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Research Article

Effect of Plasma Electrolytic Oxidation Process on Corrosion Resistance of Al-Coated AZ61 Magnesium Alloy

İdris Durna[1*], Recep Çatar[2], Hikmet Altun[1]

[1] Atatürk University, Faculty of Engineering, Department of Mechanical Engineering, Erzurum, Türkiye, idrsdurna@gmail.com, haltun@atauni.edu.tr, ror.org/03je5c526
[2] Bayburt University, Faculty of Engineering, Department of Mechanical Engineering, Bayburt, Türkiye, recepcatar@yahoo.com, ror.org/050ed7z50
[*]Corresponding Author

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ABSTRACT

Magnesium alloys are extensively utilized in aerospace, aviation, and automotive sectors due to their low density and high specific strength, which offer significant advantages for lightweight structural applications. Their low density especially directs the attention of the aviation sector, where lightness is necessary, to these alloys. However, one of the disadvantages that restricts their use in the industry is their low corrosion resistance. In this study, the Plasma Electrolytic Oxidation (PEO) process was applied in a silicate-based electrolyte to AZ61 magnesium alloy pre-coated with aluminum using the electric arc spraying technique. To examine the characteristics of the fabricated coatings, their phase structures, surface features, and elemental compositions were investigated through XRD, SEM, and EDS analyses. Corrosion performances of coatings were investigated by potentiodynamic polarization test in 3.5% NaCl solution. As a result of electrochemical tests, corrosion current density values for the base sample (AZ61) and PEO coating (AZ61-PEO) were determined as 36,29 $\mu A.cm^{-2}$ and 6,53 $\mu A.cm^{-2}$, respectively. The findings highlight that the Al_2O_3 layer formed following PEO treatment significantly enhanced the corrosion resistance compared to the base sample.

1. Introduction

Since World War II, magnesium (Mg) and its alloys have significantly developed and have demonstrated their critical role in various technological applications [1]. With increasing demand and applicability in automotive, aviation, and many other areas, magnesium alloys exhibit sufficient strength and a density-to-weight ratio. With a density value of 1.74 g/cm^3, it is 65% lighter than aluminum (Al) and can replace metals such as iron (Fe) and Al, which are structural materials intended for aviation applications. Magnesium and its alloys provide a significant advantage in fuel economy, especially in aviation.

However, magnesium alloys have limited application areas due to their low melting point, poor wear resistance, and high chemical activity. Traditional coating methods are an effective method to improve these properties. Conventional coating methods include anodizing, thermal coatings, physical vapor deposition (PVD), and electroless nickel plating (EN). For the application areas of magnesium and its alloys to become widespread, their resistance to corrosive environments must be increased [2-4]. It has generally been stated that a single coating or a single surface treatment on magnesium alloys cannot meet various application requirements. Therefore, the application of complex surface treatments, such as some multilayer coatings, contributes to the increase in the corrosion resistance of these alloys and, thus, the widespread use of their areas [5-8].

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Plasma Electrolytic Oxidation (PEO) is an advanced anodizing technique employed to form durable ceramic oxide layers on lightweight metals and their alloys, such as aluminum, magnesium, titanium, and zirconium. The PEO process is carried out in a suitable electrolyte according to the desired coating composition after some pretreatments, such as surface polishing and cleaning. In the PEO system, an electrode pair consisting of a cathode (usually stainless steel) and an anode (workpieces) provides a suitable electrochemical setup. The PEO process uses appropriate potential and current values [9-11].

Various electrolytes have been applied in PEO processes, including alumina, phosphate, and silicate-based solutions [12]. Numerous properties of electrolytes, such as composition, concentration, conductivity, and solution pH, affect the coatings' morphology, thickness, and porosity. This naturally causes changes in the tribological and corrosion properties of the coating [13]. Literature has emphasized that PEO coatings are porous. Multilayer coatings have been used to eliminate corrosion defects. In addition, the effect of additives such as carbon nanotubes, fluoride, etc., added to PEO solutions has also been investigated.

It was coated AZ31B alloy with Micro Arc Oxidation (MAO) Ti (MAO/Ti) and TiN (MAO/TiN) with PVD process. Electrochemical tests showed that the MAO/TiN coating exhibited the best corrosion resistance among all samples. It was stated that the MAO/Ti coating had worse corrosion resistance than the MAO coating. It was concluded that this situation may be caused by micropores and cracks in the coatings' MAO/Ti coating structure [6]. In addition to the multi-walled carbon nanotube (MWCNT) (0-4 g/L), the PEO process was applied to the AZ80 magnesium alloy. When the effects of CNTs were examined, it was observed that the number of pores in the coating structure increased with an increase in the CNT ratio, especially after adding 1 g/L CNT. Electrochemical impedance analysis demonstrated that an expanded arc radius of the capacitive loop in the low-frequency region reflects a higher level of corrosion resistance in the applied coating. The largest arc diameter at a low frequency of 0.5 g/L CNT was observed, and thus, the best corrosion resistance was obtained at this value [14].

One of the methods used to protect magnesium alloys from corrosive environments is the electric arc spray method of thermal coating. The system creates an arc between two wires, and the resulting arc melts the wire ends. The melted electrode is sprayed onto the material to be coated using pressurized gas, and the coating process is completed. All materials with electrical conductivity can be used as electrode materials for coating. The wires used are generally pure or alloyed metallic (copper, zinc, aluminum, etc.) materials or wires containing ceramic-based particles [15].

A study was conducted in which aluminum coatings were applied to AZ31 magnesium alloy using the electric arc spray method. The corrosion resistance of the coated samples was assessed through salt immersion and electrochemical tests. Heat treatment was performed after spraying to enhance the coating's density and improve adhesion between the substrate and the aluminum layer. Their findings indicated that the corrosion current values of both untreated and heat-treated AZ31 samples exhibited a reduction in electrochemical tests.

As a result, it was emphasized that integrating electric arc spraying with subsequent heat treatment markedly enhanced the corrosion resistance of AZ31 magnesium alloy [16]. In a comparable investigation, the application of aluminum coatings on AZ31, AZ61, and AZ91 magnesium alloys was explored via the electric arc spray technique. The corrosion performance of the coated samples was examined in a 3.5 wt% NaCl solution. Potentiodynamic polarization analysis indicated that the aluminum coatings applied by electric arc spraying had a minimal impact on the corrosion current density values. However, the corrosion potential shifted towards more positive values, indicating an improvement in the electrochemical stability of the coated specimens [17].

The literature indicates that single-layer coatings applied to magnesium alloys are inadequate in terms of corrosion performance. However, despite extensive research on PEO and thermal spray techniques, studies on duplex coatings using these combined methods are still lacking. Therefore, unlike the literature in this study, Al coating was applied to AZ61 magnesium alloy using the electric arc spray coating method. The PEO process then formed an oxide layer on the Al-coated surface. This new duplex coating combines the barrier properties of the sputtered Al layer with the ceramic hardness of PEO oxide to provide superior corrosion resistance compared to single-layer coatings. The primary objective of this study is to demonstrate the effectiveness of this hybrid approach and highlight its potential for expanding the structural applications of magnesium alloys in harsh environments.

2. General Methods

2.1. Material

The AZ61 magnesium alloy utilized in this study had a cross-sectional diameter measuring 16 mm and a thickness of 6 mm. The alloy's chemical composition is detailed in Table 1. Before the coating process, all samples were ground with 600, 800, and 1200 (SiC) grit sandpaper. Then, the samples were cleaned with acetone and air-dried.

Table 1. Chemical composition of AZ61 (% wt.)

Al	Zn	Mn	Fe	Cu	Si	Mg
6.242	1.246	0.369	0.029	0.001	0.137	Bal.

2.2. Plasma electrolytic oxidation process

Figure 1 illustrates the schematic of the anodizing system utilized in the PEO process. The experimental arrangement features a computer-regulated power supply, a 5-liter 304L stainless steel container functioning as the cathode, a stirrer ensuring uniform electrolyte mixing, and a protective wooden enclosure to enhance electrical insulation and operational safety. Further experimental details are summarized in Table 2.

Table 2. Experimental parameters for the PEO process

Properties	Parameters	Time
Electrolyte composition	10 g/L Na_2SiO_3 + 5 g/L KOH	
Constant Current Mode	0.140 A/cm^2	15 min.
Voltage	400-460 V	

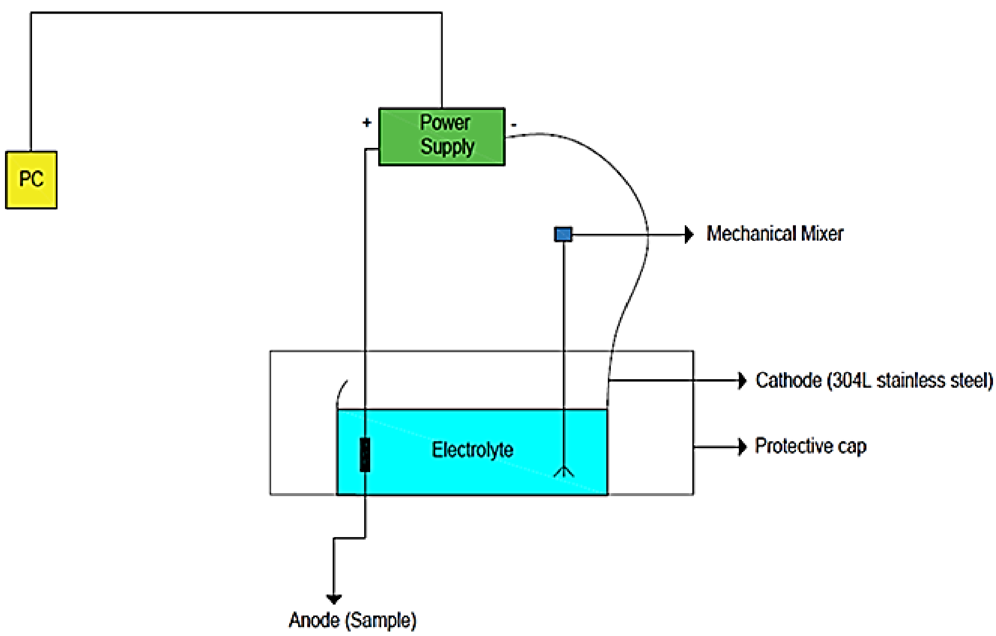


Figure 1. Schematic view of the PEO process

2.3. Electric arc spray process

The aluminum coating on AZ61 magnesium alloy was applied using an Oerlikon Metco Flexi.

Emin Metal supplies the Arc 300 electric arc spray system. The coating process utilized pure aluminum electrodes with a diameter of 1.6 mm. Table 3 presents the experimental parameters of the electric arc spray process.

Table 3. Experimental parameters for the electric arc process

Voltage (V)	Current (A)	Spray distance (mm)	Air pressure (bar)
26-29	60-70	150	6

2.4. Material characterization

The phase composition of the coatings was identified using a D8 Discover X-ray diffractometer. The analysis was carried out with Cu-Kα radiation (λ = 1.5406 Å), employing a scanning speed of 2° per minute over a 2θ range from 20° to 80°. Surface morphology was examined using a field-emission scanning electron microscope (FE-SEM, FEI Nova NanoSEM 450). Energy-dispersive X-ray spectroscopy (EDS) was conducted using an EDAX Element system integrated with the SEM, operating at an accelerating voltage of 10 kV to determine the elemental composition.

2.5. Potentiodynamic corrosion tests

The electrochemical corrosion behavior was examined using a VersaSTAT 3 potentiostat coupled with a three-electrode corrosion cell provided by AMETEK. A silver/silver chloride ($Ag/AgCl_2$) electrode was the reference, with a graphite rod as the counter electrode and the coated samples as the working electrode. The open-circuit potential (OCP) was initially monitored in a 3.5 wt% NaCl solution until stabilization. Subsequently, the samples were polarized across a potential range from –0.2 V to 2.0 V, and the resulting potentiodynamic polarization curves were recorded.

3. Results and Discussion

3.1. XRD analyses

Figure 2 shows the XRD spectra of both uncoated and coated AZ61 alloy samples. Analysis of the diffraction patterns revealed strong aluminum peaks in the duplex-coated specimens. The absence of Al_2O_3 phase signals in the XRD results is attributed to the considerable thickness of the aluminum layer deposited via the electric arc spray technique. Nevertheless, the observed peaks align well with findings reported in comparable studies from the literature [18-20].

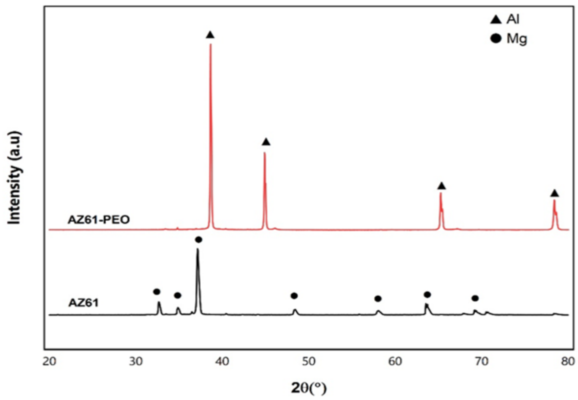


Figure 2. XRD patterns of samples of AZ61 and AZ61-PEO

3.2. SEM-EDS analyses

Figure 3a presents SEM images of AZ61-PEO coatings at different magnifications. When the SEM images of the AZ61-PEO coating were examined, a homogeneous and porous structure was observed. The pore diameters were approximately in the range of 1-2.5 µm by image analysis technique. The formation of pores and microcracks within PEO coatings was noted, with increasing pore sizes observed under higher voltage and current conditions [18-21].

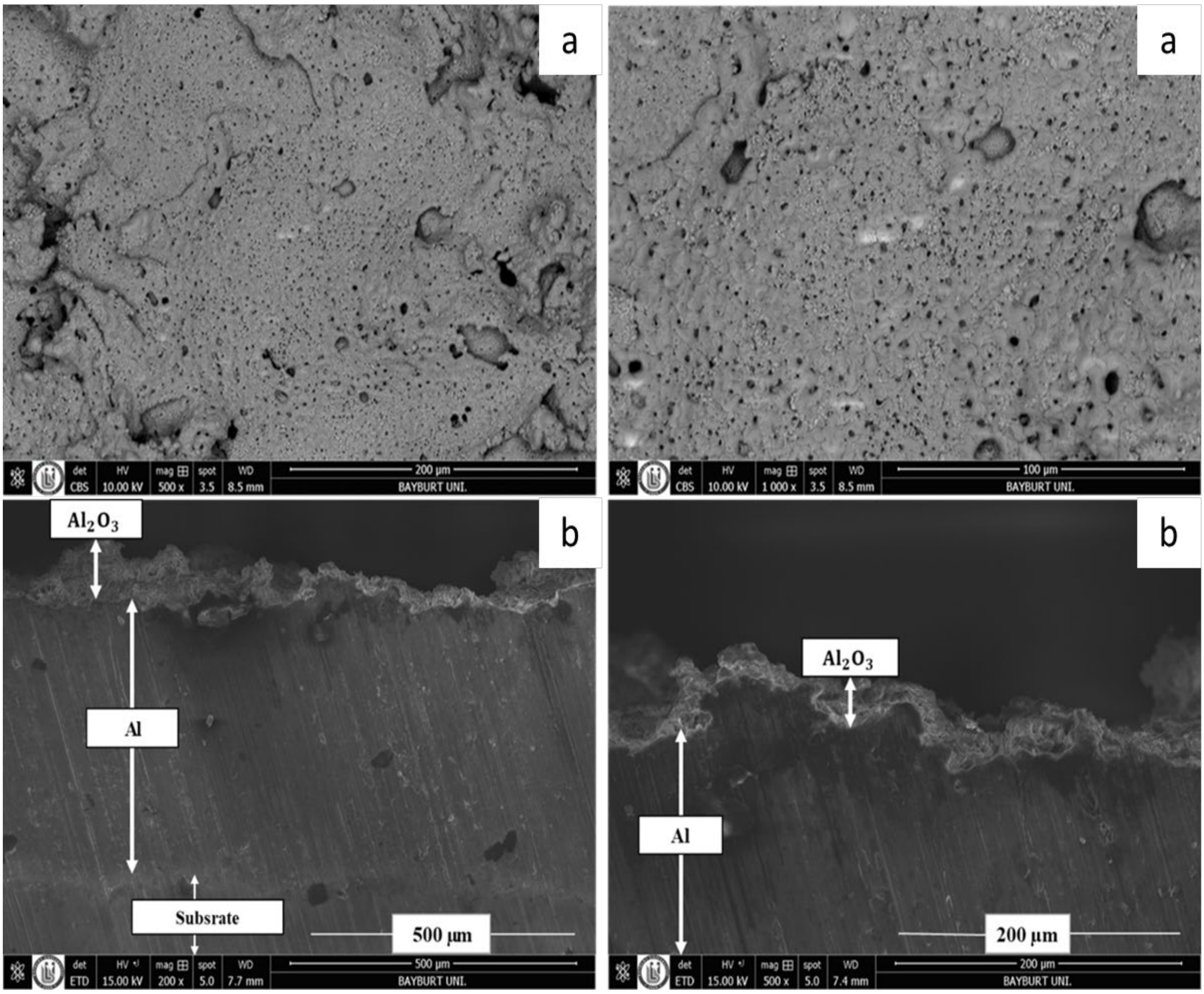


Figure 3. a) SEM images of AZ61-PEO b) SEM cross-section images of AZ61-PEO

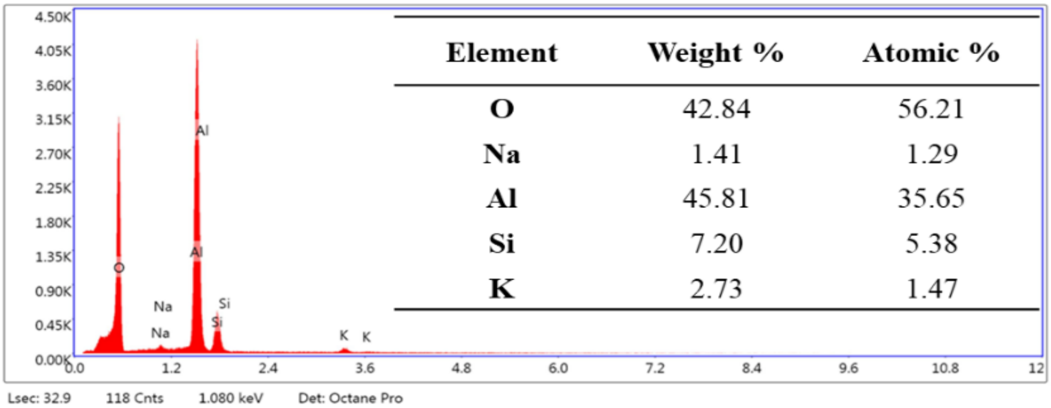
Figure 3b displays the cross-sectional micrographs of the AZ61-PEO sample. The thickness of the aluminum layer was measured to be approximately 350–400 µm, while the PEO coating on the AZ61 substrate exhibited a thickness in the range of 70–75 µm.

Figure 4: The elemental composition analysis of the AZ61-PEO specimens, conducted with an energy-dispersive X-ray spectroscopy (EDS) system coupled to a scanning electron microscope (SEM). The study determined that the coating contained 45.81% Al, 42.84% O, 7.20% Si, 2.73% K, and 1.41% Na by weight.

When the amount of the O element is considered in the EDS analyses in Figure 5, an oxide layer on the coating surface is in question. When the SEM cross-section images in Figure 4 are examined, the Al_2O_3 phase was not observed in the XRD analyses when the PEO coating thickness was considered. It was stated that the PEO coating thickness caused this situation [19]. When EDS and XRD analyses were evaluated, the presence of high-intensity Al in XRD analyses was supported by EDS analysis results. Although the Al_2O_3 phase could not be observed in XRD analyses due to the PEO coating thickness, the presence of the oxide layer was observed in EDS analyses.

3.3. Corrosion tests

The potentiodynamic curves of the base sample and the AZ61-PEO sample are shown in Figure 5. The AZ61-PEO specimen exhibited a lower corrosion current density than the uncoated substrate, while its corrosion potential shifted toward more positive values. Moreover, the anodic polarization region showed a reduction in current density, suggesting improved corrosion resistance.



Element	Weight %	Atomic %
O	42.84	56.21
Na	1.41	1.29
Al	45.81	35.65
Si	7.20	5.38
K	2.73	1.47

Figure 4. EDS results of AZ61-PEO

Table 4 summarizes the E_{cor} and I_{cor} values from the polarization tests. The findings are consistent with the trends observed in Figure 5. The corrosion current densities for the AZ61 and AZ61-PEO samples were measured at 36.29 µA/cm² and 6.53 µA/cm², respectively. The lower corrosion current density observed in the AZ61-PEO sample, compared to the untreated AZ61, indicates a significant improvement in corrosion resistance.

Table 4. E_{cor} and I_{cor} values of samples

Samples	E_{cor} (mV)	I_{cor} ($\mu A.cm^{-2}$)
AZ61	-1504	36.29
AZ61-PEO	-658.507	6.53

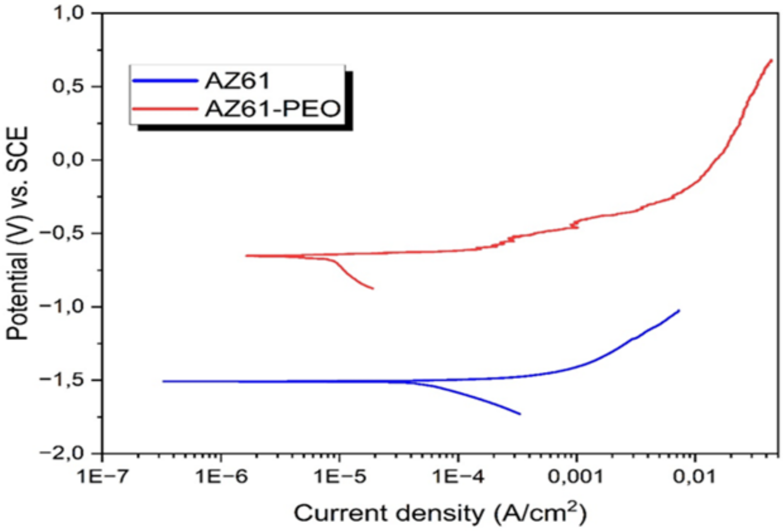


Figure 5. Polarization curves of AZ61 and AZ61-PEO

The PEO coating process is generally carried out using a constant current method, and monitoring the voltage changes in electrochemical cells over time plays a critical role in understanding the process stages. While the applied voltage directly affects coating thickness, higher voltages produce thicker coatings, but surface smoothness and coating density are negatively affected. At lower voltages, oxide coating formation becomes more difficult, reducing coating efficiency. Therefore, the correct voltage selection is crucial for coating quality and energy efficiency. Generally, increasing the treatment time results in a thicker oxide layer. However, this increase in thickness can have different effects on coating behavior. The treatment time of the PEO treatment affects not only the oxide layer thickness but also the surface roughness. The increase in coating roughness causes the average diameter of the discharge channels to increase linearly with coating time. The presence of pores is due to discharge during the PEO treatment. As the PEO treatment time increases, the number of pores decreases, while their size increases [3, 22].

The literature indicates that sodium silicate is decisive in producing homogeneous and compact coatings during the PEO process. As a result, PEO coatings obtained in silicate-based electrolytes can exhibit superior corrosion resistance [23]. It has been reported that silicate ions enhance the stability of the passive film and contribute to the inhibition of localized corrosion [24]. A comparative study investigating the effects of SiO_3^{2-}, PO_4^{3-}, MoO_4^{2-}, and AlO_2^- anions in silicate-based solutions on AZ31 alloy demonstrated that the silicate-containing electrolyte provided the highest corrosion resistance [25]. This improvement is attributed to the formation of anodic coatings by incorporating silicate ions with Mg and Al species. In addition, the ceramic oxides generated during this process are vital in improving corrosion resistance [23].

The aluminum layer applied by arc spraying provides a thick and continuous barrier that effectively prevents chloride ions from penetrating the magnesium substrate. As a result, direct anodic dissolution of Mg is strongly suppressed, and corrosion processes occur primarily on the aluminum surface. In this environment, the Al layer readily forms stable corrosion products such as Al_2O_3 and $Al(OH)_3$, which act as passive films and further inhibit ionic transport. Following PEO treatment in a Na_2SiO_3 + KOH electrolyte, the protective system is reinforced with a ceramic-rich oxide layer consisting primarily of Al_2O_3 and Si-containing phases. Because the dense Al coating significantly restricts the access of Cl^- ions to the Mg substrate, the contribution of MgO or Mg_2SiO_4 is expected to be minimal. Therefore, this bilayer structure effectively isolates the magnesium substrate from the corrosive environment, while the Al/PEO bilayer provides superior corrosion resistance compared to conventional single-layer coatings. Accordingly, in the present study, the presence of Al_2O_3 within the AZ61-PEO coating was found to play a critical role in significantly enhancing its corrosion resistance compared to the uncoated base alloy.

4. Conclusions

E_{cor} values for AZ61 and AZ61-PEO samples were -1504 mV and -658.507 mV, respectively. As a result of the PEO coating, E_{cor} values increased towards positive values compared to the base sample.

I_{cor} values for AZ61 and AZ61-PEO samples were 36.29 $\mu A.cm^{-2}$ and 6.53 $\mu A.cm^{-2}$ respectively. The AZ61-PEO showed superior corrosion resistance compared to the base sample. These findings confirm that the oxide layer formed via the PEO process showed a markedly improved corrosion resistance compared to the uncoated AZ61. Future studies could focus on the PEO solution and chemistry with particle additives (graphene, etc.) to allow magnesium alloys to exhibit superior corrosion properties. Furthermore, multilayer coatings obtained from other coating methods (PVD, thermal coatings, etc.) applied over PEO coatings could lead to superior corrosion resistance in magnesium alloys.

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No conflict of interest or common interest has been declared by authors.

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No artificial intelligence tools were used while writing this article.

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