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RESEARCH ARTICLE

Microstructure and elemental analysis of ZnO-coated AZ91D magnesium alloy via anodic oxidation coating for corrosion protection

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ABSTRACT - The AZ91D Magnesium alloy is a highly demanded structural material, valued for its exceptional mechanical and physical properties. Its versatility has led to widespread applications in industries such as automotive, aerospace, electronics, and biomedical. However, a significant limitation of AZ91D alloy is its poor corrosion resistance, which hinders broader adoption despite its advantages. To mitigate this weakness, surface treatments are essential to improve its corrosion protection. Among the various methods, anodizing stands out due to its low operational cost, short processing time, and straightforward setup. This research investigates the effect of coating time on the quality of anodic oxidation coatings with zinc oxide (ZnO) on AZ91D Magnesium alloy. The surface morphology, structural characteristics, and corrosion resistance of the ZnO-coated alloy were analyzed using Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), and corrosion testing. The findings demonstrate that anodizing effectively increased the thickness of the ZnO oxide layer on the AZ91D Magnesium alloy, significantly improving its corrosion resistance. Notably, the coating thickness increased proportionally with the duration of coating time. The sample with a 3-minute coating time exhibited the lowest corrosion rate of 2.17 mm/year, alongside the best coating quality, as confirmed by SEM, EDX, and XRD analysis. These results indicate that the elemental composition and compactness of the coating layer play a critical role in enhancing corrosion resistance.

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1. INTRODUCTION

The term 'light-weighting' has gained widespread popularity in the automotive industry. As a result, Magnesium alloy, known for its remarkable lightness with a density of 1.74g/cm^3 is to replace Aluminum and Titanium [1, 2]. However, the use of Magnesium alloy is hindered by its susceptibility to high corrosion rates. Consequently, applying surface treatments to enhance its resistance to corrosion is essential. It is widely acknowledged that exposing Magnesium alloys to harsh environments during processing and storage can trigger electrochemical corrosion [3] . Therefore, preventing the initial stages of corrosion in the preparation process is vital for ensuring robust corrosion protection for Magnesium alloy substrates. In recent decades, a variety of surface treatment techniques have been utilized to improve the corrosion resistance of Magnesium alloys [4-13]. Among these techniques, anodic oxidation coating, commonly known as anodizing, stands out as the most frequently used surface treatment for Magnesium alloy. This method is favored for its short processing time, ease of operation, and cost-effectiveness [14]. Through the process of anodic oxidation coating, a substantial oxide film forms on the surface of the Magnesium alloy, serving as a protective layer. The anodic oxidation coating technique involves immersing the substrate in pre-treatment and treatment baths and applying electric power to achieve the growth of the oxide film on the substrate surface. This growth is facilitated by the interplay between chemical dissolution and precipitation within the chemical solution and the substrate material, aided by supply of electrons through electric power.

Traditionally, coating solutions have included components such as Chromate, Carbonate, Phosphate, and Fluoride. Chromate-based coatings have long been recognized for their excellent corrosion protection of Magnesium alloys. However, their usage has been restricted due to concerns regarding the carcinogenic and toxic nature of Hexavalent Chromate compounds [15]. In contrast, various researchers have explored eco-friendly alternatives that are less toxic than Chromate conversion coatings. These alternatives have demonstrated their effectiveness in protecting Steel and Zinc from corrosion in diluted aqueous solutions [16]. Researchers have also investigated the impact of different electrolytes on the quality of coatings. Among these methods, the incorporation of corrosion inhibitors has gained popularity due to several advantages, including ease of preparation, cost-effectiveness, and high efficiency [17, 18]. Within the wide spectrum of corrosion inhibitors, ionic liquids (ILs) emerge as promising environmentally friendly inhibitors for material protection, mainly owing to their exceptional solubility and high thermal stability [19]. Additionally, their unique molecular structure, comprising both anions and cations, enables ILs to readily adhere to metal surfaces, thus showcasing exceptional

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corrosion inhibition properties when applied to metal substrates exposed to corrosive environments [20]. Amongst the existing ILs, ZnO stands out as one of the most promising materials due to its exceptional properties, including thermal and mechanical stability at room temperature, strong physical and chemical resilience, eco-friendliness, widespread availability, and affordability [21]. There are reports highlighting the use of ZnO/TiO_2 [22], polylactic acid/ZnO [23], and polypyrrole/ZnO [24] as corrosion inhibitors in various corrosive environments.

Therefore, in this study, the application of a ZnO-based corrosion inhibitor for AZ91D Magnesium alloy through anodic oxidation coating is investigated. The Zinc Nitrate aqueous solution was used to produce oxide film on AZ91D Magnesium alloy and the oxide film thickness was controlled by manipulating the coating time. The correlation between Zn content and oxide film thickness to the corrosion resistance of AZ91D Magnesium alloy is also discussed.

2. METHODS AND MATERIALS

AZ91D Magnesium alloy samples were used in this study. The chemical composition of the as-received AZ91D Magnesium alloy is listed in Table 1. The Magnesium alloy ingot was cut into square samples, measuring 10 mm × 10 mm × 10 mm. The sample was then mounted with epoxy resin, leaving only one exposed surface of 10 mm × 10 mm. Then, The sample surfaces were sequentially polished using 600, 1000, and 1200 grit waterproof abrasive paper. Next, the samples were degreased in ethanol for 3 minutes. Finally, the samples were rinsed with deionized water and immediately dried. All chemicals were supplied by Kanto Electronics Chemicals (M) Sdn. Bhd.

The shcematic illustration of anodic oxidation coating on AZ91D Magnesium alloy is shown in Figure 1. The anodic films on AZ91D Magnesium alloy were produced using a constant voltage of 20 V for various coating times of 1, 3 and 5 minutes. The samples served as the anode, while a stainless steel plate acted as the cathode. The electrolyte consisted of 0.3 M $Zn(NO_3)$ aqueous solution. All chemicals were supplied by Kanto Electronics Chemicals (M) Sdn. Bhd. The electrolyte temperature was maintained at room temperature. After the oxidation process, the samples were rinsed with distilled water and dried in an oven for 24 hours at 70 °C.

Table 1. Chemical composition of AZ91D Magnesium alloy (wt.%)

Element	Al	Zn	Mn	Si	Cu	Fe	Mg
Content (wt.%)	8.3	0.35	0.13	0.10	0.030	0.005	Balance

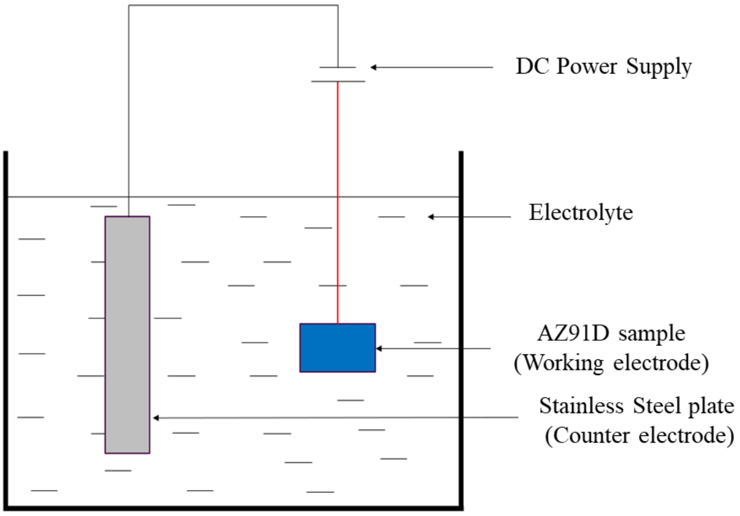

Figure 1. Schematic illustration of anodic oxidation coating on AZ91D Magnesium alloy

The corrosion test was performed by immersing the samples in NaCl solution for 72 hours, at a constant temperature of 35 ± 2°C. The corrosion rate of AZ91D Magnesium alloy at different coating times was evaluated by analyzing the weight change before and after the immersion test of the sample based on the use of Eq. (1).

$$R = \frac{8.76 \times 10^4 \times W}{A \times D \times t} \tag{1}$$

where, W, A, D, and t are weight loss (in gram), active area (in cm^2), density (in g/cm^3), and immersion time (in hours), respectively.

A TM3000 Scanning Electron Microscope (SEM, Hitachi) was used to observe the surface and cross-sectional morphologies of the coatings and Energy dispersive X-ray (EDX) was employed to analyze the element species and element distribution on the surface. The phase composition of the coatings was determined by an X-ray diffractometer

(XRD, Bruker D2 Phaser) under the Cu Kα radiation of 30 kV and 10 mA, and the detection angle was set at a 2θ range of 10°– 80° with a step size of 0.1 s/step.

3. RESULTS AND DISCUSSION

3.1 Microstructural Characterization

Figure 2 shows the SEM images of the anodized samples, providing insights into the varied morphologies achieved with different coating times. The uncoated sample in Figure 2(a) exhibited grinding scratches with α matrix and β phase (Mg17Al12). The surface morphology of the sample changed significantly, and it became an irregular porous microstructure with a relatively rough surface after anodizing. The 3- min coating sample revealed a dense, solid layer with fewer microscopic pores. However, the 5-minute coating sample had larger pores on the coating surface. Since smaller pores are better for corrosion resistance, a shorter coating time is advantageous for the coating's quality.

Due to the thick layer that generally precipitates on the metal surface, the average coating thickness appears to rise with increasing coating time. Figure 3 shows the relationship between coating thickness and coating time. According to Figure 3, it is evident that the growth of coating thickness rose from 10.82 µm to 20.25 µm when coating time increased from 1 to 3 min before reaching the peak at 56.66 µm at 5 min. This finding agrees with A. Basheer Ahmed [25], who found that the coating thickness of AZ31D Magnesium alloy in 3M KOH solution increases with an increase in anodizing time.

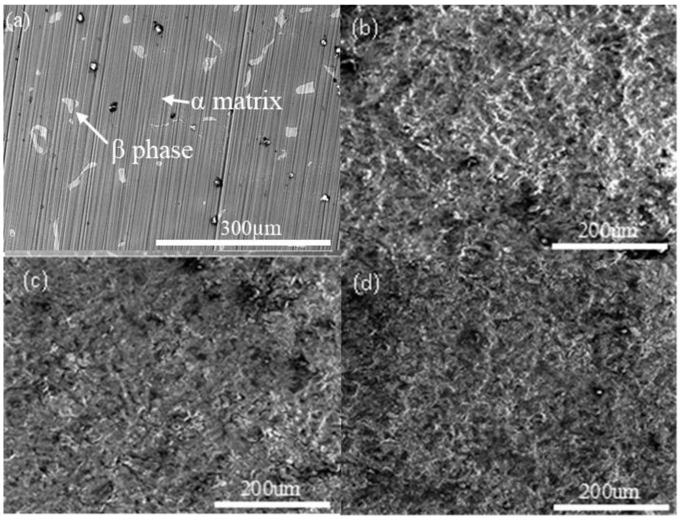

Figure 2. SEM morphology of the samples: (a) uncoated and after coating with, (b) 1 min, (c) 3 min, and (d) 5 min of coating time

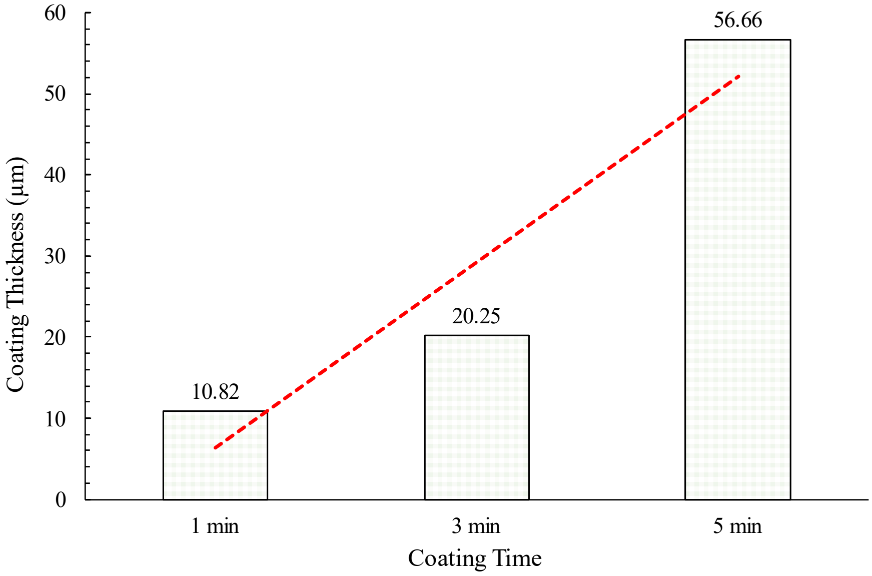

Figure 3. Coating thickness of anodized samples at different coating times

Table 2. EDX Analysis of samples before and after coated with ZnO

Coating Time	Mass Percentage of Element (wt.%)			
	Mg	Zn	O	Al
Uncoated	88.70	0.30	3.30	7.60
1 min	25.38	1.32	47.13	6.63
3 min	26.42	2.73	43.38	7.97
5 min	26.18	1.49	44.31	7.88

Table 2 presents the results of the EDX analysis, which includes the mass percentages of Mg, Zn, O, and Al at different coating times. The analysis indicates that the mass percentage of Magnesium (Mg) significantly decreased after the substrate was coated with ZnO, while the mass percentage of Oxygen (O) substantially increased. These results suggest that an oxide film grew on the surface of the substrate. Furthermore, the uncoated sample had a native Zn content of only 0.3 wt.%. After anodizing the sample with a ZnO coating, the Zn content gradually increased and reached a peak of 2.73 wt.% after 3 minutes of coating. However, when coated for 5 minutes, the Zn content decreased to 1.49 wt.%. The mass percentage of Aluminum (Al) remained consistent across all samples, but it reached its highest value during the 3-minute coating time. The results from the EDX analysis consistently showed that the optimal coating time with the highest Zn content was 3 minutes.

Figure 4 shows the XRD patterns for uncoated and coated samples with different coating times. There are two peaks assigned for magnesium have been detected at the diffraction peak of $2\theta = 32.4°$ and $36.6°$ for the uncoated sample. After anodizing, a significant peak at $2\theta = 36.8°$ corresponding to ZnO (101) could be observed with the coated samples, and the peak increased as the coating increased showing better crystallinity. However, the peak for the 3 min coating is somewhat low at $2\theta = 36.8°$, another ZnO (201) peak is still visible at $2\theta = 69.2°$, indicating a polycrystalline structure.

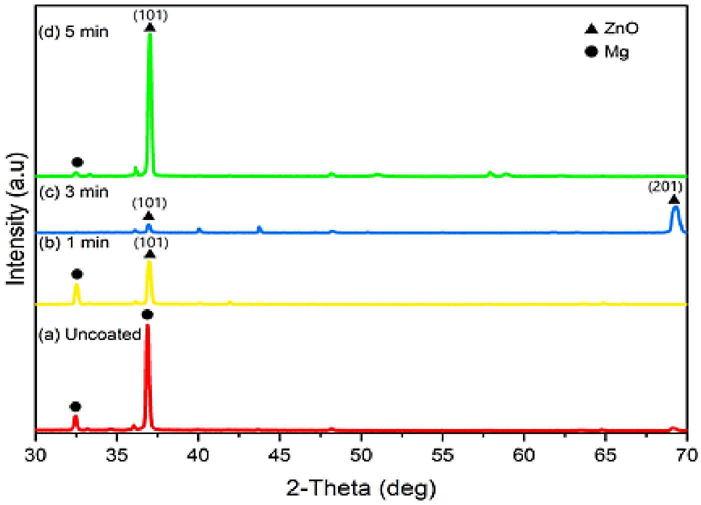

Figure 4. XRD spectra of Zinc Oxide (ZnO) coating on samples: (a) uncoated and after coating with, (b) 1 min, (c) 3 min, and (d) 5 min of coating time

3.2 Corrosion Analysis

In this corrosion analysis section, the surface sample was analyzed again using SEM, and XRD after undergoing an immersion test. Figure 5 clearly shows that anodic oxidation coating improves the corrosion resistance of the AZ91D Magnesium alloy substrate. Without the ZnO coating, the uncoated sample had the highest corrosion rate of 22.54 mm/year. However, applying a ZnO coating to the substrate's surface reduced the corrosion rate to 9.24 mm/year after just one minute of anodizing. The lowest recorded corrosion rate of 2.17 mm/year was achieved with a 3-minute coating time, representing the best outcome. However, the corrosion rate accelerated after 5 minutes of anodizing even though its coating thickness was the highest compared to other samples.

Figure 6 shows the SEM images of the uncoated and coated samples with different coating times that experienced corrosion after the salt immersion test. Initially, the surface was smooth and slightly scratched from sample preparation changed to an extremely porous oxide layer with cracks and holes. Similarly, samples coated for 1, 3, and 5 min were entirely covered in oxide surface, but their anodized layers have become more inconsistent, displaying additional cracks and holes. Notably, the anodized sample with a coating time of 3 min exhibited less damage than the other coated samples of 1 and 5 min.

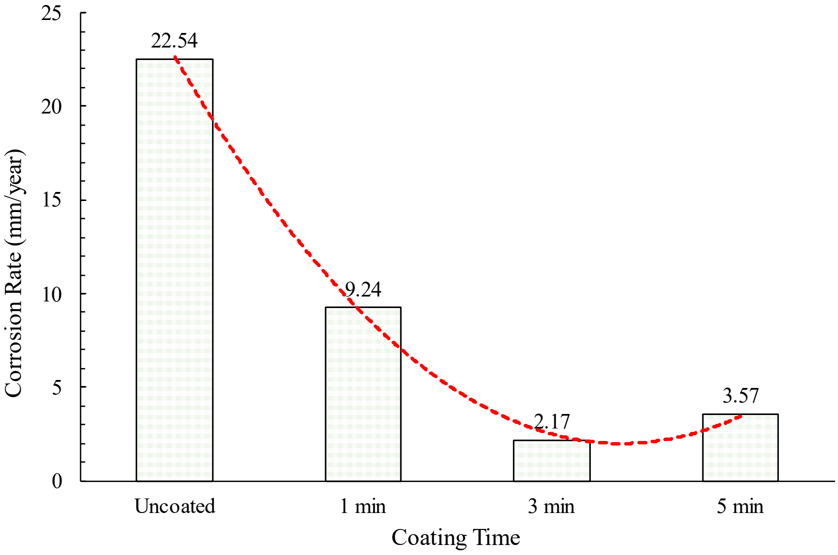

Figure 5. Corrosion rate of samples at different coating times after the salt immersion test

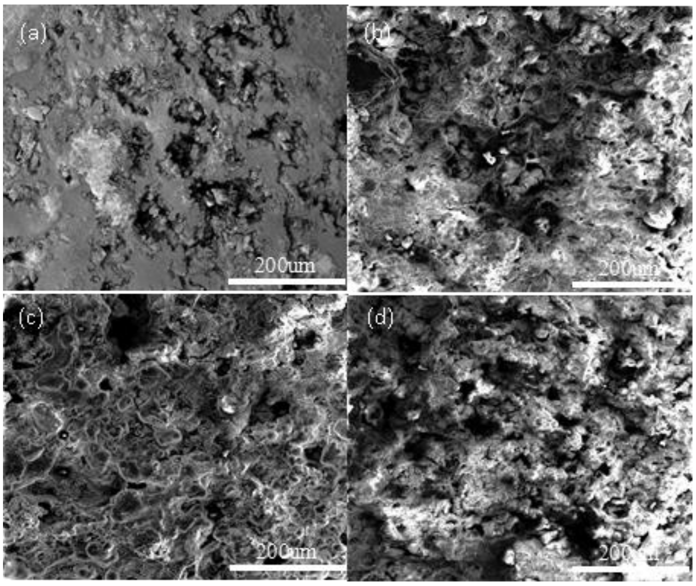

Figure 6. SEM morphology of samples after salt immersion test: (a) uncoated, (b) 1 min, (c) 3 min, and (d) 5 min of coating time

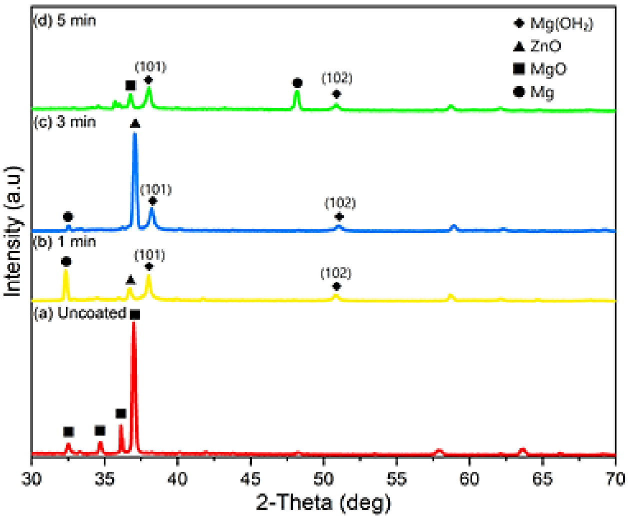

Figure 7. XRD spectra of the uncoated sample: (a) and coated samples at, (b) 1 min, (c) 3 min, and (d) 5 min of coating time after the salt immersion test

The XRD patterns for samples after the salt immersion test are shown in Figure 7. The uncoated sample only had Magnesium Oxide (MgO) on its surface. However, the coated samples regardless of the coating time had peaks for Magnesium Hydroxide ($Mg(OH)_2$), Zinc Oxide (ZnO), and MgO. After the chemical reaction with NaCl solution during the immersion test, the corrosion product of the coated samples consists of $Mg(OH)_2$, ZnO, and MgO which shows that these chemical compounds have inhibited the corrosion on AZ91D Magnesium alloy. The XRD pattern for the 3 min coating sample revealed the most promising outcome when compared to other samples due to the significant peak of ZnO detected at $2\theta = 37.03°$ [26] further demonstrating the compact formation of ZnO coating, as described in earlier surface morphology and structural studies, which disclose the higher corrosion resistance for AZ91D Magnesium alloy.

3.3 Correlation of Coating Time of Film Thickness, Film Composition, and Corrosion Rate

The mechanism of anodic oxidation can be influenced by a range of factors, including the type of substrate, the composition of the electrolyte solution, and the voltage and current applied during the process. Typically, anodic oxidation begins with the formation of a thin oxide layer on the surface of the substrate. As the process continues, the thickness of the oxide layer gradually increases, which in turn can lead to the development of a more substantial coating. However, it's worth noting that the thickness of the coating may be limited by factors such as the solubility of the oxide in the electrolyte solution. At some point during the anodic oxidation process, the oxide layer may become destabilized due to changes in the pH of the electrolyte solution. This can result in the breakdown of the coating, as well as the release of gases such as hydrogen and oxygen. The precise nature of this destruction phase depends on various factors, such as the specific properties of the substrate and the electrolyte solution. However, in general, the process can result in the formation of cracks, pores, or other defects in the coating. These defects can be undesirable, as they can reduce the coating's protective properties and make it more vulnerable to corrosion or other types of damage [27].

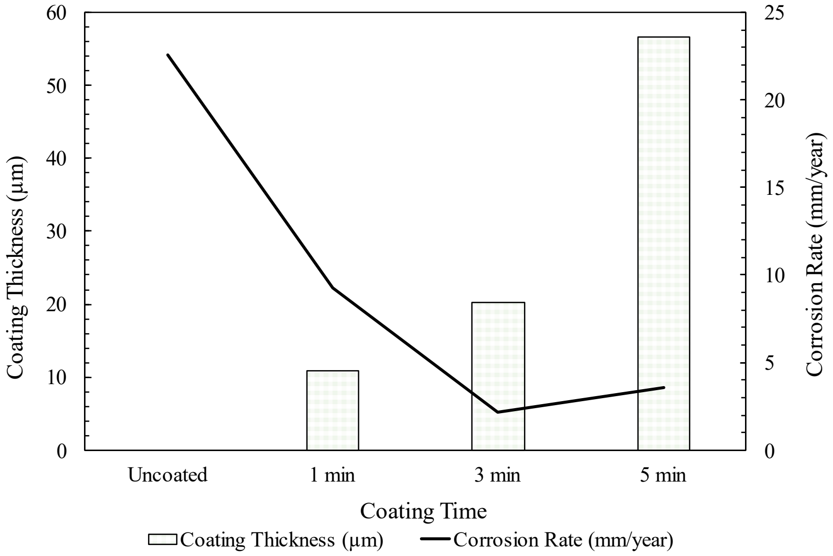

Figure 8. Relationship of coating thickness and corrosion rate to coating time

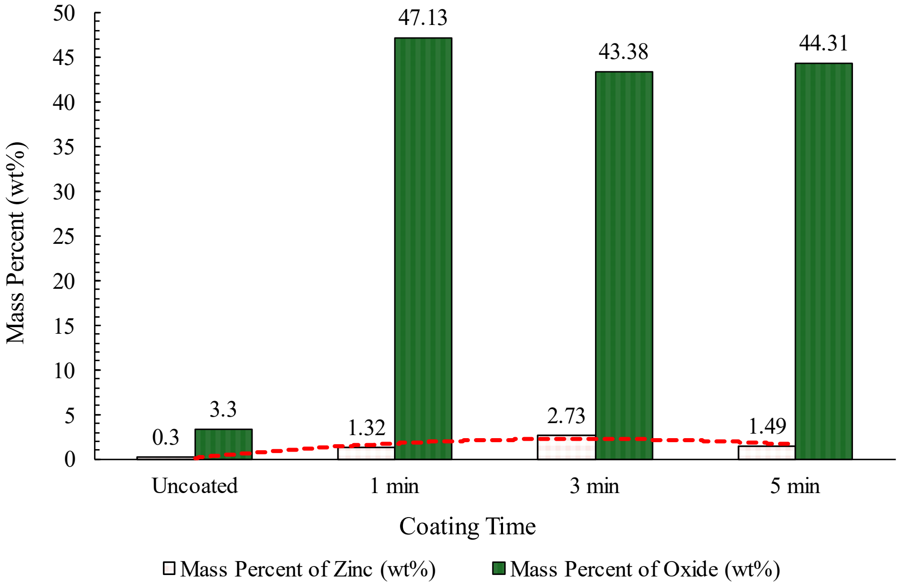

Figure 9. Coating composition of coating sample at different coating times

Based on the cumulative corrosion rate depicted in Figure 8, it is concluded that the sample coated for 5 min has reached the destruction stage. This is consistent with the research done by A. Zaffora et al. [28], who discovered that a prolonged anodizing time may harm the coating's inner layer. Additionally, it led to the development of a layer of porosity that was thicker, which would adversely affect corrosion resistance. Figure 9 shows the elemental composition of the coating sample at different coating times. Prior to reaching 3 minutes of coating time, the mass percentage of zinc increased with the increment of coating time. However, it is believed that when the coating duration reached 5 min, it had already entered the destruction stage because both the Zinc element and the rate of corrosion began to slow down. Only 3.30 wt.% of the uncoated sample's content was oxide, and that oxide came directly from the substrate. Because of the chemical reaction that took place during the anodizing process, the amount of oxide was greatly increased after coating with ZnO.

The results of the experiment indicate a direct correlation between the duration of ZnO coating and the mass percentage of zinc, which influences the corrosion rate. This aligns with the findings of Wei He et al. [29], who studied the impact of zinc content in AZ91D Mg alloy substrates on the corrosion resistance of micro-arc oxidation (MAO) coated layers. In their research, the coating layers were produced using a MAO-polycaprolactone-modified ZnO composite coating, and corrosion resistance improved with higher zinc content, as evidenced by 20-hour exposure tests in simulated body fluid (SBF).

4. CONCLUSIONS

Anodic oxidation coatings of the ZnO-coated AZ91D Magnesium alloy were prepared in the electrolyte consisting of $Zn(NO_3)_2$ with 1, 3, and 5-minute oxidation times. The following conclusions were drawn from the study of the microstructure, phase composition and corrosion resistance of the coatings:

i) The sample with a coating time of 3 min showed the best compactness with the lowest porosity, the fewest defects, and an optimal coating thickness.

ii) The anodic oxidation coated-AZ91D magnesium alloy formed in the $Zn(NO_3)_2$ electrolyte was mainly composed of ZnO.

iii) The 3-min anodic oxidation coated-ZnO coating on AZ91D magnesium alloy had the best corrosion resistance of 2.17 mm/y under salt immersion testing.

iv) After the salt immersion test, the coated AZ91D magnesium alloy produced ZnO, MgO, and $Mg(OH)_2$ compounds for corrosion protection against the salt solution.

Overall, the experimental results demonstrated that the corrosion resistance of the anodic oxidation coating on AZ91D magnesium alloy was strongly correlated with the elemental composition and oxide layer compactness over film thickness.

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CONFLICT OF INTEREST

The manuscript has not been published elsewhere and is not under consideration by other journals. All authors have approved the review, agree with its submission and declare no conflict of interest on the manuscript.

AUTHORS CONTRIBUTION

L. Y. Vee (Conceptualization; Methodology; Writing - Original Draft Preparation)
Z. Iberahim (Data Curation; Formal Analysis; Validation)
M. Zamzuri (Supervision; Conceptualization; Resources)
N. Zainon (Conceptualization; Methodology)
H. Jaafar (Data Curation; Validation)
A. F. Aiman (Writing-Reviewing and Editing)
C. C. Lee (Characterization; Data Interpretation)

AVAILABILITY OF DATA AND MATERIALS

The data supporting this study’s findings are available on request from the corresponding author.

ETHICS STATEMENT

This research complies with the ethical guidelines of Universiti Malaysia Perlis. Since the study does not involve human participants, animals, or hazardous materials, no specific ethical approval was necessary.

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