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Effect of current step-down on the growth and hardness of PEO coatings on Al6061 alloy

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Abstract

In this study, plasma electrolytic oxidation (PEO) treatments were conducted on Al6061 plates using silicate containing alkaline electrolyte. The hardness variation with the growth of the PEO coating was investigated under two different regimes of applied AC current. The first was a continuous supply with a constant current density of 500 mA/cm² for 60 or 90min. The other was a current supply of 500mA/cm² for 60 min followed by a current step-down to 200 mA/cm² and continuous treatment for 30 min. In the case of a constant current for 60 min, the coating thickness and Vickers hardness increased to 150 μm and 1300 Hv, respectively. With further processing in the same manner, the coating grew thicker while its hardness gradually decreased to 900 Hv. In the case of a current step-down, no significant thickness increase was observed, but there was a hardness increase up to 1800 Hv. Analysis of the PEO coatings by SEM/EDS and XRD revealed that all their function layers were comprised mainly of α-Al₂O₃ with hardness dependent on density. The current step-down method contributes to the densification of the function layer in a PEO coating, and thus increases the hardness.

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Keywords: plasma electrolytic oxidation; Al6061; function layer; hardness

1. Introduction

Plasma electrolytic oxidation (PEO) is an anodic oxidation method in which plasma or arc discharge in an electrolyte under a high electric field is employed for the surface finishing of aluminium and its alloys

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[1]. PEO yields excellent mechanical properties, such as hardness and wear resistance of the surface, in comparison with other conventional anodizing methods. It has been reported that the Vickers hardness values of PEO coatings on various Al alloys can reach 900-2000 Hv [2]. This dramatically high hardness is basically attributable to the presence of the α - Al_2O_3 phase, but is variously influenced by processing factors. Therefore, many studies on the properties of PEO coatings have focused on the effects of current conditions, temperature, substrate composition, and electrolyte constituents [3]. With respect to current conditions, not only the densities supplied in processes are considered important but also the waveforms. In early developments, a constant supply of AC current was generally used, whereas various types of current including DC, squarewave AC, and pulse bipolar are presently in use. Furthermore, a type of multi-step process has been reported. Matykina et al. have shown that PEO after pre-anodizing, a conventional porous anodizing treatment, can achieve a maximum hardness almost 2.5 times higher than that without such treatment [4]. In addition, there are benefits in terms of coating growth and energy consumption.

In this study, a new modified-current regime, with a current step-down after the high current process, was applied to PEO treatment of Al6061 alloy. The influences on the growth and mechanical properties of the coating were investigated.

2. Experimental

Al6061 alloy sheets, with a thickness of 2 mm and exposed area of 30cm^2 , were used as specimens. The electrolyte for PEO was a dilute alkaline solution containing silicate, 2g/L KOH, and 5g/L Na_2SiO_4 . Its temperature was maintained between 20 and 45 °C during the process. PEO treatments were conducted with a high power 60Hz AC rectifier (Hyunsung Electric Co.), and voltage responses were recorded using a multimeter device (Keithley DMM 2100) during the process. As listed in Table 1, three different process regimes for PEO were applied, in order to make comparisons.

The thicknesses of the coatings prepared by the PEO treatments were determined with a measuring device (QuaNix Keyless). A scanning electron microscope (Hitachi S4800) was used to observe microstructures, and Cu $K\alpha$ radiation (Bruker D8) was used to obtain X-ray diffraction patterns for a phase analysis. Before the XRD measurements, all specimens were polished to a thickness of 70-80 μm . In addition, elemental distributions were assessed using an electron probe micro-analyzer(CAMECA SX100).

To evaluate mechanical properties, hardness values and wear resistances were measured. Vickers hardness values for cross-sections of the coatings were measured at 0.05kgf loading (Mitutoyo HM-100). The abrasive wear resistance was evaluated by a ball-on-disc test of the polished surface under set conditions (i.e. loading:10N loading, diameter of wear track: 10mm, and sliding speed: 0.1m/sec). The wear rate (mm^3/Nm) was then calculated from the volume loss of the coating after 1200m of sliding.

Table 1. Definitions of process names and regimes.

Process name	Process regime
Constant current(60min)	Constant current density of 500 mA/cm^2 for 60 min
Constant current(90min)	Constant current density of 500 mA/cm^2 for 90 min
Step-down current	Constant current density of 500 mA/cm^2 for 60min + Constant current density of 200 mA/cm^2 for 30 min

3. Results

3.1. Formation of PEO coatings

Voltage-time responses during the PEO treatments are shown in Fig. 1. In both cases, for either the constant current (90min) or the step-down current, the behavior was similar until 60 min into the process. Voltages rapidly increased to approximately 250 V within 1 min after the start and then gradually increased to approximately 350 V. In the case of the step-down current, the voltage began to decrease after 60 min in response to a step-down of current density from 500 to 200 mA/cm². In the case of the constant current (90min), however, the voltage continuously increased to approximately 370 V.

Fig. 2 shows the increase in thickness (i.e., the growth of a PEO coating) with processing time. Even with the current density constant at 500mA/cm², the growth rate of the coating was not uniform over the entire 90 min processing time. During the initial 20 min, the thickness gradually increased to 20 μm with a growth rate of 1 μm/min. Over the next 40 min, the thickness rapidly increased to 150 μm with a growth rate of 3.6 μm/min. During the final 30 min at 500mA/cm², the growth rate reverted to 1 μm/min, and therefore, the thickness increased to approximately 180 μm. Therefore, the constant supply of high current density always made a contribution to coating growth in spite of a variation in growth rate. However, with the current step-down to 200 mA/cm² after 60 min, the PEO coating showed no appreciable thickness increase during the final 30 min of processing. In this case, further growth of the PEO coating was abandoned.

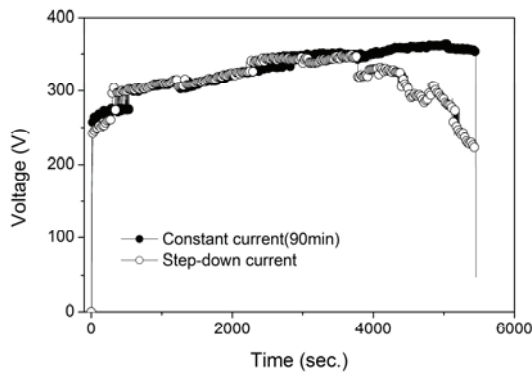


Fig. 1. Voltage-time responses during PEO treatments with processing time.

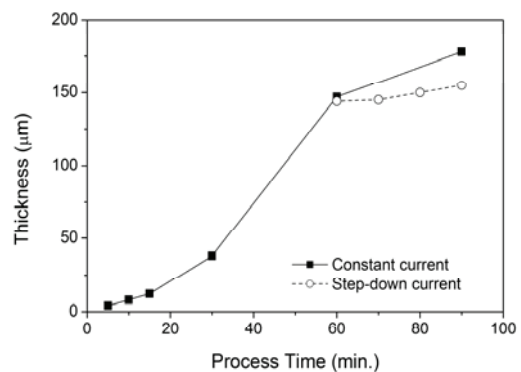


Fig. 2. Growth of PEO coating with processing time.

3.2. Structure of PEO coatings

Fig. 3 shows a cross-sectional micrograph of the PEO coatings prepared under each regime. All three PEO coatings exhibit a typical structure that consists of two oxide layers: a porous outer layer and a dense inner layer. The porous outer layer occupies almost 30% of the coating thickness. However, the excellent mechanical properties of PEO coatings are mainly contributed by the dense inner layer, the so-called function layer. Throughout the interfacial region between the outer layer and the inner layer, many pores are found. The overall thicknesses were 150, 175, and 155 μm with (a) the constant current (60min), (b)

the constant current (90min), and (c) the step-down current, respectively.

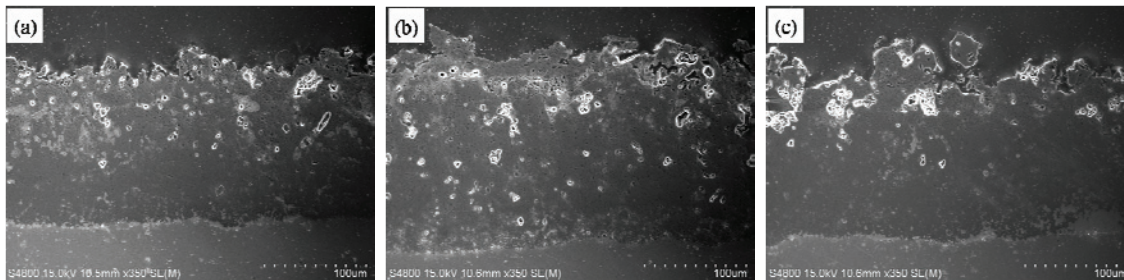


Fig. 3. Cross-sectional micrographs of PEO coatings: (a) for constant current (60min); (b) for constant current (90min); and (c) for step-down current.

Fig. 4 shows X-ray diffraction patterns of the PEO coatings after removing their outer layers. Among patterns (a-c), a notable difference in the angles or intensities of the peaks cannot be found, and this reveals that the constituent phases of the coatings were the same in all three cases. The major peaks indicate that the main constituent phases of the coatings were α - Al_2O_3 and γ - Al_2O_3 . In the patterns, minor peaks are also found and these reveal the presence of slight amounts of δ - Al_2O_3 and mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$). To investigate the locations of the phases within the coatings, the distributions of Al, O, and Si were analyzed by EPMA. It was found that the dense inner layer was composed entirely of alumina phases, while most of the mullite existed in the outer layer with SiO_2 .

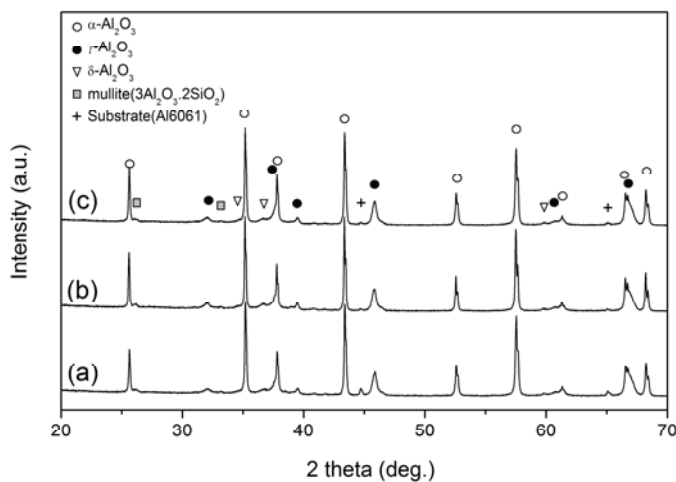


Fig. 4. X-ray diffraction patterns of PEO coatings after removing outer layer: (a) for constant current (60min); (b) for constant current (90min); and (c) for step-down current.

Fig. 5 shows further magnified images of the function layers as observed by SEM. There are significant differences with respect to structural defects such as porosity. A greater number of large pores are observed in the case (b) of the constant current (90min) than in the case (a) of the constant current (60min). That is, a structural degradation occurred. The pores in a PEO coating originated near the arc-

discharge region from an entrapment of evolved oxygen during the melting and rapid cooling of alumina in the process [5]. The assumption is that excessive processing at high current density caused an abundant evolution of oxygen gas, and therefore, the coating became porous. An abnormal dissolution of alumina may also have occurred in this manner. However, the case (c) of the step-down current exhibits the lowest porosity and the densest structure. Therefore, a densification phenomenon such as sintering presumably occurred, without negative effects from the excessive process, during the period of relatively low current density after the current step-down.

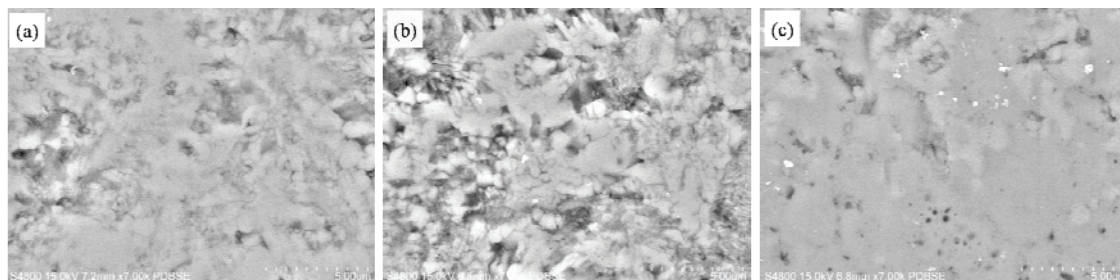


Fig. 5. Magnified cross-sectional micrographs of inner layers in PEO coatings: (a) for constant current (60min); (b) for constant current (90min); and (c) for step-down current.

3.3. Microhardness and wear resistance

Fig. 6 shows the Vickers hardness profiles of the inner layers, which are associated with the mechanical properties of the coatings. Each was measured along the distance outward from the interface between the substrate and the coating. In all cases, the maximum microhardness corresponded with a distance of approximately $40\mu\text{m}$. The peak values were 1300, 900, and 1800 Hv with constant current (60min), constant current (90min), and step-down current, respectively. That is, the microhardness of step-down current was two times higher than that of constant current (90min) although they involved the same total processing time. In addition, the hardness of the constant current (90min) was even lower than the constant current (60min). Because there were no differences in constituent elements or phases among the cases, the decrease in microhardness was evidently related with the high porosity shown in Fig. 5. Therefore, the degradation of a mechanical property, the hardness, was caused by the structural degradation from excessive processing at high current density. The increased hardness from the step-down current can be explained by a higher degree of sintering than in the other cases. If sintering is facilitated during the period of low current density, the inner layer becomes denser, which leads to increased microhardness.

To evaluate a tribological property, abrasive wear tests were carried out by the ball-on-disk method. Fig. 7 shows the wear rates calculated from the volume loss of the coatings. All of the PEO coatings experienced a significantly lower wear rate in comparison with uncoated Al6061 alloy. In the case of a constant current, the wear rate of the coating treated for 60minutes was $4.2 \times 10^{-7} \text{ mm}^3/\text{Nm}$, while that of the coating treated for 90 min was even higher at $9.7 \times 10^{-7} \text{ mm}^3/\text{Nm}$. In the case of the step-down current, however, the wear rate was much lower at $1.4 \times 10^{-7} \text{ mm}^3/\text{Nm}$. With regard to the wear resistance of the coating produced, a step-down current is better than a constant current (60min), which in turn is better than a constant current (90min).

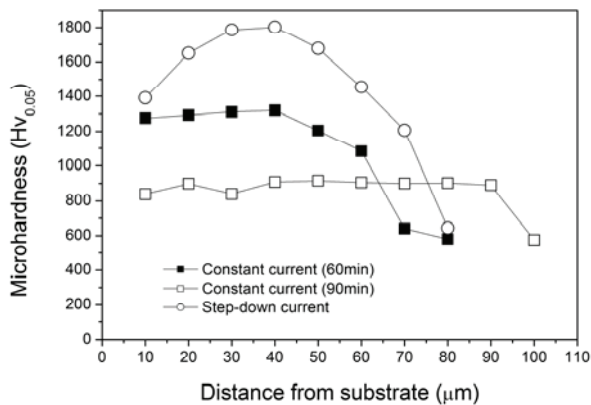


Fig. 6. Microhardness of inner layers with a distance from substrate.

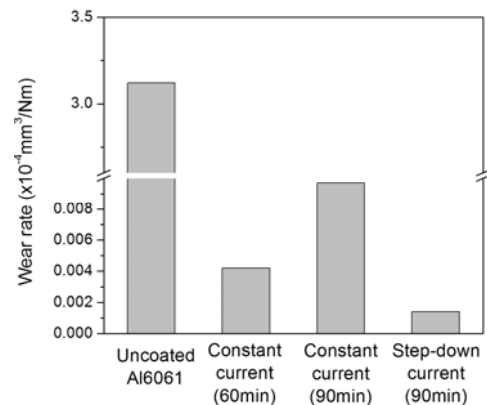


Fig. 7. Wear rates of PEO coatings prepared under different current regimes.

4. Conclusion

A current step-down was examined as a new type of current regime for PEO on aluminium alloy. The characteristics of coatings were investigated, and compared with those of coatings prepared using continuous AC supply of constant current density. Based upon the variations in coating thickness, a high current density is obviously conducive to growth of the coating overall. However, the results of SEM observations, hardness tests, and wear resistance tests all indicate that excessive processing in this manner causes the function layer of the coating to become more porous consequently leads to a degradation of its mechanical properties. In the case of the step-down current, during the stepped down period at low current density, the densification of the coating by sintering phenomena is more dominant than growth of the coating. This densification corresponds with a decreased number of pores, and thus, the microhardness and wear resistance of the function layer in the PEO coating are increased. Therefore, current step-down is useful to secure remarkably good mechanical properties without any degradation during PEO process for thick coating.

References

- [1] A.L. Yerokhin, X. Nie, A. Leyland, A. Matthews, S.J. Dowey. Plasma electrolysis for surface engineering. *Surf Coat Tech* 1999; **122**:73-93.
- [2] E. Matykina, R. Arrabal, P. Skeldon, G.E. Thompson. Investigation of the growth processes of coatings formed by AC plasma electrolytic oxidation of aluminium. *Electrochim Acta* 2009; **54**:6767-6778.
- [3] R.H.U. Khan, A. Yerokhin, X. Li, H. Dong, A. Matthews. Surface characterisation of DC plasma electrolytic oxidation treated 6082 aluminium alloy: Effect of current density and electrolyte concentration. *Surf Coat Tech* 2010; **205**:1679-1688
- [4] E. Matykina, R. Arrabal, A. Mohamed, P. Skeldon, G.E. Thompson. Plasma electrolytic oxidation of pre-anodized aluminium. *Corros Sci* 2009; **51**:2897-2905.
- [5] J.A. Curran, T.W. Clyne. Porosity in plasma electrolytic oxide coatings. *Acta Mater* 2006; **54**:1985-1993.