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# Effects of cathodic voltages on structure and wear resistance of plasma electrolytic oxidation coatings formed on aluminium alloy

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#### ABSTRACT

Plasma electrolytic oxidation (PEO) coatings were prepared on aluminium alloy using pulsed bipolar power supply at constant anodic voltage and different cathodic voltages. The samples were prepared to attain the same coating thickness by adjusting the processing time. The scanning electron microscope (SEM), energy dispersive spectrometer (EDS), X-ray diffraction (XRD) and tribometer were employed to investigate the microstructure, element content, phase composition and wear resistance of the coatings respectively. It was found that the coating growth rate enhanced obviously and the coatings exhibited a more compact structure with thicker inner layer and lower surface roughness when the cathodic voltage increased. The coatings were mainly composed of crystalline  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> and amorphous silicate oxides and their relative content changed with the cathodic voltage. The wear resistance of the coatings improved significantly with the increase of cathodic voltage.

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#### 1. Introduction

Aluminium alloys have received much attention in various machinery and transportation system especially in aerospace and automobile products due to their advantages of high strength to weight ratio, proper corrosion resistance and lightweight. However, their shortcomings, such as low hardness, low wear resistance and difficulty to lubricate, have seriously restricted their extensive applications [1]. In the past decades, various surface treatments, such as chemical vapor deposition [2], physical vapor deposition [3], and ion beam assisted deposition [4] and thermal spraving [5], have been applied to improve their generally poor mechanical and tribological properties. However, most of the aforementioned methods involve high processing temperature, which may degrade the coatings and/or substrates [6]. Alternatively, employing plasma electrolytic oxidation (PEO) technique can produce thick and hard alumina coatings on aluminium alloys at bulk material temperatures of less than 100 °C by plasma discharge in aqueous electrolyte solutions [7]. The yielded PEO ceramic coatings have significantly improved the wear resistance of aluminium alloys [8,9].

It is well known that PEO is a multifactor-controlled process [9]. The qualities of PEO coatings are determined by many factors, such as substrate materials [10,11], electrolyte composition [12–14],

applied power modes [15,16], electrical parameters [17,18], processing temperature [19], oxidation time [20] and additives [21,22]. To obtain the desired coatings, many investigations on the influence factors of PEO process have been done in recent years and many progresses have been made in this technique especially in the applied power modes. The commonly used power modes for PEO process include direct current (DC), alternative current (AC), pulsed unipolar and bipolar current modes [23-25]. Currently, pulsed bipolar current mode has received much attention and is most commonly used. From the plasma discharge point of view. the application of pulsed bipolar current can reduce or eliminate the strongest plasma discharges and thus the high plasma temperature spikes by the significant influence of cathodic component as well as the longer off-time duration [23,24]. The PEO coatings formed using a pulsed bipolar current mode have been reported to have a more compact structure with fewer defects and uniform coating thickness, compared to the coatings formed using DC, AC or pulsed unipolar current mode [23–26].

In the pulsed bipolar power mode, the cathodic electrical parameters play an important role in the PEO process. Wu et al. [27] studied the effects of cathodic voltage on pitting corrosion performance of the PEO coatings on Ti alloy and found that employing cathodic voltages of 20–45 V could obtain porous nanocrystalline titania coatings consisting mainly of rutile phase and having high pitting corrosion resistance. Liu et al. [28] investigated the effects of cathodic voltage on friction coefficient and corrosion resistance of the PEO coated NiTi alloy and came to the conclusion that the PEO







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coated NiTi alloy exhibited a higher friction coefficient and lower corrosion resistance which showed a poorer biocompatibility with increasing the cathodic voltage. The effects of cathode current density on corrosion resistance of the PEO coatings on Mg alloy were studied by Su et al. [29]. Results showed that the increase of cathode current density made the coatings more compact and improved their corrosion resistance. However, to the best of our knowledge, the effects of cathodic voltage or current density on wear resistance of PEO coatings were not yet reported. Therefore, in this work, the effects of cathodic voltage on structure and wear resistance of PEO coatings on aluminum alloy were investigated.

#### 2. Experimental details

Rectangular coupons ( $39 \text{ mm} \times 20 \text{ mm} \times 4 \text{ mm}$ ) of 2024 Al alloy (3.8-4.9% Cu, 1.2-1.8% Mg, 0.5% Fe, 0.5% Si, 0.3% Mn, 0.25% Zn, 0.1% Ni, 0.05% Ti and balance Al) were used as the substrates. Prior to the PEO treatments, the samples were ground and polished with 320 # and 1200 # abrasive papers respectively, ultrasonically degreased in acetone and cleaned with distilled water.

The PEO processes were carried out in the electrolyte of 10 g/LNa<sub>2</sub>SiO<sub>3</sub> and 1 g/L KOH, under constant voltage mode, using 20 KW pulsed bipolar power supply, with a frequency of 150 Hz, positive pulse width of 1.0 m s and negative pulse width of 1.5 m s. The Al alloy sample was connected to the anode and a spiral stainless steel pipe was used as counter electrode as well as water cooling system. In the PEO processes, a constant anodic voltage of 520 V and different cathodic voltages of 0 V, 40 V, 90 V, 140 V and 190 V were applied. During the PEO processes, the temperature of the electrolyte was always maintained in the range of 20–30 °C by the water cooling system and constant mechanical stirring. After the PEO processes, the specimens were rinsed with distilled water and air dried. The PEO coatings prepared at different cathodic voltages of 0 V, 40 V, 90 V, 140 V and 190 V were denoted as C0, C40, C90, C140 and C190, respectively, in the following text.

To avoid the effect of coating thickness on wear resistance, the samples were prepared to attain similar coating thickness in the range of 30–33  $\mu$ m by adjusting the processing time. The thickness of the PEO coatings was measured using a MINITEST 1100 microprocessor coating thickness gauge (Elektro-Physik Koln, Germany), which utilizes eddy currents induced in the substrate to measure coating thickness with an accuracy of about  $\pm 1 \,\mu$ m. The surface roughness of the PEO coatings was measured by an RM-20 Surface Roughness Tester (TIME Group Inc. China).

A JSM-5600LV scanning electron microscope (SEM) was employed to observe the surface and cross-sectional morphologies of the PEO coatings. The cross sections for SEM observation were prepared by embedding the PEO coated Al alloys in epoxy resin and careful polishing after its solidification. The element content of the coatings was studied by energy dispersive spectrometer (EDS). The phase composition of the coatings was analyzed by X-ray diffraction (XRD, Rigaku D/Max-2400 X-Ray Diffractometer), using Cu-K $\alpha$ radiation as the excitation source, with a grazing angle of 5°.

The friction and wear properties of the PEO coatings were evaluated on a reciprocating ball-on-disk UMT-2MT tribometer (CETR, USA). Si<sub>3</sub>N<sub>4</sub> ceramic balls with a diameter of 3 mm were used as counterpart materials. All the friction and wear tests were performed for 30 min, using a frequency of 5 Hz and an oscillating amplitude of 5 mm, at ambient temperature and humidity (relative humidity is about 30%).

#### 3. Results and discussion

Fig. 1 shows the processing time for the PEO coatings with similar thickness deposited at different cathodic voltages. The thickness

Fig. 1. The processing time for the PEO coatings with similar thickness prepared at different cathodic voltages.

of the coatings C0, C40, C90, C140 and C190 was almost the same and in the range of  $30-33 \,\mu$ m, while their respective processing time was 65 min, 60 min, 35 min, 25 min and 18 min. Therefore, the processing time for the PEO coatings with similar thickness decreased when the applied cathodic voltage increased, indicating that the coating growth rate sharply increased with the increase of cathodic voltage.

The anodic and cathodic current density transient curves of the PEO coatings fabricated at different cathodic voltages are presented in Fig. 2. It can be seen from Fig. 2(a) that all the PEO processes started from the same anodic current density of about 26.9 A/dm<sup>2</sup> and their transient curves nearly overlapped together under the same anodic voltage of 520V and different cathodic voltages of 0-190 V. It also shows that the anodic current density in the PEO processes decreased with the processing time increasing. Moreover, the decrease of the anodic current density in the early stage of the processes was much quicker than that of the later stage. It is well known that the PEO coatings deposited on Al alloys had poor electrical conductivity and their resistance enhanced when the coatings grew thicker, which resulted in the decrease of anodic current density under the same anodic voltage. The decreasing speed of the current density had strong correlation to the coating growth rate. Therefore, it can be deduced that the coating growth rate was higher in the early stage of the PEO processes and decreased with the increase of processing time.

Fig. 2(b) shows that the cathodic current density in the PEO processes increased when the applied cathodic voltage increased. There was no cathodic current density under the cathodic voltage of 0 V. With the applied cathodic voltages of 40–190 V, all the cathodic current density decreased with the processing time increasing. It can also be seen that the higher cathodic voltage was applied, the quicker decrease of the cathodic current density was recorded. The decreasing characteristic of the cathodic current density suggested that the coating growth rate was higher employing higher cathodic voltage which was consistent with the result of Fig. 1.

Fig. 3 displays the surface morphologies of PEO coatings prepared under different cathodic voltages from 0V to 190V. All the samples showed the typical PEO porous structures with many micropores and microcracks. It was thought that the micropores were formed by molten oxide and gas bubbles thrown out of discharge channels and the microcracks were resulted from the thermal stress due to the rapid solidification of molten oxide in the relatively cold electrolyte [9,30]. Three types of micropores could be clearly identified on the surface of the PEO coatings, which was consistent with the previous report [31]. Two of them (region A and



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