

CURRENT DENSITY EFFECTS ON PLASMA EMISSION DURING PLASMA ELECTROLYTIC OXIDATION (PEO) ON AZ91D-MAGNESIUM ALLOY

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Received Day Month Day

Revised Day Month Day

The effect of bipolar pulse mode current ratio on plasma behavior was investigated in PEO on AZ91D Mg-Alloy. Two cases of current ratio including 1.20 and 0.88 were applied to the sample. Plasma emission behavior was studied using plasma images and plasma emission measured by photodetector and Intensified Charged-Couple Device (ICCD) camera. The current ratio of greater than 1 shows the continuous increase and then stabilization in emission intensity with a gradual increase in voltage throughout the PEO process. In contrast, the current ratio of less than 1, a sudden drop in plasma emission intensity with voltage was found after 786s. Therefore, PEO process can be divided into two regimes, arc regime and soft regime, before and after voltage drop respectively. Results of measured spectra show that a soft regime does not have atomic or ionic excitation during PEO process. It is demonstrated that the growth of porous layer during PEO can be controlled, which is benefit for the protective oxide coating of sample.

Keywords: Plasma Electrolytic Oxidation (PEO); AZ91D Mg-alloy; Arc regime; Soft regime.

1. Introduction

In recent years, Mg and its alloys have a wide range application in various industrial fields, such as, aerospace, automotive and electronics. Comparing to other metals, Mg and its alloys present many advantages of low density, good electromagnetic shield, high machinability, and so on.¹ But the poor corrosion and wear resistance restrict their

applications. Mg-alloys show better corrosion resistance by alloying with Mn, Al or Zn. The corrosion resistance capability is still not good enough.

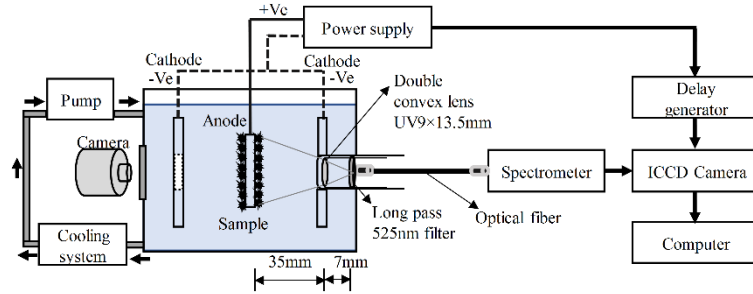
PEO is a promising technique to form the protective oxide coating. However, limitation for the growth of porous layer during PEO is necessary. Many researchers introduced special current regimes such as bipolar current pulses.²⁻⁵ The anodic to cathodic current ratio is an important parameter to reduce arcing that usually causes defects in oxide layer.^{6,7} The soft sparking regime can be included to conventional PEO regime in order to achieve thick, dense and homogeneous layer. But, plasma emission behavior with respect to time was not reported yet. Therefore, in present work, bipolar pulse constant current mode with different current ratio was applied to a sample. The effect of current ratio on plasma emission behavior was studied during PEO. The emission was monitored as a function of anodic pulse (200 μ s) throughout PEO process using photodetector and ICCD camera. The process was continued 3600 s, which is referred as PEO process time. The plasma image was also recorded during the PEO process.

2. Experimental setup

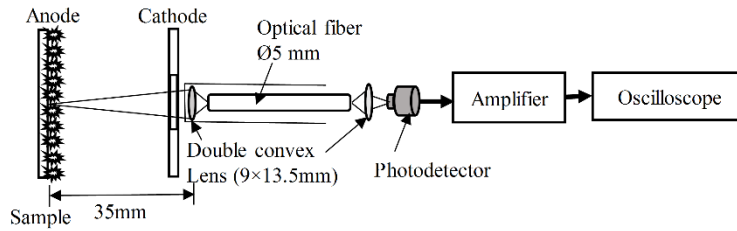
The schematic diagram of experimental setup in this study is shown in Figure 1. A rectangular block of AZ91D Mg-alloy sample was used as anode and two rectangular SUS plates with 16 mm hole in center was used as cathode. The distance of anode and each cathode was 30 mm. The electrolyte solution was prepared using $(\text{NaPO}_3)_6$, NaF, NaOH and NaAlO_2 while keeping pH about 12. The electrolyte solution in the container was the circular flow by the cooling system and pump.

Figure 1(a) shows the experimental setup for plasma emission measurement. The emitted light was collected using a double convex lens with diameter of 9 mm and the focal length of 13.5 mm. The collected light was transferred to a spectrometer (Japan Spectroscopic Co. Ltd., CT-10) using the optical fiber with core diameter of 600 μ m. The entrance slit of spectrometer was set to 20 μ m in width. The grating of 300 grooves per mm was used to acquire wide spectral range from 480 nm to 880 nm. The decomposed light from spectrometer was detected by the ICCD camera (Princeton Instruments, PI-MAX- Gen II). ICCD camera was thermoelectrically cooled to 263 K for high quantum efficiency. The detected signal was recorded by the computer. The time between power supply for cathode and anode and ICCD camera was controlled by the delay generator. The system calibration was carried out using the standard light (Stellar Net, SL1-CAL) before the experiment.

Figure 1(b) shows the experimental setup for emission measurement using photodetector. Emitted light was collected using the same lens in Figure 1(a). The collected light was transferred using the optical fiber with core diameter of 5 mm. At the other end of optical fiber, another lens was used to concentrate light to photodetector (Hamamatsu photonics S1336-44BQ). The distance between the end of optical fiber, lens and photodetector was set to collect the maximum light. The signals detected by photodetector were monitored using the oscilloscope (Tektronix TPS 2024 Oscilloscope). Table 1 lists the experimental parameters during the PEO process.



(a) Plasma emission measurement using spectrometer and ICCD camera.



(b) Plasma emission measurement using photodetector.

Fig. 1 Experimental setup for emission measurement during the PEO process.

Table 1 PEO process parameters

Case	Bipolar pulse timing (μs)				Frequency (Hz)	Duty Ratio (%)	Anode Current (A)	Cathode Current (A)	Current Ratio (-)
	Anodic pulse		Cathodic pulse						
	t_{ON}	t_{OFF}	t_{ON}	t_{OFF}					
I	200	800	200	800	500	10	1.2	1.0	1.20
II	200	800	200	800	500	10	1.5	1.7	0.88

3. Results and Discussion

The measurement results of sample anodic voltage were shown in Figure 2. The voltages show the linear increase at the beginning with weak emission intensity, and then the voltages increase evenly with plasma emission in case I and case II. However, in case II, the voltage decreases rapidly after 786s. The phenomena can be observed according to the plasma images of PEO process time, as illustrated in Figure 3. The plasma quenched very rapidly from 786s to 788s and the plasma emission became very weak after 840s. These images show the change of plasma emission from arc regime to soft regime. The results indicate that the soft regime was included when the current ratio was less than 1 for case II, which reduces the plasma emission. It is necessary to understand the plasma emission behavior during arc regime and soft regime with time. Therefore, the plasma emission was detected by photodetector and ICCD camera in PEO process.

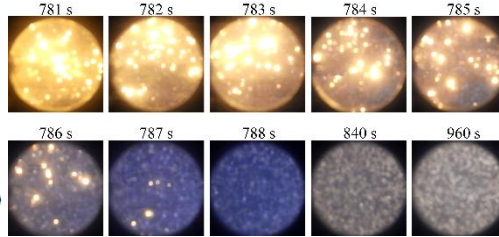
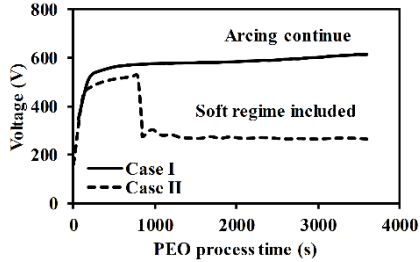


Fig.2 Anodic voltage response in different cases Fig.3 Plasma images by camera in case II from arc regime to soft regime

Figure 4 shows the anodic voltage pulse along with simultaneously measured plasma emission signal by photodetector, which show that plasma emission from the discharge sample correlates with applied bipolar current pulse. It is found that discharge behavior changes according to the change of current ratio. For case I in Figure 4(a), the emission intensity increases with increasing the anodic pulse time. However, case II in Figure 4(b) shows different emission behavior during arc and soft regimes. Arc regime in case II shows $14\mu\text{s}$ delay in the PEO process of 900 s. The emission intensity also increases rapidly first and then reduces and becomes constant. In soft regime, the emission intensity is very weak, which increases with raising voltage and reduces gradually with increase in anodic pulse time. Therefore, the PEO process can be controlled by the current ratio.

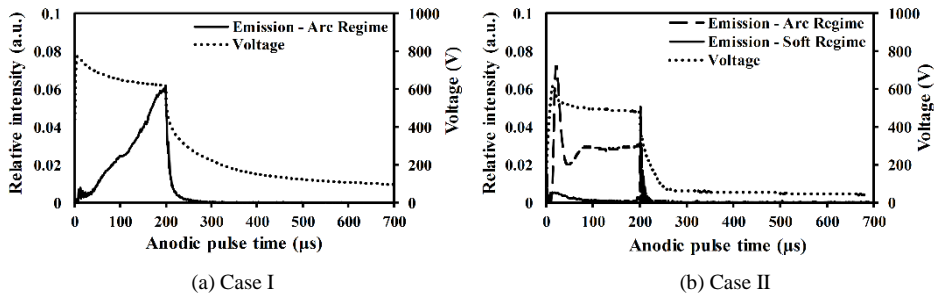


Fig. 4 Measured plasma emission behaviour using photodetector in different cases

The plasma emission was also detected by ICCD camera. Figure 5 shows the measured spectra of case II in arc regime and soft regime during PEO process. In arc regime, the measured spectra show the atomic emission lines of Na at 589nm and 819nm, H at 656nm, which are from the electrolyte solution. The background of measured spectra is non-uniform resulted by bremsstrahlung radiation of electrons and collision radiative recombination.^{8,9} Only continues spectra are observed in soft regime in Figure 5. But plasma images and emission measured by photodetector shows emission intensity. Therefore, emission in soft regime was measured for large number of cycles. Figure 6 shows the measured emission spectra in soft regime at 840 s for 10 accumulations and 350 accumulations. These results prove that there is no obvious atomic or ionic excitation in soft regime. The observed emission in soft regime was created by charged free particles because of free radiation. According to the measurement results of plasma emission in

different cases, the soft regime presents the improved performance for protective oxide coating.

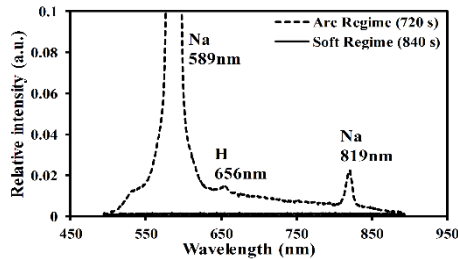


Fig. 5 Emission spectra of case II

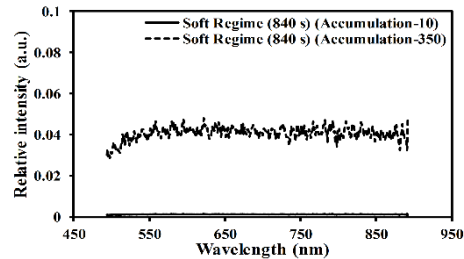


Fig. 6 Emission spectra of case II in soft regime for different accumulation

4. Conclusion

Plasma Electrolytic Oxidation (PEO) is a promising technique to form the protective oxide coating for the solid alloy. The bipolar pulse constant current mode with different current ratio was applied to a AZ91D Mg-alloy sample to discuss the effect of current ratio on plasma emission behavior. Plasma behavior was affected by applied bipolar current ratio. The current ratio greater than 1 shows only arc regime. However, the current ratio less than 1 shows both arc regime and soft regime. Arc regime show atom emission. However, soft regime does not show the atomic excitation. The soft regime presents the enhanced ability for protective oxide coating. Therefore, the PEO process can be controlled by the current ratio to improve the performance of alloy.

References

1. Y.L. Song, Y.H. Liu, S.R. Yu, X.Y. Zhu and Q. Wang, *Applied Surface Science* **254** (2008) 3014-3020.
2. F. Mecuson, T. Czerwec, T. Belmonte, L. Dujardin, A. Viola and G. Henrion, *Surface and Coating Technology* **200** (2005) 804-808.
3. Y. Guan, Y. Xia and G. Li, *Surface and Coating Technology* **202** (2008) 4602-4612.
4. C. S. Dunleavy, I. O. Golosnoy, J. A. Curran and T. W. Clyne, *Surface and Coating Technology* **203** (2009) 3410-3419.
5. R. O. Hussein, X. Nie and D. O. Northwood, *Surface and Coating Technology* **205** (2010) 1659-1667.
6. F. Tjiang, L.W. Ye, Y. J. Huang, C. C. Chou and D. S. Tsai, *Ceramics International* **43** (2017) S567-S572.
7. F. Mecuson, T. Czerwec, G. Henrion, T. Belmonte, L. Dujardin, A. Viola and J. Beauvir, *Surface and Coating Technology* **201** (2007) 8677-8682.
8. M. D. Klappkiv, H. M. Nykyforchyn and V. M. Posuvailo, *Material Science* **30** (1995) 333-343.
9. S. Stojadinovic, R. Vasilic, M. Petkovic, Z. Nedic, B. Kasalica, I. Belca and Lj. Zekovic, *Electrochimica Acta* **55** (2010) 3857-3863.