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Plasma Electrolytic Oxidation of AZ31 Mg Alloy in Bipolar Pulse Mode and Influence of Corrosion to Surface Morphology of Obtained Coatings

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The plasma electrolytic oxidation method was used with AZ31 magnesium alloy plates for improving the corrosion resistance of the alloy. Process parameters for the plasma electrolytic oxidation setup were optimized by studying the effects of KOH concentration, operating voltage, and pulse properties on the obtained coating. These conditions were then used to produce plasma electrolytic oxidation coated AZ31 sample and were tested by immersion in 3 % NaCl solution for 1 week. Three types of modifiers were used in the electrolyte and concentrations of the modifiers were varied to study the effect of concentration on the performance of coating obtained. The extent of corrosion was visually examined, and it was found that an electrolyte recipe with all three modifiers produced the best results.

Keywords: plasma electrolytic oxidation, AZ31, corrosion resistance, self-healing magnesium alloys.

1. INTRODUCTION

Magnesium and its alloys are highly susceptible to corrosion. This has historically limited their use in the automotive and aerospace industries, where harsh operating conditions cannot be avoided [1]. Its low density and good mechanical properties make it a very serious candidate in the automotive industry to replace other metals and their alloys, at a time when climate directives are reducing emissions and fuel consumption already for a few decades and making it increasingly difficult for car manufacturers to comply [2 – 5].

The easiest way to avoid corrosion is to coat the magnesium alloys with a barrier layer to prevent contact with the environment [4]. However, in the case of magnesium alloys, the technology of surface protection coatings production is limited by the low allowable thermal effects during their processing, as it can irreversibly affect the properties of the magnesium alloy substrate [6].

Over the last few decades, the surface treatment of magnesium and its alloys has developed a great deal, but only a few processes have gained commercial importance. Chemical conversion treatment is by far the most common surface treatment method for magnesium alloys like AZ31. The surface of magnesium metal is basic in nature, so its surface must be pre-treated to make it more compatible with paints and other organic coatings to improve adhesion. The main active ingredient in most of the chemical conversion processes for magnesium and its alloys is the hexavalent chromium (VI) ions, which are carcinogenic and toxic [6, 7]. A lot of effort has been directed toward finding effective substitutes for chromate processing using, for example, phosphates. However, chromate modification is still the method of choice for obtaining a protective coating on magnesium parts. This is especially true for components used in highly corrosive environments. The most common conversion coatings used in industry are chromium brine, dichromate, chromium manganese, iron nitride brine and phosphate treatment.

In recent years, it has been possible to use less harmful Cr^{3+} compounds, but their efficiency is much lower, and the costs of the technological process are quite high [8]. The use of vanadium and rare earth modifiers for corrosion resistance is also proposed as an alternative, however, the main disadvantage of both methods is the very high cost and, compared to the use of Cr^{6+}, also less efficient corrosion resistance [9, 10].

Electrochemical methods such as anodizing, and plasma electrolytic oxidation (PEO) are also widely used in the production of protective coatings for magnesium alloys.

Anodic treatment is performed by applying an electric current to the magnesium alloys to be treated, while it is immersed in a specially developed anodizing solution-electrolyte. During this, the surface oxidizes, and a relatively dense barrier layer is formed, however, pores are observed in it, due to which it is not able to completely protect the magnesium alloys from corrosion.

Plasma electrolytic oxidation is one of the most promising forms of magnesium alloy processing today. It is like anodizing, but the method operates on much higher currents and voltages. Unlike anodizing, the process is very short - sample processing takes only 1 – 5 minutes. First, the

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surface of magnesium alloys forms a thick oxide of the parent metal. Then, as soon as the dielectric strength of this film reaches the level of the applied compressed stress, an arc discharge occurs at the metal/solution interface. The heat generated by the arc discharge breaks down the chemical precursors in the solution and at the same time causes the oxides of other elements to precipitate. The result is a thick and hard coating that significantly changes the surface properties and promotes the adhesion of the after-treatment coating, such as paint. Anodizing processing requires higher capital investment and higher operating costs than most chemical conversion methods. But they are also generally more environmentally friendly than chemical conversion processes, despite the fact that most of the electrolytes used contain fluoride salts, which improve the wear resistance and hardness of the material. Anodized coatings are porous in nature and, if not properly sealed with paint or resin, are unsuitable for corrosive environments, so the treatment method must be supplemented with other surface protection methods, making it more expensive. The porous anodized surface can be impregnated with organic sealants to provide greater resistance to corrosion in aggressive environments [1, 11 – 13].

Increasingly, both anodizing and PEO processes are performed using bipolar power sources and pulse mode. This allows the simultaneous introduction of different modifiers into the coating, improving the mechanical and anti-corrosion properties of the coating [14 – 17].

Often the methods of obtaining coatings are combined to obtain the best possible result. PEO coatings are often coated with self-curing agents, providing high corrosion resistance even in the case of mechanical damage to the coating. Scientists are increasingly investigating which modifiers to incorporate into the coating to achieve effective corrosion protection of the coating. The main candidates are phosphates, silicates, nanoparticles of graphene, rare earth elements, like praseodymium, and conversion coating [18 – 23]. In this paper, a novel approach is employed to use a combination of phosphate, zirconate, and silicate in the PEO coating and their effect on corrosion resistance is studied.

2. MATERIALS AND METHOD

Magnesium alloy AZ31 plates (SMW group) with a chemical composition of 3 % Al, 1 % Zn, 0.20 % Zn, 0.10 % Si, and the rest of Mg and trace elements are used as the substrate materials for the study. The platinum counter electrode (99.99 %), potassium hydroxide (≥ 85 %; Sigma Aldrich) for the electrolyte, sodium phosphate dibasic dodecahydrate (98.5 – 102.5 %; Sigma Aldrich), tetraethyl orthosilicate TEOS as silicon modifier source (Sigma Aldrich), zirconium acetate solution (solution in acetic acid, Sigma Aldrich), deionized water (prepared with Adrona Crystal 7 Pure water purification system), acetone (for analysis, Merck), NaCl (≥ 99 %, Sigma Aldrich) were used as raw materials in this research. Processes performed in the study are summarized in Fig. 1.

Custom made 10 kW bipolar pulse generator ELGOO PEO v3 (Applied Electronics labs) was used as a power source during the PEO process to obtain corrosion resistant coatings on the AZ31 Magnesium alloys surface. The LPEO v2.8 (Applied Electronics labs) PC software was used to set up and control current, voltage, pulse amplitudes, duration, and repetition rate during experiments. The bipolar mode was chosen to modify the resulting coating with silicon, phosphorus, and zirconium-containing ions in various combinations, depending on the chosen electrolyte, simultaneously with the surface PEO treatment. Such an approach ensures a homogeneous distribution of modifiers over the entire volume of the coating.

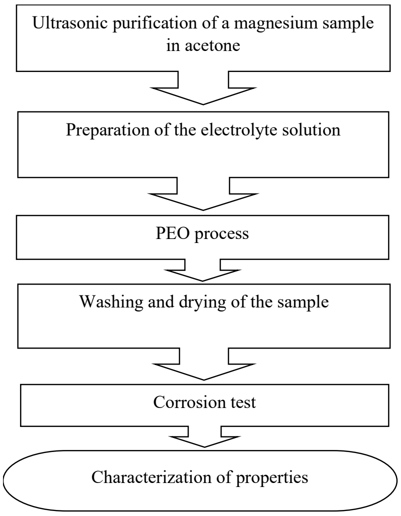


Fig. 1. Flowchart of processes performed in the study

Magnesium alloy AZ31 plates (2 × 2 cm; thickness 2 mm) were placed in an ultrasonic bath Retsch UR1 with acetone and degreased for 30 minutes. The processes performed in the study are summarized in Fig. 2.

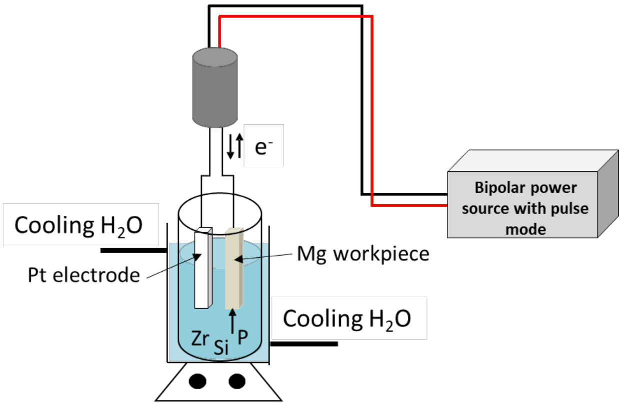


Fig. 2. Equipment schematic diagram for PEO process

Different compositions of electrolytes were prepared to study the effect of modifier concentration on the PEO coating. The different series of electrolytes had the following electrolytes dissolved in 1 L of distilled water:

- Series A: 0.5 g KOH;
- Series B: 0.5 g KOH and 0.1 – 5 g $NaH_2PO_4 \cdot 9H_2O$;
- Series C: 0.5 g KOH, 1 g $NaH_2PO_4 \cdot 9H_2O$ and 0.5 – 5 ml TEOS Series D: 0.5 g KOH, 1 g $NaH_2PO_4 \cdot 9H_2O$, 1 – 10ml 0.1 % TEOS and 1 – 10 ml of 0.1 % zirconium acetate solution;
- Series E: 0.5 g KOH, 2 or 4 g $NaH_2PO_4 \cdot 9H_2O$, and 1 or 5 ml 0.1 % zirconium acetate solution;

- Series F: 0.5 g KOH, 2 or 4 g $NaH_2PO_4 \cdot 9H_2O$, 5 or 10 ml 0.1 % TEOS and 1 or 5 ml 0.1 % zirconium acetate solution. Once the electrolyte solutions have been prepared, they were transferred to the water-cooled reactor located on the stirrer (Fig. 2). The reactor is cooled with cold water flow during the PEO process to prevent boiling of the electrolyte solution.

Once the magnesium alloy plate has been cleaned, it and the platinum plate are placed in a sample holder, and immersed in the electrolyte solution so that the working surfaces of the electrodes are completely below the level of the solution surface, but the holder elements do not touch the electrolyte solution and connected to a bipolar power supply.

For bipolar power supply, the lengths of positive and negative pulses were set to 9999×10^{-6} sec and the length of positive and negative pause was set to 3×10^{-6} sec. The current I+ was set to 5A and I− to 2.5 A. In series A the content of electrolyte solutions was not changed, but anodic voltage U+ was varied in the range of 200 – 550 V and cathodic voltage U− was changed in the range of 200 – 300 V. In all other series, the voltage was constant, U+ = 500 V and U− = 300 V.

When all values were set on the bipolar power supply software, the PEO process was performed for 2 min. After the PEO process sample was removed from the sample holder, rinsed, and dried in a universal laboratory oven Memmert UN55 at 110 ºC for 4 hours.

3. EXPERIMENTAL

3.1. Corrosion test

Once the samples were dried, two diagonal lines on one of the surfaces were drawn with a scalpel, damaging the coating, and exposing the metallic surface of the alloy. This was done to compare the behaviour of the exposed surface with the coating. The samples were placed vertically in separate vessels containing 3 % NaCl solution so that the sample is completely immersed in the solution. To minimize the evaporation of liquid and to ensure that at least 2 – 3 cm of liquid always remained above the samples, the container was closed with a lid. A corrosion test was performed for 168 h (1 week), and the salt solution was stirred once a day, allowing the sediment from the surface of the sample to settle to the bottom of the vessel. The containers were kept in a chamber with an average hourly temperature of 18.5 °C. The fluctuation in the temperature was ± 2.5 °C and was deemed not high enough to affect the corrosion performance over a period of 3 weeks. After the corrosion test, the samples were rinsed in deionized water.

Table 1. Summary of electrolyte composition and selected PEO process parameters

Sample	U^+ / U^-, V	KOH, g	$NaH_2PO_4 \cdot 9H_2O$, g	TEOS, ml	0.1% TEOS, ml	0.1% zirconium acetate, ml
A1	200 / 200	0.5				
A2	250 / 250	0.5				
A3	300 / 300	0.5				
A4	350 / 300	0.5				
A5	400 / 300	0.5				
A6	450 / 300	0.5				
A7	500 / 300	0.5				
A8	550 / 300	0.5				
B1	500 / 300	0.5	0.1			
B2	500 / 300	0.5	0.5			
B3	500 / 300	0.5	1			
B4	500 / 300	0.5	2			
B5	500 / 300	0.5	5			
C1	500 / 300	0.5	1	0.5		
C2	500 / 300	0.5	1	1		
C3	500 / 300	0.5	1	2		
C4	500 / 300	0.5	1	5		
D1	500 / 300	0.5	1		1	1
D2	500 / 300	0.5	1		1	1
D3	500 / 300	0.5	1		1	10
D4	500 / 300	0.5	1		10	1
D5	500 / 300	0.5	1		10	10
E1	500 / 300	0.5	2			1
E2	500 / 300	0.5	2			5
E3	500 / 300	0.5	4			5
F1	500 / 300	0.5	2		10	1
F2	500 / 300	0.5	2		10	5
F3	500 / 300	0.5	4		10	5
F4	500 / 300	0.5	2		5	1
F5	500 / 300	0.5	2		5	5
F6	500 / 300	0.5	4		5	5

3.2. Morphology, chemical and phase content analysis

Characterizations of physical properties as morphology, chemical and phase content, were done by scanning electron microscope Hitachi S-3000N, equipped with an energy-dispersive X-ray spectroscopy (EDS) Bruker Quantax System XFlash 4010 detector and software QUANTAX ESPRIT 1.8.2 to determine the chemical composition of samples and X-ray diffractometer Bruker AXS D8 Advance, respectively.

4. RESULTS AND DISCUSSION

4.1. Corrosion test

The corroded sample after 168 h (1 week) of immersion in 3 % NaCl solution is shown in Fig. 4. The AZ31 sample coated with series A electrolyte (KOH only) show very bad corrosion resistance with the whole sample corroded and discoloured compared to the untested sample.

For series B, sample B4 shows very good corrosion resistance with 0.5 g/L KOH and 2 g/L $NaH_2PO_4{\cdot}9H_2O$ electrolyte, while B1 and B6 perform very poorly. Series A with no added modifiers show the most intensive damage, followed closely by series B. In the series A, the highest voltage (A8) shows the least corrosion damage, and this voltage setting was used with the modified electrolyte. The B1 sample with the lowest phosphate modifier in the electrolyte has extensive damage and pitting corrosion on the surface. But there is a slight improvement compared to the A series. Series D samples have comparatively the least damage among all series. A combination of this series has two modifiers in the electrolyte unlike series F, which also shows good corrosion resistant properties (sample F2), but there is still more corrosion than series F. The best performance is shown by the D series with all three samples with very little corrosion, unlike other series where some concentrations lead to completely blackened samples.

4.2. XRD Analysis

The XRD patterns of the corroded coating on the AZ31 alloy substrate are shown in Fig. 3, where the patterns 1 – 5 correspond to the sample D1, B2, F3, D5 and A7, respectively. Several peaks corresponding to Mg can be seen in the patterns with the most prominent at $2\theta = 36.9^{o}$. Only series A patterns show peaks corresponding to $Mg(OH)_2$ at $2\theta = 37.9$ and 18.6 and were not seen in any other series. This is consistent with the 2θ value related to $Mg(OH)_2$ reported elsewhere [24].

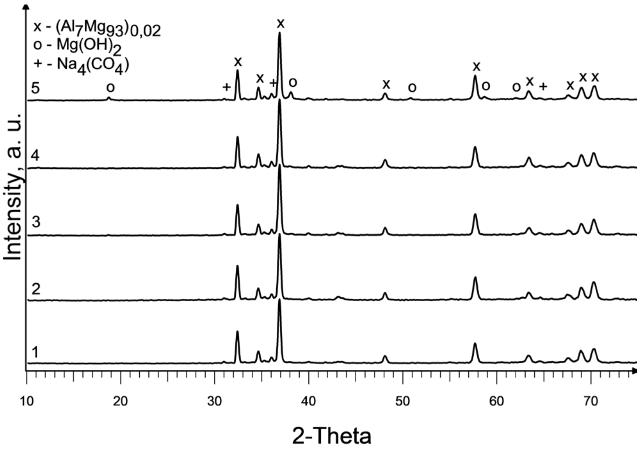


Fig. 3. XRD patterns of PEO coated sample

4.3. Coating morphology after corrosion test

Micrography was performed to study the surface morphology of the corroded sample. SEM images of samples B3, E3 and D4 are shown in Fig. 5 after corrosion testing. As can be seen in the higher magnification image of B3 (Fig. 5 b) and D4 (Fig. 5 f), the coating retains the porous structure after the corrosion. Sample E3 (Fig. 5 c, d) with PEO coating with only phosphorus modifier shows a high degree of corrosion and cracks at the micro scale, even though visual inspection shows that the surface is not damaged. Corrosion spreads to the scratched region as well and the oxide coating is also extensively damaged.

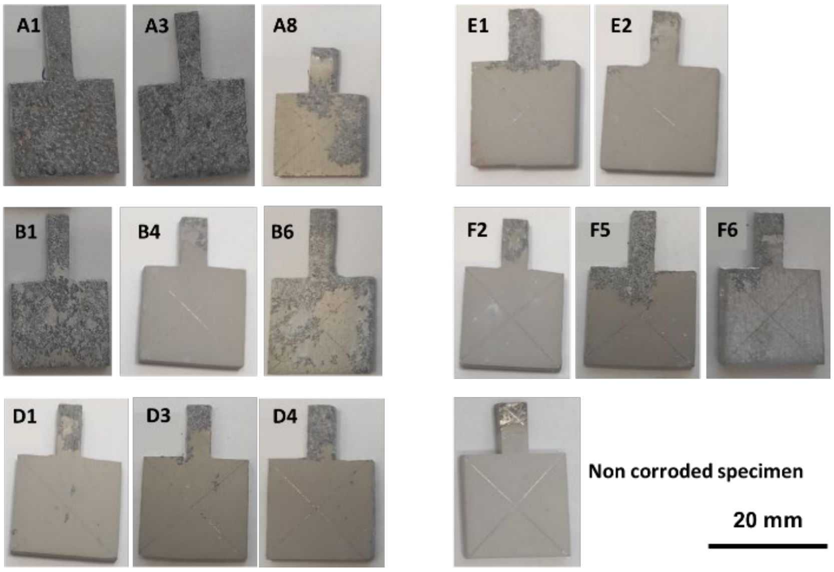
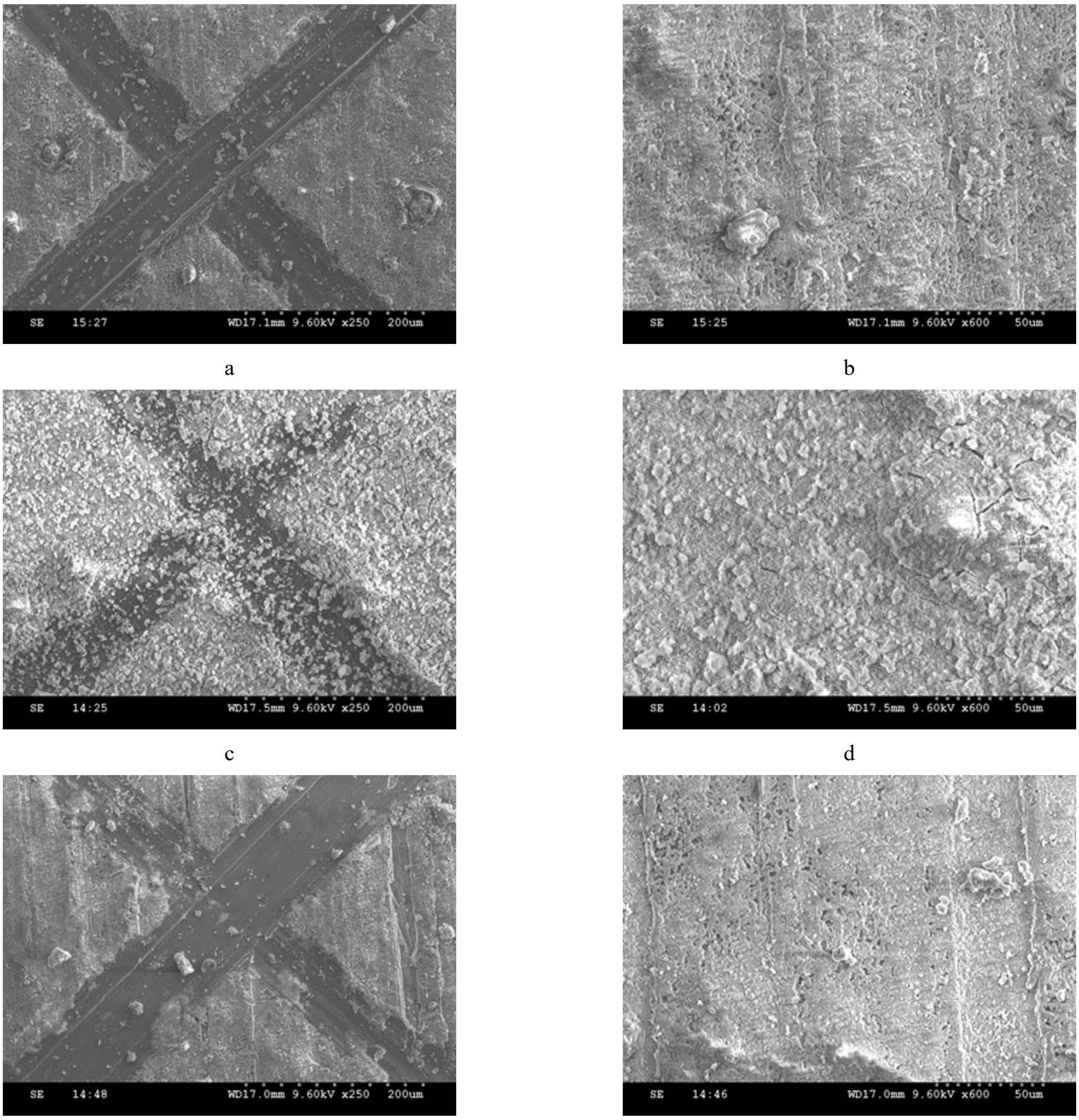


Fig. 4. Pictures of corroded sample of PEO coated AZ31 of various electrolyte series



a b

c d

e f

Fig. 5. SEM images of corroded PEO coated sample: a, b – B3; c, d – E3; e, f – D4 at 250x (right) and 600x (left) magnifications, respectively

Sample D4, with oxide coating modified with all three additives, shows the best performance among all the samples. The scratched mark shows no signs of corrosion as well, indicating that the coating has self-healing and corrosion retarding capabilities. The PEO coating significantly slows down and even stops the corrosion of even exposed base Mg alloy substrate. The surface looks pristine as opposed to the case in E3 where the surface is covered with loosely bound powder like particles. These particles are formed as a result of the corrosion action on the PEO surface. One explanation of this observation can be that the pores on the series sample are larger and thus, allows for more corrosion to take place.5. Conclusions

The PEO process for AZ31 was optimised to obtain operating anodic and cathodic voltage with KOH as an electrolyte. The study shows that 500/300 V voltage was optimum for running PEO on the prepared Mg alloy sample. Proper concentrations of modifiers in the electrolytes were obtained by studying the effect of concentration on the corrosion resistance of the AZ31 sample. It was found that adding silicate, phosphate and zirconium modifiers in the KOH based electrolyte improves the corrosion resistance of Mg alloy AZ31 in immersion type 3% NaCl corrosion test. Sample D4 with a combination of all 3 types of modifiers in the electrolyte solution was found to provide the best corrosion protection to the AZ31 Mg alloy.

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