



Influence of 8-hydroxyquinoline on properties of anodic coatings obtained by micro arc oxidation on AZ91 magnesium alloys

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ABSTRACT

The influence of 8-hydroxyquinoline (8-HQ) on formation and properties of anodic coatings obtained by micro arc oxidation (MAO) on AZ91 magnesium alloys was studied by scanning electron microscope (SEM), energy dispersive spectrometry (EDS), Fourier transform infrared (FT-IR) spectroscopy and potentiodynamic polarization tests. The results demonstrate that 8-HQ can decrease the solution conductivity, take part in the coating formation and change the coating color. By developing anodic coatings with increasing thickness, insoluble Mg(HQ)₂ and small pore size, 8-HQ improves the corrosion resistance of the anodized magnesium alloys. The coating shows the best corrosion resistance in the solution of 10 g/L NaOH and 18 g/L Na₂SiO₃ with 2 g/L 8-HQ.

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1. Introduction

Micro arc oxidation (MAO), developed under the traditional oxidation, is an effective method to improve the corrosion and wear resistance of magnesium alloys. The coating properties depend on several factors, such as the composition of the substrate [1,2], electric parameters [2,3], the concentrations and compositions of the electrolytes [2,4–6]. Among these factors, the used electrolytes play an important role in determining the coating property. Due to the health and environmental pressure, some environmentally friendly processes have been developed in alkaline solutions mainly containing inorganic electrolytes such as silicate [1,4–17], aluminate [2,5,6] and borate [4,5,7,11,15,18]. At present, some organic additives, for example, triethanolamine [4], glycerol [9,10,12], benzotriazole [11], surfactants [12], ethylene glycol [13,14], citrate [15,18,19], sodium acetate [20] and phytic acid [21] have been used in MAO on magnesium alloys. 8-Hydroxyquinoline (8-HQ), one of the nitrogen-heterocyclic aromatic hydrocarbons with moderate toxicity in rats (oral LD50 1200 mg/kg) [22], is widely used in many areas. For instance, 8-HQ is used as a dye [23], a masking agent [24], a metal inhibitor toward the corrosion of steel [25], copper [26], aluminum [27] and magnesium [28,29]. The inhibitory mechanism of 8-HQ has been investigated and the results show that the formation of complex chelating compounds

such as Cu(HQ)₂, Al(HQ)₃ and Mg(HQ)₂ on metal surface plays an essential role in the inhibition of metal corrosion [26–29]. Inhibiting action is also related to the adsorption of 8-HQ molecules on the surface, avoiding the adsorption of aggressive ions, such as Cl⁻ or OH⁻ [25,26,29]. In view of insolubility and stability of Mg(HQ)₂ in water solution, it is speculated that 8-HQ may be helpful for improving the coating property obtained by MAO on magnesium alloys. However, to the best of our knowledge, there is no report on this area. In this paper, the influence of 8-HQ on formation and property of anodic coatings was systematically studied.

2. Experimental

An AZ91 magnesium alloy ingot with a nominal composition of 9.0 wt.% Al, 1.0 wt.% Zn and balance Mg was chosen as the substrate. Samples for MAO treatment were masked with sealant leaving an area of 5 cm × 6 cm exposed, with a 3 mm diameter hole drilled for connection with the anode by a screw. The equipment for MAO consisted of a MAOI-50C power supply (Chengdu Pulsetech Electrical Co., Ltd, China), a stainless steel barrel and a stirring and cooling system that controlled the solution temperature below 40 °C. In a base solution of 10 g/L NaOH, different concentrations of 8-HQ and 18 g/L Na₂SiO₃ were added to form five solutions and they were separately called as 5Q, 18Si, 2Q-18Si, 5Q-18Si and 8Q-18Si. Electrolyte compositions and operating parameters are listed in Table 1.

Chemical reagents were weighed by BP211D electronic scales (precision 0.01 mg, Sartorius, Germany) and the solution conductivity was measured by a DDS-307W Microprocessor Conductivity Meter. Surface and cross-sectional morphologies of the anodized samples were observed by a SIGMA field emission scanning electron microscope (FE-SEM) after they were rinsed with distilled water, dried in a cool air stream and coated with gold. Chemical compositions of anodic films were determined by energy dispersive spectrometry (EDS) in the SEM. The coating structure was analyzed using a D8 advance X-ray diffractometer with Cu K α radiation. Anodic coatings obtained by MAO were scrapped off from magnesium alloys with a razor [30] and were characterized by a Nicolet 460 Fourier transform

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Table 1
Electrolyte constituents and operating conditions for MAO on magnesium alloys.

Solution	Electrolyte constituents	Operating conditions
5Q	10 g/L NaOH and 5 g/L 8-HQ	Temperature 15–40 °C, current density 40 mA/cm ² , frequency 2000 Hz, duty cycle 20%, anodizing time 3 min
18Si	10 g/L NaOH and 18 g/L Na ₂ SiO ₃	
2Q-18Si	10 g/L NaOH, 18 g/L Na ₂ SiO ₃ and 2 g/L 8-HQ	
5Q-18Si	10 g/L NaOH, 18 g/L Na ₂ SiO ₃ and 5 g/L 8-HQ	
8Q-18Si	10 g/L NaOH, 18 g/L Na ₂ SiO ₃ and 8 g/L 8-HQ	

infrared (FT-IR) spectrometer in the range of 400–4000 cm⁻¹. In comparison, the original 8-HQ powder was also analyzed by KBr pellet technique. Potentiodynamic polarization tests were measured in 3.5 wt.% NaCl solution using a CHI760C Electrochemical Workstation. A classical three-electrode cell was used with platinum as the counter electrode and a saturated calomel electrode as the reference electrode. The quiet time was 1200 s and scan was conducted with a constant rate of 0.001 V/s from initial potential of -1.8 V vs. SCE towards more noble direction until the film breakdown occurred.

3. Results

3.1. Effects of 8-HQ on electrolyte conductivity

The conductivity of aqueous solutions measured in different solutions at 14 °C is shown in Fig. 1.

The conductivity (*k*) of 10 g/L NaOH is 47.7 mS/cm. After 5 g/L 8-HQ and 18 g/L Na₂SiO₃ were separately added into the solution above, the conductivity values became 41.5 and 55.8 mS/cm, correspondingly (Fig. 1), indicating that 8-HQ can decrease the solution conductivity, while Na₂SiO₃ can increase it. After 2, 5 and 8 g/L 8-HQ were added into the solution of 10 g/L NaOH and 18 g/L Na₂SiO₃ (18Si), the conductivity values continually decreased and reached 53.7, 51.4 and 48.7 mS/cm, respectively (Fig. 1). As one kind of amphiprotic compounds, an 8-HQ molecule is ionized into an H⁺ and a C₉H₆NO⁻ in an alkaline solution. After 8-HQ is added into the alkaline solution, H⁺ ions from 8-HQ will react with OH⁻ ions from the alkaline solution to produce water. The ion conductivity of OH⁻ is much larger than that of water molecules. After OH⁻ ions are counteracted into water, the solution conductivity decreases. With the increase of 8-HQ concentration, more OH⁻ ions are consumed resulting in the continual decrease of the solution conductivity.

3.2. Effects of 8-HQ on coating formation

Fig. 2 shows voltage variations versus time during MAO treatment in 5Q, 18Si and 5Q-18Si.

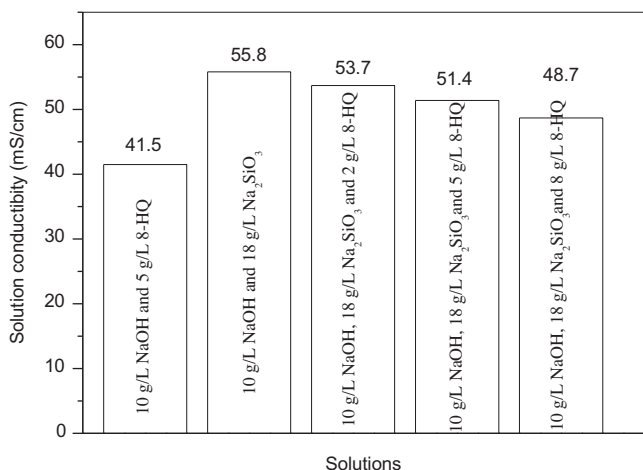


Fig. 1. The solution conductivity measured at 14 °C in different solutions.

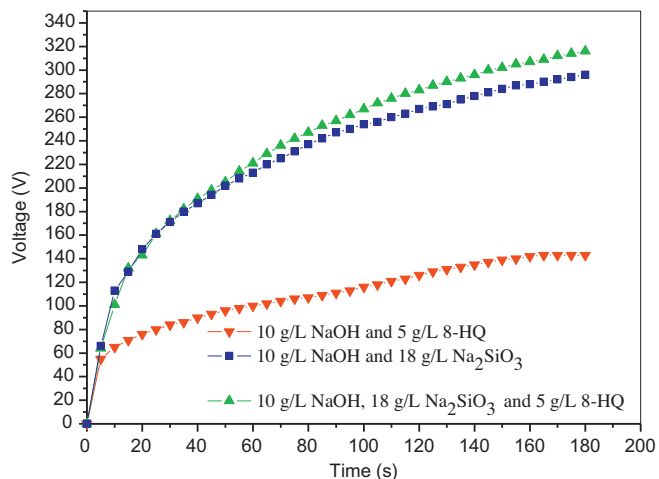


Fig. 2. Variations of voltage with time during MAO treatment in the solutions of 5Q:18Si and 5Q-18Si under current density 40 mA/cm², frequency 2000 Hz, duty cycle 20% and anodizing time 3 min.

During the first 10 s, the voltage increases rapidly with time in three solutions. After 10 s, the curve slope of voltage with time in 18Si is slightly smaller than that in 5Q-18Si but much larger than that in 5Q. After anodizing for 3 min, the final voltages treated in 5Q, 18Si and 5Q-18Si are separately 143 V, 296 V and 316 V.

The MAO process is a competition between the destruction of old film and development of new film and the variation of voltage with time during the process can indicate which step dominates the process [5,7,31]. If the voltage increases with treatment time, the process is dominated by the coating formation, otherwise it is determined by the old film destruction [5,7,31]. In addition, the curve slope of voltage with time can indicate the speed of coating formation. In generally, the larger the curve slope of working voltage with time is, the faster the coating is formed on the substrate [15]. Fig. 2 indicates that 8-HQ is not an effective electrolyte as sodium silicate but can play a role in promoting coating formation, which is further verified by the appearance of the anodized specimens.

The pictures of the anodized specimens obtained in three solutions are shown in Fig. 3.

As shown in Fig. 3, after MAO treatment of magnesium alloys in 5Q, a discontinuous film is developed on the sample surface (Fig. 3a), which indicates that 8-HQ is not an effective coating agent. After treatment in 18Si, a concrete grey film is developed on magnesium alloys (Fig. 3b) and it becomes light blue after addition of 5 g/L 8-HQ (Fig. 3c). In the light of our previous work under the same MAO conditions as used in the paper, where we found that anodic coatings could not be developed in a solution only containing NaOH [21], it is concluded that 8-HQ can take part in the coating formation and change the coating color.

3.3. Effects of 8-HQ on morphology, composition and structure

Surface morphologies of anodic coatings obtained in 5Q at two kinds of magnification are shown in Fig. 4.

After treatment in 5Q, the obtained coating is not continuous but many white particles are formed on the substrate (Fig. 4a). At higher magnification, it is evident that leaf-like material is developed under the action of 8-HQ. As shown in Fig. 4b, chemical compositions on a planar area (without anodic coatings) such as Point A and on an area with the leaf-like material such as Point B are detected by EDS analysis. Point A contains 17.7% C (in at.%, the same below), 0.2% N, 9.1% O, 69.3% Mg, and 3.6% Al, showing

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