s) corresponded to the main period of rapid growth of MAO, involving large numbers of white and bright sparks (Fig. 2(b)), as a result of highlight plasma micro-arc discharge. The value of voltage reached a maximum of 370 V at about 200 s. In this stage, the porous film solidified rapidly because of the effect of high-temperature micro zone and “quenching” in the electrolyte. The thickness of coating increased by 25 μm and the value of growth rate got to a maximum of 13.15 μm/

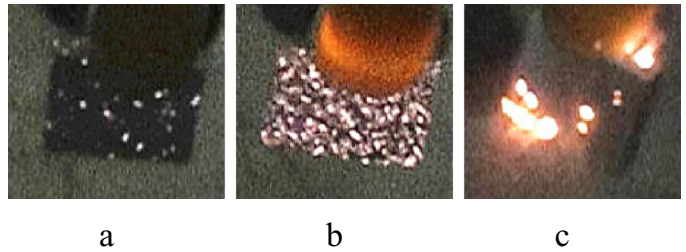


Fig. 2. Spark discharge characteristics at different time points.

min. After 200 s, plasma point discharge concentrated upon the places of edges or corners by the effects of high voltage and strong electric field, which led to the breakdown, and even dissolution and desquamation of part of the membrane layer. Compared with the second stage and third stage, the growth rate decreases significantly after 200 s. The voltage curve of the fourth stage (200–250 s) presents a brief drop; the voltage value drops slightly. Through macroscopic observation, the number of sparks reduced, but single spark became larger. So this stage can be named local large arc discharge (Fig. 2(c)). The last stage was characterized by a voltage plateau from 250 s to the end in which many micro-cracks and micro-pores were disappeared gradually and the surface also became smooth. According to the macroscopic experimental phenomena and microstructure, this stage played a key role to control the quality of the film through self-repairing.

#### 3.2. Analysis of microstructure

The surface and cross-section micro-morphologies of MAO coating at different stage are shown in Fig. 3. At the time of 60 s, passive film was punctured and large numbers of micro discharge channels appeared on the surface (Fig. 3(a)). When oxidation time lasted for 180 s, the variations of surface morphologies show normal regularity that the quantity of the micro discharge channels decreased but the size of micro-pores increased sharply. Most of the pore diameters are in the range of 10 to 15 μm. In addition, continuous micro-cracks and loose micro-pores both existed in the surface and cross-section of coating due to the effect of quenching stress (Fig. 3(b) and Fig. 3(e)). At the low-voltage self-repairing stage of 480 s, the number of micro-pores decreased to some degree because plenty of melt accumulated on the surface and covered parts of discharge channels as seen in Fig. 3(c). The average pore diameter decreased to 4.3 μm. At last, continuous micro-cracks of the coating disappeared gradually or transformed into discontinuous micro-cracks, and cross-section morphologies of the coating became smooth and dense (Fig. 3(d) and (g)). The average thickness of the coating reached 65 μm.

#### 3.3. Analysis of growth characteristic

For growth characteristic of MAO coating by high-current mode, Fig. 4 shows the dynamic changing process of coating thickness in two sides. It can be found that the growth rate of the two sides is basically synchronous without huge difference. The growth rate is nearly close to 0 before 70 s, namely, only an

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