



Full Length Article

Growth behaviour of low-energy plasma electrolytic oxidation coatings on a magnesium alloy

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Abstract

Plasma electrolytic oxidation (PEO), a promising surface treatment method to improve the corrosion and wear resistance of magnesium and its alloys, operates at high voltages, resulting in a relatively high energy cost. To make the PEO process more economically viable, its energy efficiency needs to be improved. This study investigates the growth behaviour and microstructural characteristics of low-energy PEO coatings on an AM50 magnesium alloy in a concentrated electrolyte containing sodium tetraborate. The surface morphology of the coatings was different from typical PEO coating morphologies and a large voltage oscillation was observed during treatment. Using different characterisation techniques, and based on a micro-discharge model, a correlation was made between the voltage-time behaviour, micro-discharge characteristics and the composition and microstructure of the coated samples. The results suggest electrolyte chemistry can somewhat control discharge behaviour, which plays an important role in PEO coating growth.

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1. Introduction

In recent years, magnesium and its alloys have attracted significant interest for applications in the automotive, aerospace, communications and biomedical industries because of their special properties, which include low density, high strength-to-weight ratio, good electromagnetic, noise-reducing and thermal conductivity properties, recycling ability, and superior biocompatibility and biodegradability. However, their application is restricted by poor wear resistance and a susceptibility to corrosion [1–4].

Surface treatment is the general approach adopted to overcome these deficiencies. Several treatments have been devel-

oped, among which plasma electrolytic oxidation (PEO) has proved promising and attracted significant interest recently. During PEO, also known as micro-arc oxidation (MAO), many short-lived micro-discharges are created by dielectric breakdown of the growing oxide film, resulting in the formation of ceramic-like oxide coatings composed of species from both the electrolyte and the sample. These coatings are strongly bonded to the substrate, with good protective properties. An interesting possibility with the PEO process is the incorporation of various types of nano- and micro-sized particles that are added into the electrolyte [5].

PEO operates at high voltages, typically several hundred volts, which results in a relatively high energy cost. For example, the energy cost of PEO coatings on Al alloys can be 20–50 times more than conventional anodizing [6]. For PEO to become more economically viable, its energy efficiency needs to be improved. However, there are few studies exclusively addressing the low-energy formation of PEO coatings. Many factors can influence the PEO process and

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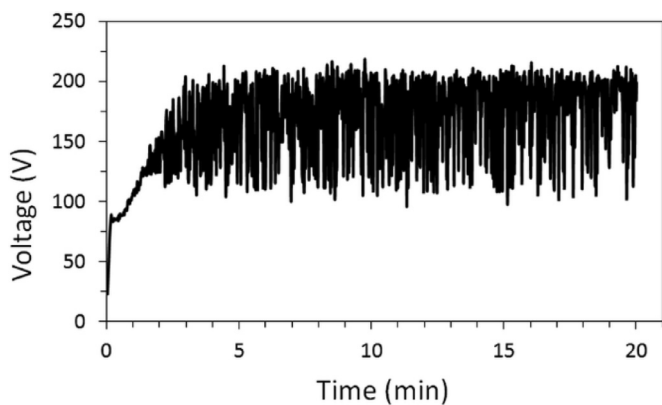


Fig. 1. Voltage-time response during PEO treatment of an AM50 Mg sample at a constant current density of 40 mA/cm².

the properties of the resulting coatings, including the electrical parameters (current density and mode), the electrolyte composition, the substrate material, and the duration of the coating process. Selecting the optimum electrical parameters, modifying the cell geometry design and electrolyte composition, and pre-anodizing to form a precursor oxide prior to PEO are strategies by which the energy consumption of the PEO could be reduced [6,7].

Electrolyte composition plays an important role in the formation of PEO coatings and in determining their properties. Many studies have investigated the effect of electrolyte additives on the properties of the coatings; however, it is difficult to compare the results of these studies, since processing conditions generally differ [8]. The most common electrolytes used for the PEO coating process on Mg alloys are based on potassium and/or sodium hydroxide with additions of phosphates and/or silicates [9–13]. Potassium fluoride [14], sodium fluoride [15,16], and sodium aluminate [8] have also been used. Few studies suggest that the addition of sodium tetraborate can improve the corrosion performance of PEO coatings. Using a concentrated electrolyte solution containing NaOH, Na₂SiO₃, and Na₂B₄O₇, Zhao et al. [17] reported that dense, protective coatings, with a thickness of 30 μm, were obtained on a Mg substrate after 17 min at a relatively low energy cost. Sreekanth et al. [18] reported that the least porosity and best corrosion resistance were obtained using a silicate-based electrolyte solution containing 2 g/L Na₂B₄O₇·10H₂O at a current density of 60 mA/cm². Unfortunately, they did not provide the voltage-time behaviour observed during coating. Ono et al. [19] reported that increasing the concentration of electrolytes containing phosphate, silicate, and aluminate resulted in a decrease in the breakdown and operating voltage of the coating process on the AZ31 Mg alloy.

Despite these endeavours, the influence of the process parameters and electrolyte chemistry on the growth of PEO coatings still needs further investigation, since its effect on the morphology, microstructure, and phase composition of the coatings is complex and inadequately understood [20]. Furthermore, most studies have focused on the influence of electrolyte composition on coating properties, such as

corrosion and wear resistance. The role of electrolyte composition in determining discharge behaviour, which significantly influences the microstructure and morphology of the coatings [21], has received little attention. In this regard, an improved fundamental understanding of the role that electrolyte composition plays in discharge phenomena and how it affects the mechanism of coatings formation is required.

The research presented in this communication is focused on an investigation of the evolution, growth behaviour, and microstructural characteristics of low-energy PEO coatings on a die-cast AM50 Mg alloy in a concentrated electrolyte solution containing sodium tetraborate. The dominant focus is on the correlation between voltage-time behaviour, micro-discharge characteristics and the composition and microstructure of the coatings produced.

2. Material and methods

Specimens of the die-cast AM50 Mg alloy with a nominal chemical composition (wt.%) of 4.5–5.3 Al, 0.28–0.5 Mn, <0.2 Zn, <0.05 Si, <0.008 Cu, <0.001 Ni, <0.004 Fe, and Mg balance, were mounted in epoxy resin and the exposed surface areas ground using emery papers up to P1200 grit. The surface was then degreased in ethanol, rinsed with deionised water and immediately dried with Ar prior to PEO.

PEO was performed in an electrolyte solution containing 20 g/L NaOH, 80 g/L Na₂SiO₃·5H₂O, and 80 g/L Na₂B₄O₇·10H₂O using a direct current power supply. All samples were coated at a constant current density of 40 mA/cm². Samples with an active area of 1 cm², and a rectangular stainless steel wire mesh (3 x 5 cm), were used as the anode and cathode, respectively. The electrolyte was constantly mixed using a magnetic stirrer and its temperature maintained at 22 ± 3 °C during the coating process. Samples were coated for different durations ranging from 1 to 20 min to study the evolution in the properties of the PEO coatings.

The morphology, chemical composition and microstructure of the coatings were analyzed using Hitachi S-4500 field emission and Hitachi SU3500 variable pressure scanning electron microscopes (SEM). Samples were sputtered with gold prior to SEM inspection. Energy dispersive X-ray spectroscopy (EDX) was conducted on a Hitachi SU3500 Variable Pressure SEM in combination with an Oxford Aztec X-Max50 X-ray analyzer. Aztec software allowed for both point analyses as well as the acquisition of EDX maps. Analysis of the phase composition of coatings was performed with an Inel CPS X-ray diffractometer using Cu K α radiation, in a glancing angle configuration with an incident angle of 4° to minimize interference from the Mg substrate.

3. Results

3.1. Voltage-time response

Fig. 1 shows the voltage-time plot obtained when a constant current density of 40 mA/cm² was applied to an AM50 Mg specimen. The voltage-time plots recorded when coating

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