

Article

Influence of Titanium Coating Thickness on Microstructure, Residual Stress, and Corrosion Behaviour of Magnetron-Sputtered WE43 Magnesium Alloy

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Abstract

Magnesium alloys are attractive for temporary orthopaedic applications because of their biodegradability and favourable mechanical properties; however, their relatively rapid degradation under physiological conditions remains a challenge. In this study, the microstructural evolution, residual stress and electrochemical corrosion behaviour of WE43, a rare earth-containing magnesium alloy with yttrium, neodymium and zirconium as alloying elements, coated with titanium, were investigated as functions of deposition time using direct-current magnetron sputtering. Titanium coatings were deposited for 1, 1.5, 2 and 3 h, producing coating thicknesses of approximately 500, 650, 1000 and 1400 nm, respectively. Field-emission scanning electron microscopy, atomic force microscopy and grazing-incidence X-ray diffraction revealed progressive changes from fine-grained to dense and, subsequently, coarse-grained morphologies with increasing deposition time. The 2 h coating exhibited the largest crystallite size (27.17 ± 3.42 nm) and a moderate compressive residual stress of 605.3 ± 15.11 MPa. Potentiodynamic polarisation measurements showed that the 2 h coating produced the lowest corrosion current density (0.133 ± 0.026 mA/cm²) and calculated corrosion rate (2.93 ± 0.57 mm/year), representing a 67% reduction relative to independently measured, uncoated WE43 (8.87 ± 1.11 mm/year). The 3 h coating exhibited a higher compressive residual stress of 992.4 ± 21.7 MPa and a higher corrosion rate of 4.81 ± 0.74 mm/year, accompanied by localised microcracking after corrosion testing. Contact-angle measurements performed on the uncoated alloy and the 2 h coating showed an increase from $75.0 \pm 2.1^\circ$ to $83.0 \pm 1.8^\circ$. Overall, the results indicated that corrosion performance was governed by the combined effects of coating morphology, crystallographic development and residual stress, with the 2 h deposition condition providing the most favourable balance under the present experimental conditions.



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Keywords: magnesium alloy; WE43; magnetron sputtering; coating thickness; residual stress; corrosion behaviour

1. Introduction

Titanium alloys and stainless steels are widely used for the fixation of bone fractures or the repair of bone deformities, due to their superior mechanical and corrosion-resistant properties [1,2]. However, the need for secondary surgery to remove implants leads to an

increase in cost and risks to patients. Hence, the development of biodegradable materials is a desirable advancement among various materials, such as magnesium alloys. Magnesium (Mg) alloys, particularly WE43, a rare earth-containing magnesium alloy, are promising materials for temporary orthopaedic applications because of their biodegradability, favourable compatibility with bone tissue and potential for use in temporary implants [3]. Biodegradable Mg alloys can also avoid the need for secondary implant-removal surgery associated with permanent metallic implants [4]. However, under physiological conditions, the degradation rate of WE43 can exceed the rate of bone healing, which may compromise its mechanical integrity and lead to premature failure [5]. Using techniques such as Filtered Cathodic Vacuum Arc (FCVA), zinc (Zn) coatings have exhibited beneficial effects; however, their thickness-dependent behaviours concerning both corrosion resistance and mechanical strength necessitate more systematic investigation [6,7]. DLC coatings applied via FCVA technology are emerging, but their specific application to WE43 and the impacts of deposition parameters, such as negative bias voltage, on their properties are underexplored [8]. Defects like pores and microcracks, along with poor adhesion and residual stress due to material mismatches, remain key challenges [6,8]. Finally, a significant gap exists in the provision of long-term testing under simulated body fluid (SBF) conditions and quantitative evaluation of hydrogen gas evolution [4,5]. To mitigate this, several surface modification techniques [9–14], like PECVD (Plasma-Enhanced Chemical Vapour Deposition), have been explored. Among these techniques, Magnetron sputtering has emerged as a leading technique for depositing thin, uniform, and adherent films on magnesium (Mg) alloys, particularly WE43, for biomedical applications. Research highlights the effectiveness of Ti-based coatings in significantly enhancing corrosion resistance and mechanical strength [12,15], aligning with broader insights into how PVD coatings improve implant integration [16–20]. Surface coatings such as SnO₂ and TaN have demonstrated improved corrosion resistance and biocompatibility [21–25]. For that, Surface treatments, such as metal ion implantation [23,26], and coatings such as Ni-P [24], calcium phosphate [25,27], and bioceramics [28,29], are used to improve corrosion resistance and biocompatibility. These modifications enhance the alloy's performance for implant applications [23–30]. However, comparative studies are limited, and issues such as coating defects and the optimisation of coating parameters remain. Further research is needed to optimise these coatings for biomedical use. Most studies focus on either corrosion resistance or biocompatibility improvements in WE43 Mg alloy, but few address both simultaneously. Recent investigations have shown that magnetron-sputtered coatings can improve the corrosion behaviour of WE43 magnesium alloy; however, the effects of deposition duration on coating growth, residual stress and the resulting corrosion response remain insufficiently established [31]. In our previous study, a single 2 h Ti deposition condition was investigated as an initial proof-of-concept for magnetron-sputtered Ti coating on WE43 [31]. That study established the feasibility of Ti coating for improving the initial corrosion response of WE43, but did not systematically examine the effects of deposition duration on coating thickness, microstructural development and residual stress, or their relationships with corrosion behaviour. The present work is therefore a parametric extension of that study, with deposition durations of 1, 1.5, 2 and 3 h used to examine coating growth, morphological and crystallographic evolution, residual stress, and electrochemical corrosion behaviour. The uncoated WE43 baseline used for the present comparison was obtained independently from newly conducted triplicate experiments, rather than taken from the previously published study.

The objective of the present study is to establish the relationships among deposition duration, coating thickness, microstructural evolution, residual stress and initial electrochemical corrosion behaviour of Ti-coated WE43. In particular, this study examines whether increasing coating thickness produces a continuous improvement in corrosion performance,

or whether changes in microstructure and residual stress result in an optimum condition within the investigated deposition range.

The novelty of the present work lies in the systematic comparison of four deposition durations and in correlating coating thickness, morphology, crystallite development and residual stress with the measured electrochemical response. The results identify the 2 h deposition condition, corresponding to a coating thickness of approximately 1000 nm, as the condition providing the most favourable combination of microstructural development and residual stress, together with the lowest measured corrosion current density and calculated corrosion rate under the present test conditions. The 3 h coating, despite its greater thickness, exhibited substantially higher compressive residual stress and localised microcracking after corrosion testing. These observations demonstrate that increasing coating thickness does not necessarily improve the initial corrosion performance of Ti-coated WE43.

2. Materials and Methods

The WE43 magnesium alloy, a rare earth-containing magnesium alloy, was procured from Magnesium Elektron, Flemington, NJ, US, and used as the substrate in this study. Specimens were cut (10 mm × 10 mm × 5 mm) using a wire cut Electric Discharge Machine (w-EDM). Surface preparation involved stepwise polishing with emery papers ranging from 400 to 3000, followed by ultrasonic cleaning in ethanol. Final fine polishing was skipped to retain surface roughness, which promotes mechanical interlocking and enhances coating adhesion [32].

Thin film deposition was performed in a multi-target magnetron sputtering system (Make: Moorfield, Knutsford, Cheshire, United Kingdom. Model: MiniLab 060) (Figure 1a), which contained five magnetrons within one chamber. A high-purity (99.9%) titanium target (2" diameter, 0.250" thickness) was mounted in one of the Direct Current (DC) magnetron sources. A schematic explaining the mechanism of sputtering is presented in Figure 1b. The process chamber was vacuumed to a pressure of 10^{-7} mbar, and argon gas was flowed at 20 sccm to maintain a 0.5 Pa working pressure. The plasma generated at a DC power of 200 W. During deposition, samples were mounted on a sample holder rotating (10 rpm) in process chamber, with a target distance of 100 mm to ensure film uniformity. Pre-sputtering was performed for 10 min prior to deposition, using the Titanium target to remove surface contaminants, and ion-etching was performed for 5 min on WE43 substrates to enhance coating adhesion. Sputtering was carried out without active substrate heating. The substrates were initially at room temperature; however, plasma heating during deposition caused the substrate temperature to increase with deposition duration. The temperatures measured at the end of the deposition runs were approximately 35 °C, 45 °C, 60 °C and 72 °C for deposition durations of 1, 1.5, 2 and 3 h, respectively. Thus, deposition duration was treated as the controlled processing parameter, while the associated increase in substrate temperature was recognised as a coupled process variable. No post-deposition thermal treatment was applied, allowing the microstructural and residual stress states produced during deposition to be evaluated in the as-deposited condition.

The microstructural analysis and coating thickness were measured using a FESEM (Make and Model: Carl Zeiss, Jena, Germany, Gemini 500). The coating thickness was also verified using a Stylus profilometer (Make: KLA, Milpitas, CA, USA. Model: Alpha D600) as a step height in contact mode. Surface topography of the coated material was performed using an Atomic Force Microscope (Make: Bruker, Billerica, MA, USA. Model: Dimension Icon).

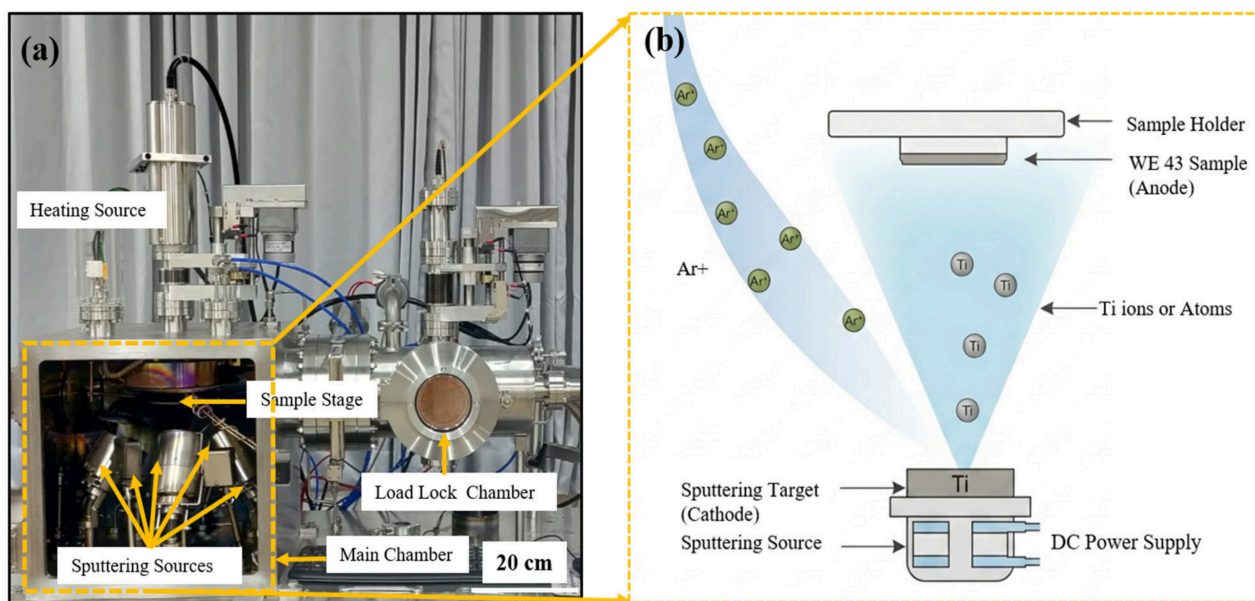


Figure 1. Magnetron sputtering machine. (a) Sputtering main chamber; (b) schematic explaining the deposition mechanism.

X-ray diffraction (XRD; Bruker D8 Advance) using Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$) was used for phase identification of the WE43 substrate. The substrate diffraction pattern was recorded over the 2θ range from 20° to 80° . The residual stress measurements of the Ti-coated specimens were performed separately using the Bragg–Brentano configuration over a 2θ range of $60\text{--}75^\circ$ at a scanning rate of $0.1^\circ/\text{s}$.

Electrochemical tests were performed (Figure 2) to evaluate the corrosion behaviours of the Ti-coated WE43 specimens. The measurements were conducted using a three-electrode cell comprising high-purity graphite as the counter electrode, Ag/AgCl as the reference electrode and the Ti-coated specimen as the working electrode. Potentiodynamic polarisation (PDP) measurements were conducted over a potential range of -250 to $+250 \text{ mV}$ at a scan rate of 1 mV/s . The corrosion current density (i_{corr}) was determined by Tafel extrapolation of the anodic and cathodic branches. For each deposition condition, three independently prepared specimens were evaluated ($n = 3$), and quantitative results are reported as mean $\pm$ standard deviation. Electrochemical measurements were performed on independent specimens for each condition. Surface and structural characterisation measurements were obtained from multiple locations where applicable to account for local variation. Representative FESEM, AFM, XRD and potentiodynamic polarisation profiles are presented in the corresponding figures. Measurements obtained from multiple locations within an individual specimen were treated as within-specimen observations and were not considered independent experimental replicates. Measured experimental results are reported as mean $\pm$ standard deviation where replicate measurements were available, whereas nominal specimen dimensions, deposition parameters and instrument operating conditions are reported as specified or recorded values. According to ASTM G102 [33], the corrosion rate (CR) was calculated in mm/year using the following equation [34]:

$$C.R = C \frac{i_{\text{corr}} \times EW}{\rho} \quad (1)$$

where C is the conversion constant, i_{corr} is the corrosion current density ($\mu\text{A}/\text{cm}^2$), EW is the equivalent weight of WE43 (12.15), and ρ is the density of the WE43 magnesium alloy ($1.8 \text{ g}/\text{cm}^3$). The deposition duration was used as the controlled processing parameter,

while the associated increase in substrate temperature during deposition was recognised as a coupled process variable. All other sputtering parameters were kept constant to enable comparison among the deposition conditions. Simulated body fluid (SBF) was prepared according to the reported procedure [35] and maintained at 37 °C during the electrochemical and wettability measurements. Wettability measurements were performed using a contact-angle analyser (Krüss Advance, Borsteler Chaussee, Hamburg, Germany). The uncoated WE43 baseline, including the electrochemical corrosion-rate data reported in the manuscript were obtained from newly conducted independent triplicate experiments to ensure direct comparability with the Ti-coated specimens.

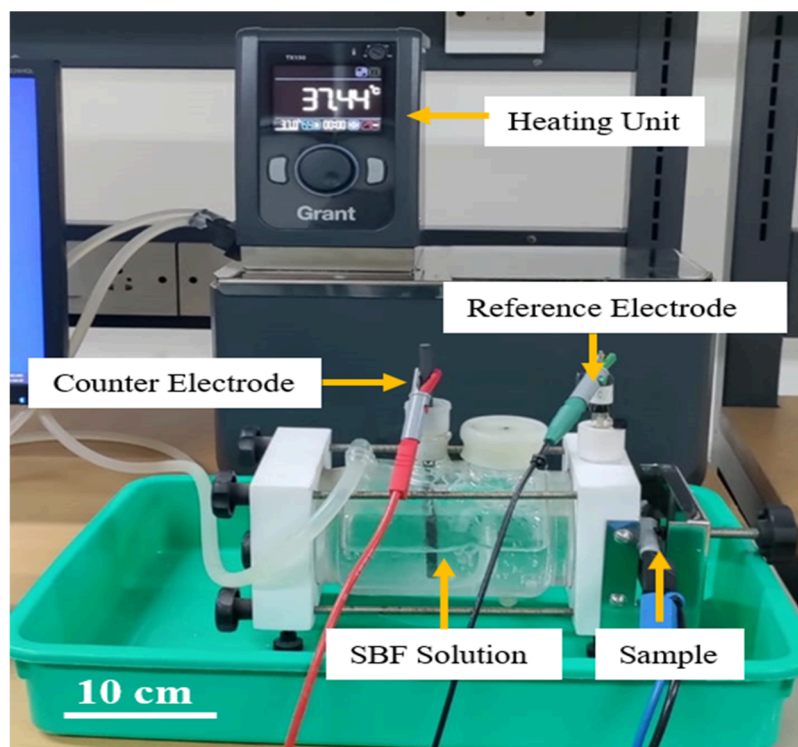


Figure 2. Experimental setup of the three-electrode electrochemical cell used for potentiodynamic polarisation (PDP) measurements of Ti-coated WE43 magnesium alloy in simulated body fluid (SBF), showing the heating unit, reference electrode, counter electrode, working electrode (sample), and electrolyte.

3. Results and Discussion

The results are discussed by correlating the evolution of coating thickness with changes in surface morphology, crystallographic characteristics, residual stress and electrochemical behaviour. Rather than evaluating each characterisation independently, the discussion establishes the processing–microstructure–property relationship responsible for the corrosion behaviour of Ti-coated WE43 alloy.

3.1. Base Material Characterisation

The surface morphology of the WE 43 Mg alloy was observed under optical microscopy and is shown in Figure 3a. It reveals a characteristic polycrystalline structure, with grains appearing relatively uniform and providing a stable foundation for the sputtering process. Atomic force microscopy (AFM) was used to characterise the surface texture at the nanoscale (Figure 3b). The 3D topographical images illustrate the localised peaks and valleys inherent to the sample after mechanical polishing. The average roughness was 8.98 ± 0.92 nm, which is low and critical for ensuring high interfacial adhesion and the formation of a

dense coating film during the nucleation stage of the sputtering process. The EDS analysis was performed to determine the chemical composition of WE43, which confirmed Y-4.0%, Nd-2.3%, Zr-0.4%, and the remaining Mg by weight (Figure 3c). The crystalline structure was confirmed using PXRD analysis (Figure 3d). The identified phases were matched with the corresponding ICDD/JCPDS reference cards: Mg (PDF-00-004-0770), Mg_{24}Y_5 (PDF-01-074-6495), $\text{Mg}_{16}\text{Nd}_{64}\text{Y}_{19}$ (PDF-01-085-8817) and Zr (PDF-00-005-0665). The diffraction shows sharp, high-intensity peaks corresponding to the α -magnesium phase (HCP structure). Additionally, secondary intermetallic phases were detected, including Mg_{24}Y_5 and $\text{Mg}_{16}\text{Nd}_{64}\text{Y}_{19}$. The precipitate Mg_{24}Y_5 is typically distributed (darker, network-like lines separating the grains) along the grain boundaries, and $\text{Mg}_{16}\text{Nd}_{64}\text{Y}_{19}$ is distributed inside the grains (small, bright clusters). These precipitates play vital roles in the mechanical strength and degradation kinetics of the base material. For the base WE43 substrate, FESEM, AFM, EDS and XRD characterisations were repeated at representative locations where applicable, and Figure 3 presents representative results from these measurements.

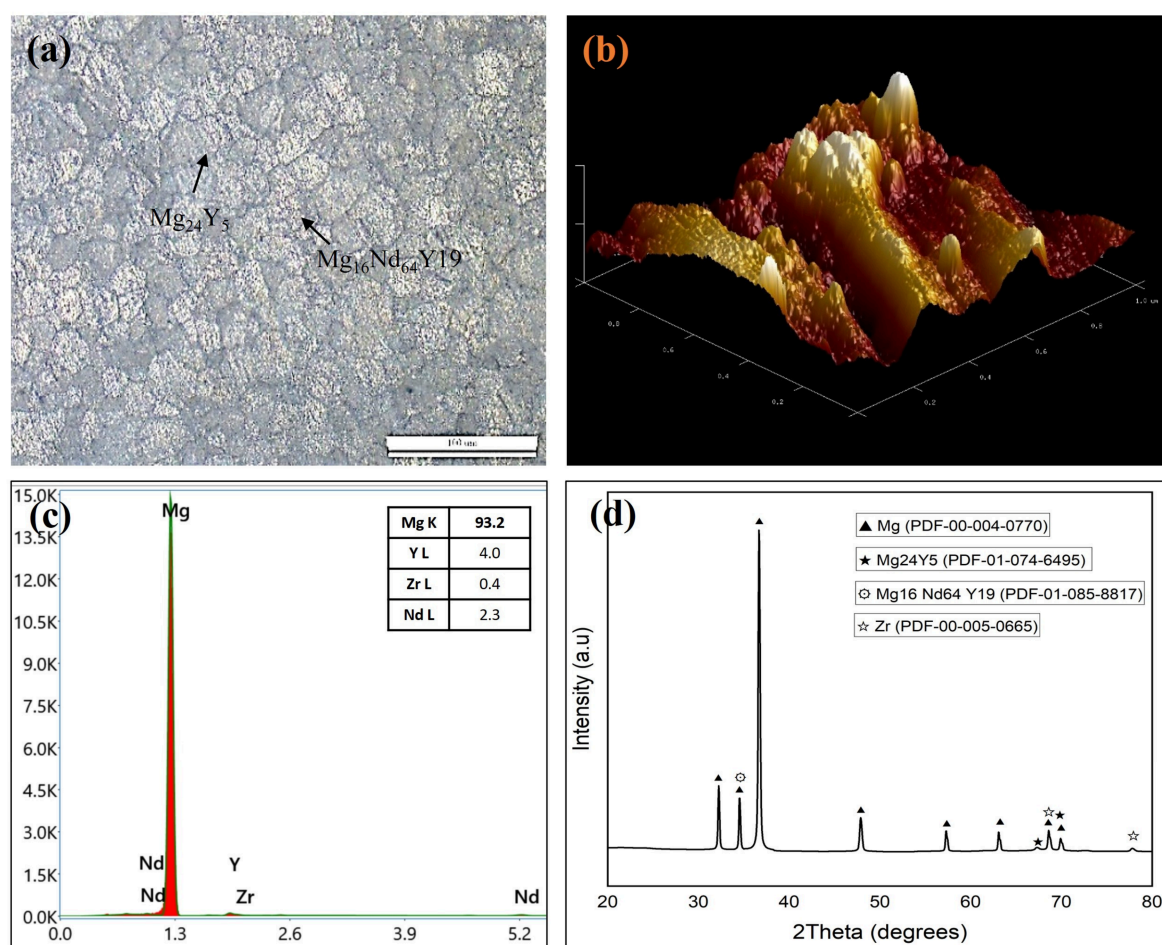


Figure 3. Base substrate characterisation. (a) The WE43 magnesium alloy morphology; (b) AFM image; (c) EDS results of WE43; and (d) XRD spectrum showing characteristic Mg peaks.

3.2. Thickness and Morphology

Titanium was sputtered on WE43 alloy by varying the deposition time, i.e., 1, 1.5, 2 and 3 h, resulting in coating thicknesses of approximately 500, 650, 1000 and 1400 nm, respectively. The surface morphologies of the deposited films were relatively uniform and homogeneous across the investigated conditions, as shown in Figure 4a–h. The coating thickness increased monotonically with deposition duration, although the increase was not strictly linear across the investigated conditions. The corresponding average deposi-

tion rate therefore varied with deposition duration. The subsequent changes in coating morphology, crystallite development and residual stress are consequently discussed in relation to deposition duration and the resulting coating thickness, rather than assuming a constant deposition rate. The observed microstructural evolution indicates progressive grain coarsening with increasing deposition duration, with smaller grains at shorter deposition times developing into larger and more clearly defined structures at longer deposition times. Deposition was performed without active substrate heating. The substrates were initially at room temperature; however, plasma exposure during deposition caused the substrate temperature to increase. The temperatures recorded at the end of the deposition runs were approximately 35 °C, 45 °C, 60 °C and 72 °C for 1, 1.5, 2 and 3 h, respectively. Thus, thermal exposure increased with deposition duration and was considered a coupled process variable in interpreting the observed microstructural evolution. The observed morphological evolution can be discussed qualitatively in relation to the Thornton Structure Zone model [36,37], although the present deposition conditions also involved a progressive increase in substrate temperature with deposition duration. Therefore, the zone description is used as a qualitative framework, rather than as a direct determination of the film-growth zone.

$$T_h = \frac{T_s}{T_m} \quad (2)$$

where T_h is the homologous temperature, T_s is the substrate temperature (in Kelvin), and T_m is the melting point of the coating material (in Kelvin). At lower deposition times, the film begins in Zone T (Transition Zone) with the fibrous grains. As the deposition time increases, the temperature also increases due to prolonged ion bombardment. This shifts the film to Zone II, resulting in a coarse and columnar structure. The observed morphological transition is consistent with competitive grain growth during prolonged sputtering. Initially, numerous nuclei form a fine-grained compact layer. Continued atom arrival promotes preferential growth of energetically favourable grains, producing larger columnar structures. Although grain coarsening reduces grain boundary density, excessive growth may introduce stress concentration sites that subsequently influence corrosion resistance.

Also, microcracks were observed at the initial stages of deposition (1 and 1.5 h), as shown in Figure 4b,d. This indicates that a critical thickness is required to overcome the initial nucleation defects. To protect the WE 43 alloy, the 1 and 1.5 h coatings are mechanically insufficient, whereas the 2 and 3 h coatings achieve a defect-free, continuous barrier to the substrate.

The AFM surface morphology (Figure 5a–h) reveals that the average roughness values of 1, 1.5, 2, and 3 h deposited samples were 3.73 ± 0.38 , 4.74 ± 0.45 , 5.73 ± 0.54 , and 7.17 ± 0.68 nm, respectively. These lower surface roughness values indicate uniform coatings, which help to reduce the surface defect and protect the films against corrosion. Also, the average surface roughness increases from 3.73 ± 0.38 to 7.17 ± 0.68 nm as the film becomes thicker. This suggests coarse grains, which create more peaks and valleys compared to fine, tightly packed grains. Also, it indicated unstable growth, whereby coarse grains prevent the filling of valleys. The average size of the grain was 10–50 nm, which was measured using image processing software (Nanoscope analysis v2.0) with the AFM micrographs. Grains were sampled from different locations to understand the size range of the grains.

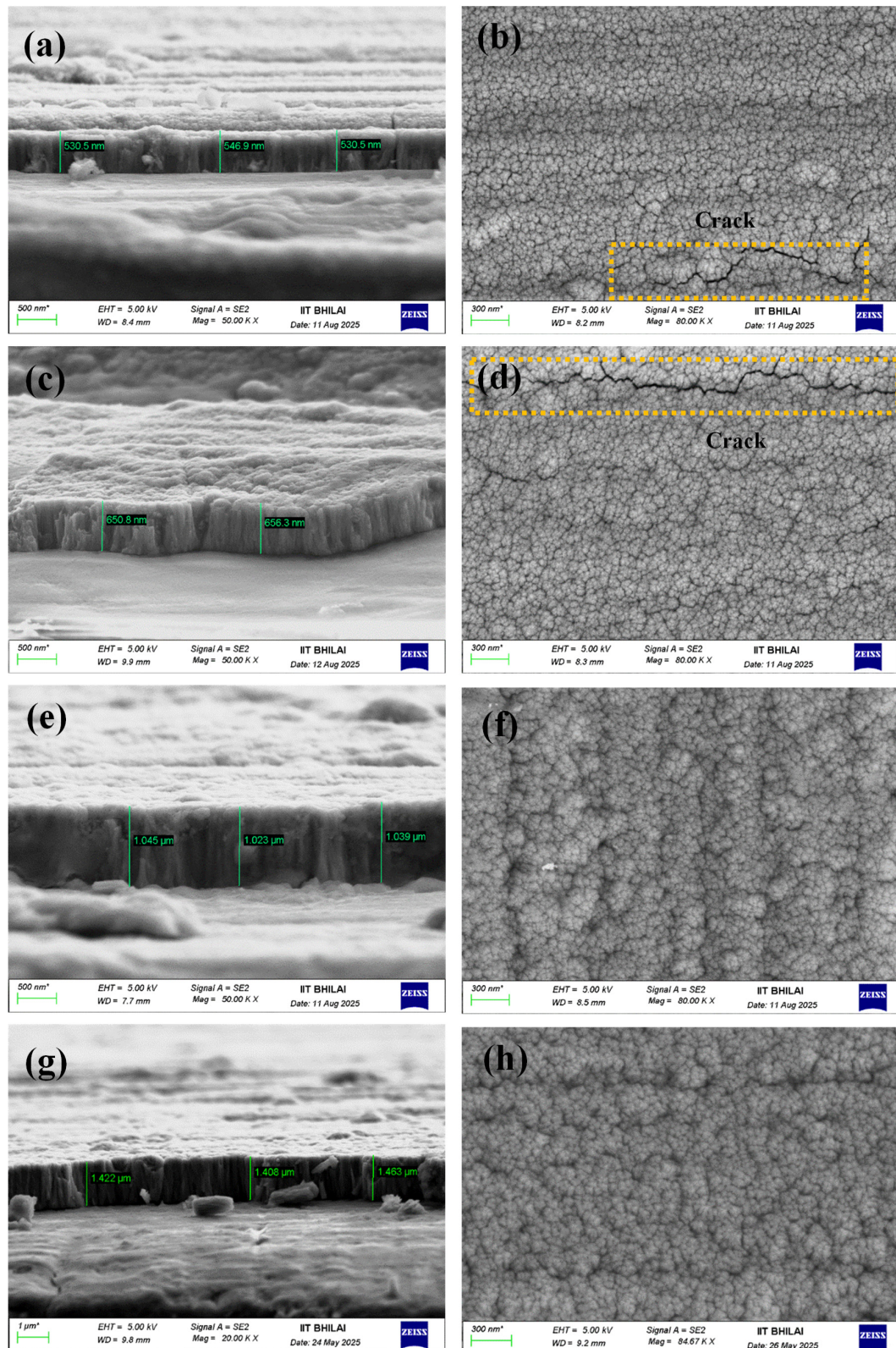


Figure 4. Coatings' cross sectional analyses: (a,b) 1 h deposition showing the coating thickness (~500 nm) and surface morphology; (c,d) 1.5 h deposition showing the coating thickness (~650 nm) and surface morphology; (e,f) 2 h deposition showing the coating thickness (~1 micrometre) and surface morphology; (g,h) 3 h deposition showing the coating thickness (~1.4 micrometres) and surface morphology. The asterisk next to the scale bar indicates that the displayed scale has been calibrated to the default stage position.

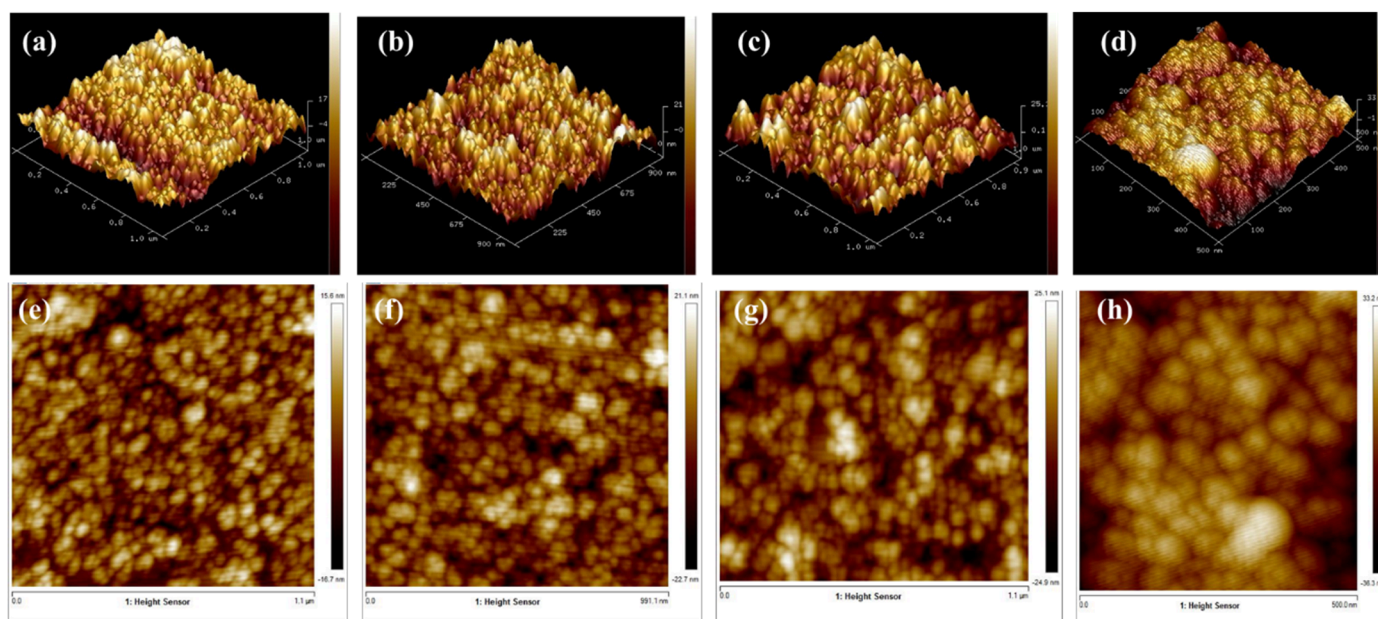


Figure 5. AFM surface morphology: (a,e) 2D and 3D images of 1 h deposition; (b,f) 2D and 3D images of 1.5 h deposition; (c,g) 2D and 3D images of 2 h deposition; and (d,h) 2D and 3D images of 3 h deposition.

3.3. Structural and Residual Stress Analysis

Grazing-incidence X-Ray diffraction (GIXRD) was used to evaluate the structural integrity of Ti-coated samples. It was observed that there was a characteristic rightward shift (Figure 6) in the diffraction peaks of Ti-coated WE 43 compared to the pure Ti target peak. This shift indicated a contraction in the lattice d-spacing, confirming the higher-energy atomic peening effect inherent in the sputtering process. This indicates that the deposited Ti film exists in a highly strained, non-equilibrium state due to the atomic peening effect during sputtering.

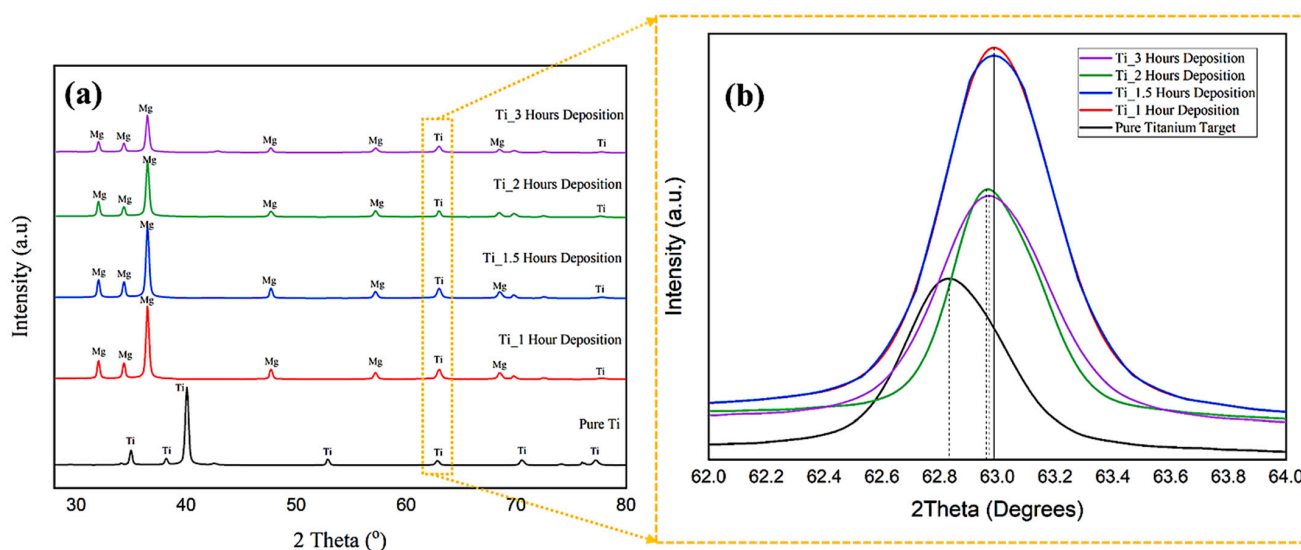


Figure 6. Grazing-incidence X-ray diffraction (GIXRD) patterns of the Ti-coated WE43 specimens and pure Ti target. (a) Diffraction patterns over the measured 2θ range; (b) enlarged view of the Ti peak region showing the peak positions relative to the pure Ti target. The diffraction peaks were indexed to Mg (PDF 00-004-0770) and α -Ti (PDF 00-044-1294). Vertical dashed lines indicate the peak centres.

Scherrer's equation [38] was used to calculate the crystallite size (Table 1). The corundum sample was used as a standard reference material, and the instrument broadening was 0.05 nm. The crystallite size of the 1 to 2 h deposited samples increased from 24.85 ± 3.15 nm to 27.17 ± 3.42 nm [39]. This suggests a systematic improvement in crystalline perfection and grain coarsening as the thickness increases from 500 nm to 1000 nm. However, at 3 h, the deposited sample crystallite size reduced to 25.52 ± 3.18 nm. This correlates with the higher internal stress, suggesting that lattice distortions limit the growth of coherent diffraction domains. Similar correlations between microstructural evolution and the resulting material performance have also been reported for additively manufactured metallic systems, highlighting the critical role of crystallographic development in determining functional properties [40].

Table 1. Microstructural characteristics of Ti coatings deposited at different sputtering durations.

Deposition Time	Thickness (nm)	Average Surface Roughness (nm)	Crystallite Size (nm)	Observed Morphology/Thornton Zone
WE 43	-	8.98 ± 0.92	-	-
1.0 h	~500	3.73 ± 0.38	24.85 ± 3.15	Fine/Cracked/Zone-1
1.5 h	~650	4.74 ± 0.45	25.83 ± 3.28	Medium/Cracked/Zone-1
2.0 h	~1000	5.73 ± 0.54	27.17 ± 3.42	Dense/Zone-T
3.0 h	~1400	7.17 ± 0.68	25.52 ± 3.18	Coarse/Zone-2

The residual stress analysis was performed using the Bragg–Brentano configuration [41]. The sample result exhibited a minor tensile stress of 20.13 MPa. In contrast, all other Ti-deposited samples showed high compressive stress (1, 1.5, 2, and 3 h samples exhibit average compressive stresses of 684.833 ± 17.74 , 691.767 ± 16.65 , 605.3 ± 15.11 , and 992.433 ± 21.7 MPa, respectively). The overall stress state in the deposited titanium coatings comprises both intrinsic growth stress and extrinsic thermal stress. Intrinsic stress originates from the ‘atomic peening’ of high-energy particles during sputtering. Additionally, upon cooling from peak processing temperatures (ranging from 35 °C for 1 h to 72 °C for 3 h) to ambient temperature, the mismatch in thermal expansion coefficients between the titanium film ($\alpha\text{-Ti} \approx 8.6 \times 10^{-6}/\text{K}$) and the WE43 substrate ($\alpha\text{-WE43} \approx 26 \times 10^{-6}/\text{K}$) generates an extrinsic compressive thermal stress. These combined contributions are consistent with the substantially higher compressive residual stress measured for the 3 h coating, 992.4 ± 21.7 MPa, and the localised microcracking observed after corrosion testing. The results indicate that residual stress and coating thickness do not exhibit simple proportional relationships with corrosion resistance. Instead, moderate compressive stress appears beneficial because it suppresses crack opening and enhances coating compactness, whereas excessive compressive stress increases stored strain energy, facilitating microcrack initiation and localised coating failure. This explains why the thickest coating did not provide the best electrochemical performance. Residual stress analysis demonstrates that structural stabilisation was achieved after 2 h of deposition, where the coating exhibited the maximum crystallite size together with the lowest compressive residual stress among the Ti-coated specimens. Beyond 2 h of deposition, the residual stress increased sharply to 992.4 ± 21.7 MPa, leading to the coarse-grained morphology observed by FESEM and AFM as the film accommodated the accumulated strain energy through grain-boundary distortion and cluster formation [39]. The present observations further demonstrate that deposition parameters govern the evolution of microstructure, residual stress and functional performance through a coupled processing–microstructure–property relationship. Similar processing-driven microstructural optimisation has also been reported in advanced surface engineering studies, where controlled thermomechanical processing produced

significant improvements in the functional properties of engineered surfaces [42]. For the present study, post-deposition annealing was intentionally omitted to preserve the as-deposited residual stress state and enable evaluation of its influence on the microstructure and corrosion behaviour. Since thermal treatment can modify residual stress and microstructural evolution, excluding it allowed the effect of coating thickness to be investigated independently. Compressive stress acts as a barrier that inhibits microcrack propagation, preventing SBF from reaching the Mg interface. Preventing a compressive layer is beneficial for resisting crack initiation. However, the effect of extreme stress at 3 h on adhesion durability needs further investigation, as excessive strain can lead to delamination. The angular distribution of residual stress for each deposition condition is presented in Figure 7a–e. The plotted values represent mean residual stress measurements, with error bars indicating the standard deviations of the repeated measurements. The uncoated WE43 substrate (Figure 7a) exhibited a low tensile residual stress with a maximum value of approximately 35 MPa near 60°, whereas all Ti-coated specimens developed compressive residual stresses over the entire angular range. The 1 h coating (Figure 7b) showed a broad compressive stress distribution, while the 1.5 h coating (Figure 7c) exhibited a gradual reduction in compressive stress with increasing rotation angle. The 2 h coating (Figure 7d) maintained the most uniform compressive stress distribution, indicating a comparatively stable stress state. In contrast, the 3 h coating (Figure 7e) displayed the highest compressive stress throughout the measured angles, reflecting substantial stress accumulation within the thickest coating. Figure 7f compares the residual stress profiles of all specimens and clearly demonstrates that the 2 h deposition provides the most balanced residual stress state, whereas prolonged deposition to 3 h results in excessive compressive stress that may compromise coating integrity despite the greater coating thickness.

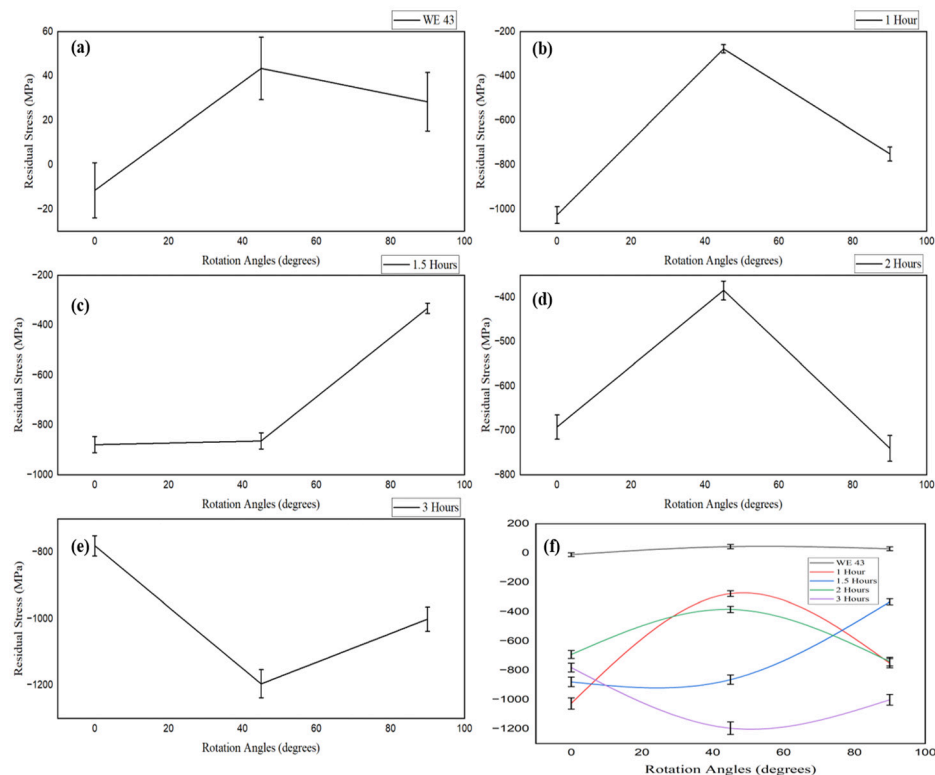


Figure 7. (a–f) Residual stress of Ti-coated samples with respect to rotation angles.

3.4. Wettability and Surface Energy Analysis

The surface wettability of uncoated WE43 and the 2 h Ti-coated specimen was assessed using the sessile drop method with SBF at 37 °C. The 2 h condition was selected as the representative coated condition because it exhibited the most favourable initial electrochemical performance. The contact angle increased from $75.0 \pm 2.1^\circ$ for the uncoated WE43 substrate to $83.0 \pm 1.8^\circ$ after 2 h Ti coating (Figure 8), indicating a moderate decrease in the apparent wettability of the surface. The contact-angle values are reported as mean $\pm$ standard deviation from repeated measurements for each condition. This change may be associated with differences in surface chemistry and nanoscale surface topography between the uncoated WE43 and Ti-coated surfaces. The Wenzel relation provides a theoretical framework for describing the influence of surface roughness on liquid wetting [43,44]:

$$\cos \theta^* = r \cdot \cos \theta \quad (3)$$

where θ is the measured contact angle, θ is the Young's contact angle for an ideal smooth surface, and r is the roughness ratio. In the present study, the Wenzel relation is used only as a theoretical framework for interpreting the possible contribution of surface roughness to the measured wetting behaviour. Because contact-angle measurements were performed only for the uncoated WE43 substrate and the 2 h Ti-coated condition, the present results do not establish a thickness-dependent wettability trend across the deposition series. The measured increase in contact angle therefore indicates a change in surface wetting behaviour following Ti deposition under the present test conditions, rather than establishing a general wettability trend with coating thickness.

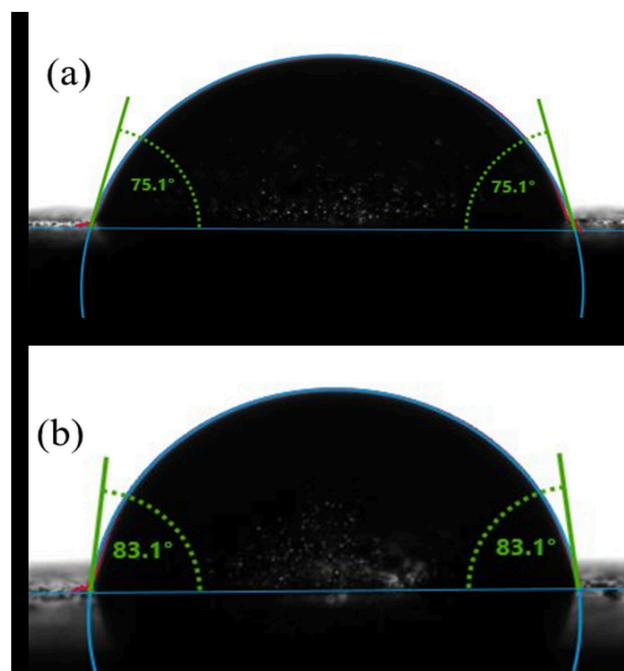


Figure 8. Surface wettability analysis. (a) Contact angle of WE43 alloy; (b) contact angle of Titanium-deposited (2 h) WE43 alloy.

3.5. In Vitro Corrosion Study

Potentiodynamic polarisation tests were conducted to evaluate the electrochemical behaviours of uncoated WE43 and Ti-coated WE43 specimens (Figure 9). A potential scan rate of 1 mV/s was used instead of the commonly reported 0.16 mV/s. The selected potential scan rate can influence the determination of Tafel slopes and corrosion current

density (i_{corr}) [45–47]. Because a relatively higher scan rate may limit the attainment of a quasi-steady electrochemical response and can influence the apparent Tafel slopes and corrosion current density, the resulting parameters should be interpreted as comparative measurements under identical test conditions, rather than as equilibrium corrosion characteristics. In the present study, the same scan rate of 1 mV/s was applied to all specimens to ensure consistent comparison of their electrochemical responses.

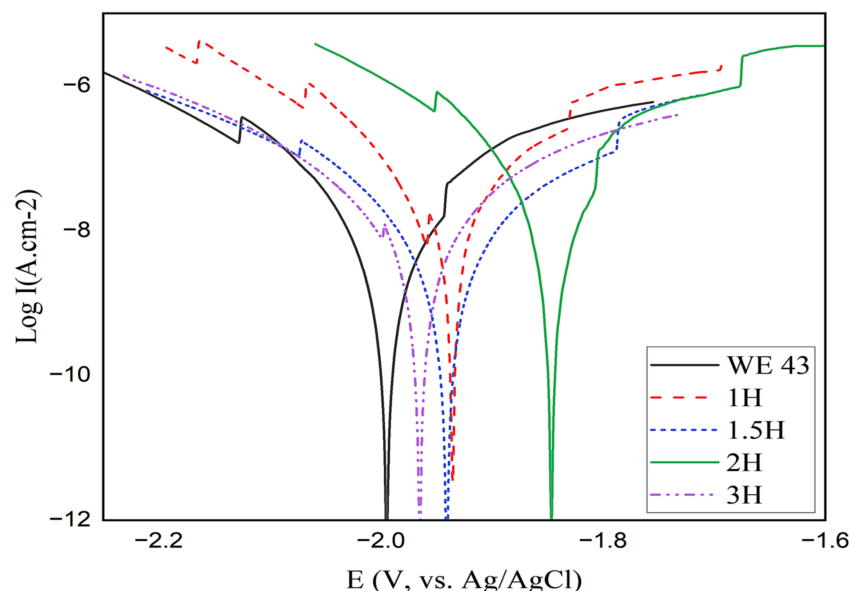


Figure 9. PDP (potentiodynamic polarisation) curves of WE43 and Ti-deposited WE43 samples.

The PDP Curves of WE43 and Ti-coated WE43 samples (Figure 9) and the consolidated data (Table 2) clearly indicate a significant improvement in corrosion resistance after the titanium coating on WE43. The uncoated sample shows the highest potential at -1.99 V (vs. Ag/AgCl). All remaining titanium-deposited samples show a noble shift towards more positive potentials, with the 2 h deposited sample reaching -1.84 V (vs. Ag/AgCl). This shift indicates that coating effectively acts as a cathodic inhibitor, reducing the thermodynamic tendency of WE 43 to oxidise. Corrosion current density (i_{corr}) directly represents the corrosion rate, decreasing from 0.402 mA/cm² for WE43 to 0.133 mA/cm² for the 2 h deposition. Consequently, the corrosion rate reduced from 8.87 mm/yr (WE43) to a highly optimised 2.93 mm/yr (2 h deposition). The electrochemical parameters derived from potentiodynamic polarisation curves (Table 2) show that the 2 h coated condition yields the lowest corrosion current density (i_{corr}) and calculated corrosion rate. The 1.5 h sample exhibits an (i_{corr}) of 0.186 ± 0.031 mA/cm², corresponding to a corrosion rate of 4.10 ± 0.68 mm/yr. The improvement in corrosion resistance cannot be attributed solely to increased coating thickness. The present results further demonstrate that optimising processing parameters is essential for achieving the desired balance between microstructural stability and functional performance. Similar processing-dependent variations in corrosion behaviour and microstructure have been reported for engineering alloys under different processing conditions [42,48]. Instead, it results from the combined effects of reduced coating defects, improved crystallinity, optimum residual compressive stress and enhanced barrier continuity. These factors collectively delay electrolyte penetration towards the magnesium substrate and suppress localised galvanic dissolution. Such a reduction in corrosion rate is desirable for temporary orthopaedic fixation because it enables controlled degradation while maintaining adequate mechanical integrity during the early stages of bone healing. In addition, the slower degradation rate helps suppress excessive hydrogen

evolution, thereby reducing the risks of gas accumulation and premature mechanical failure during the critical healing period.

Table 2. Electrochemical parameters obtained from potentiodynamic polarisation measurements in simulated body fluid.

Material	E_{corr}	i_{corr}	β_a	β_c	Corrosion Rate
	(V (vs. Ag/AgCl))	(mA/cm ²)	(mV/dec)	(mV/dec)	(mm/year)
Uncoated WE 43	-1.99 ± 0.05	0.402 ± 0.103	238 ± 110	-231 ± 92	8.87 ± 1.11
1 h deposition	-1.94 ± 0.04	0.253 ± 0.065	108 ± 32	-107 ± 28	5.59 ± 0.85
1.5 h deposition	-1.94 ± 0.05	0.186 ± 0.031	165 ± 48	-154 ± 42	4.10 ± 0.68
2 h deposition	-1.84 ± 0.10	0.133 ± 0.026	54.4 ± 16	-53.4 ± 14	2.93 ± 0.57
3 h deposition	-1.96 ± 0.03	0.218 ± 0.52	162 ± 45	-172 ± 49	4.81 ± 0.74

It is important to note that, while the 3 h deposition has a thicker film (1400 nm) than all other depositions, the 2 h deposition exhibited the most favourable electrochemical response. This behaviour is attributed to the dense morphology and the larger crystallite size observed in the 2 h deposition (explained in Section 3.2). Moreover, the 2 h deposited sample exhibited a compressive residual stress of 605.3 ± 15.11 MPa, which was the lowest among the other samples. In contrast, the 3 h deposited sample exhibited a compressive residual stress of 992.4 ± 21.7 MPa. The substantially higher residual stress in the 3 h coating coincided with the observed localised microcracking after corrosion testing and the increase in corrosion rate from 2.93 to 4.81 mm/year. These findings indicate that corrosion resistance exhibits an optimum, rather than a monotonic dependence on coating thickness. Increasing the coating thickness initially improves barrier continuity by reducing coating defects and enhancing film compactness. However, beyond the optimum thickness, the accumulation of excessive compressive residual stress offsets these benefits by promoting localised microcrack initiation and facilitating electrolyte ingress. Consequently, the electrochemical performance is governed by the balance between coating integrity and stress evolution, rather than by thickness alone. This processing–microstructure–property relationship explains why the 2 h deposited coating outperformed the thicker 3 h coating despite having a lower film thickness.

After the electrochemical test, elemental distribution was evaluated for uncoated WE43 and Ti-coated samples using field-emission scanning electron microscopy (FESEM) and energy-dispersive X-ray spectroscopy (EDX) mapping to examine the post-corrosion surface condition. The uncoated sample exhibited localised and pitting corrosion. Elemental mapping showed Mg, O, Ca and P on the corroded surface (Figure 10a). The Mg and O distributions are consistent with the formation of magnesium-containing corrosion products, while the presence of Ca and P indicates deposition of calcium- and phosphorus-containing corrosion products from the SBF environment. The 1 and 1.5 h coatings showed fragmented Ti distributions (Figure 10b,c), indicating greater local exposure of the underlying WE43 substrate after corrosion testing.

The 2 h sample, with a coating thickness of approximately 1000 nm, showed comparatively better surface retention after the corrosion test. Elemental mapping (Figure 10d) showed a relatively continuous Ti distribution with limited exposure of the underlying WE43 substrate. This observation is consistent with the electrochemical results, where the 2 h coating exhibited the lowest measured corrosion current density and calculated corrosion rate. The 2 h coating also exhibited a compressive residual stress of 605.3 ± 15.11 MPa. The corrosion rate decreased from 8.87 mm/year for uncoated WE43 to 2.93 mm/year for the 2 h coating, corresponding to an approximately 67% reduction under the present test conditions. The step-like fluctuations observed in the potentiodynamic polarisation

curves, particularly for the 1 h coating (Figure 9), may be associated with transient localised corrosion and repassivation events [49,50]. The more pronounced fluctuations for the 1 h coating are consistent with the less continuous Ti distribution observed after corrosion testing (Figure 10b).

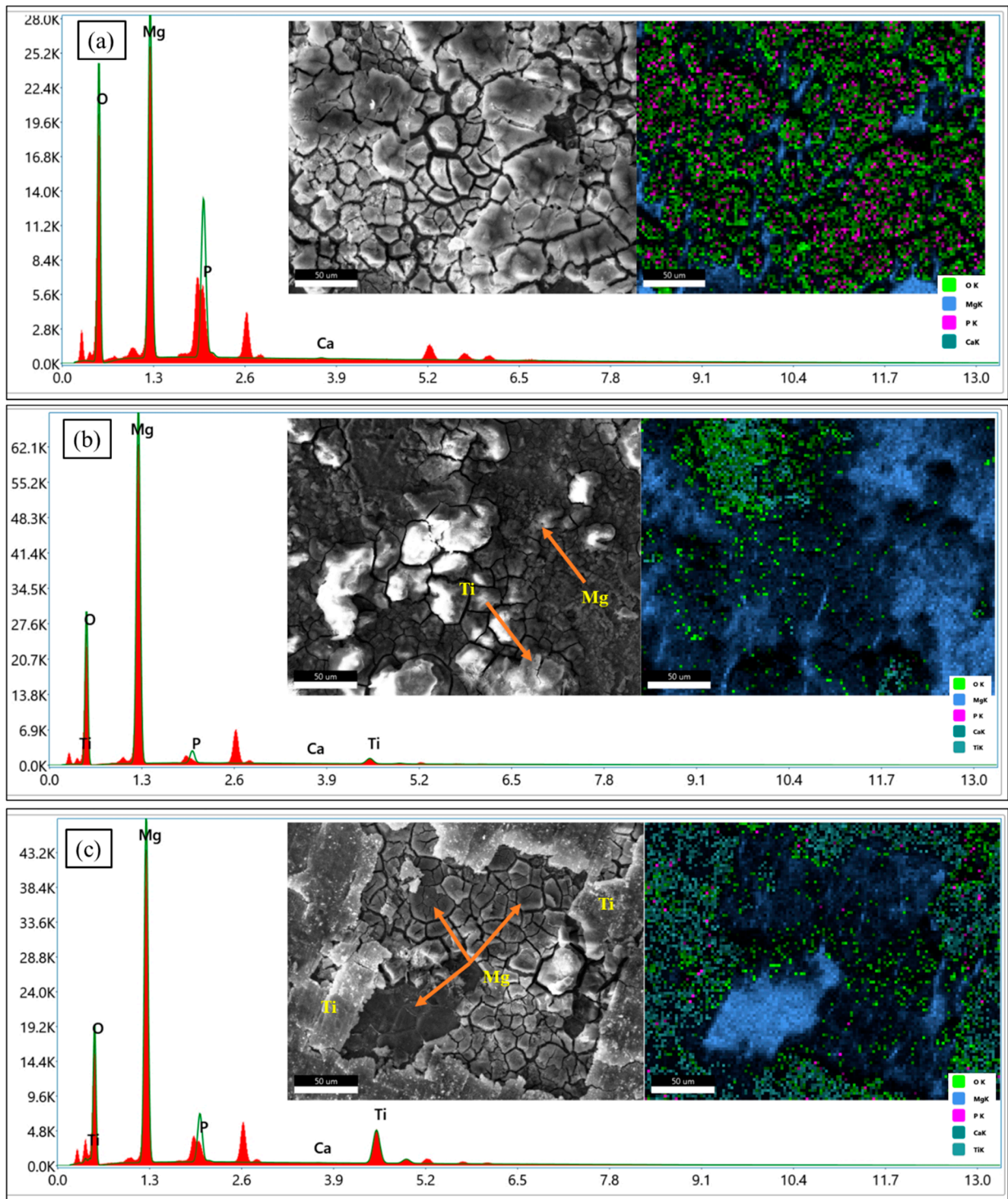


Figure 10. *Cont.*

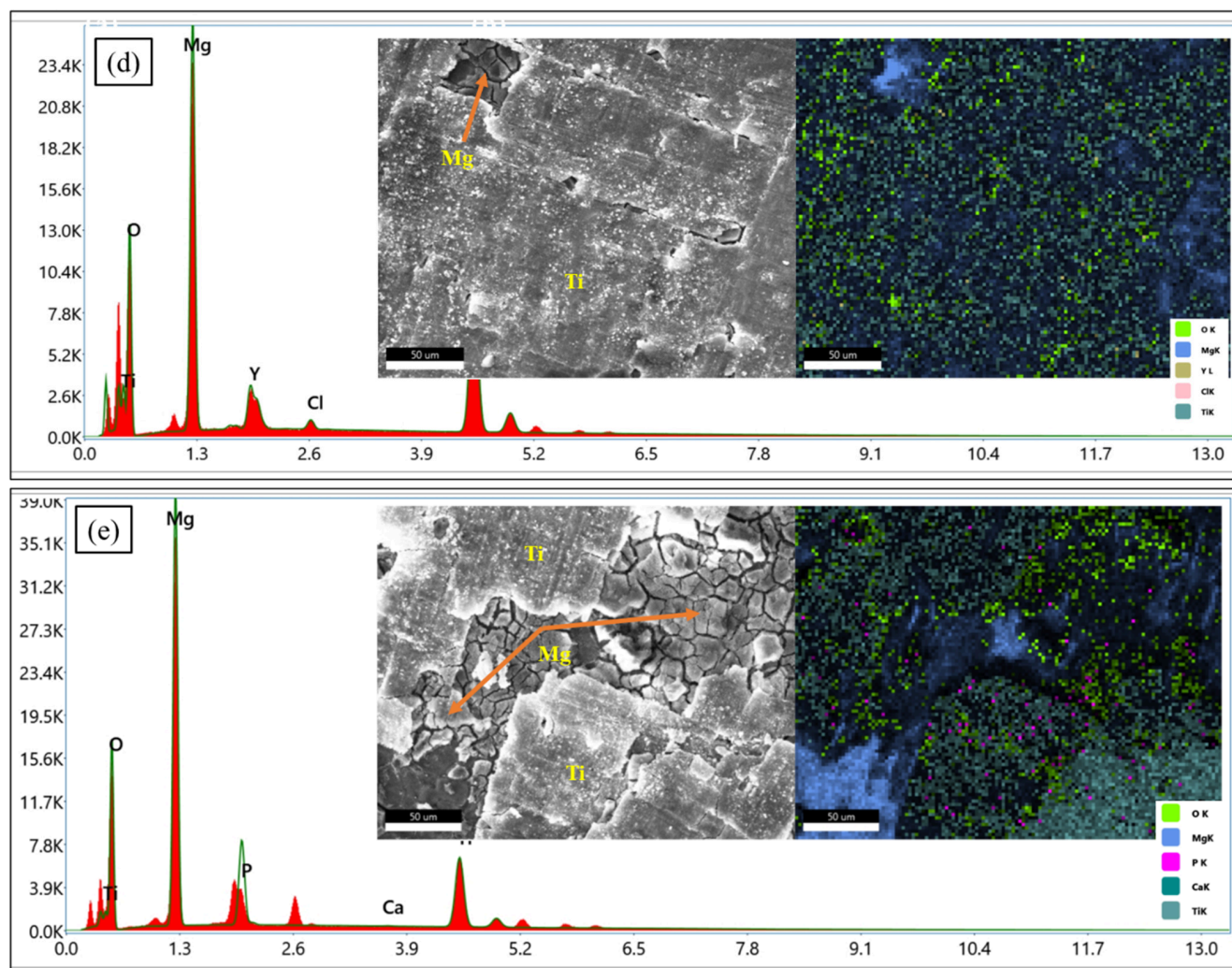


Figure 10. Elemental mapping of the corroded surface. (a) WE 43; (b) 1 h deposition surface; (c) 1.5 h deposition surface; (d) 2 h deposition surface; and (e) 3 h deposition surface.

The 3 h coated sample showed localised microcracks in the post-corrosion FESEM images. Mapping (Figure 10e) indicated Mg-rich regions at the base of the cracks. This is because of the high compressive stress of 992.433 ± 21.7 MPa, which promotes crack propagation when exposed to the SBF environment. This resulted in a localised loss of barrier integrity, increasing the corrosion rate to 4.81 mm/yr. Overall, the proposed corrosion mechanism is consistently supported by FESEM, AFM, GIXRD, residual stress analysis, wettability measurements and electrochemical characterisation. The agreement among these independent characterisation techniques demonstrates that the superior corrosion resistance of the 2 h deposited coating originates from the combined effects of a dense microstructure, optimum crystallinity and moderate compressive residual stress, which together suppress electrolyte penetration and maintain coating integrity. This multi-technique validation strengthens the reliability of the proposed processing–microstructure–corrosion relationship and confirms that corrosion performance is governed by the combined evolution of microstructure and residual stress, rather than coating thickness alone. While potentiodynamic polarisation and post-corrosion microstructural characterisation support the improved corrosion protection provided by the optimised 2 h coating, future investigations utilising long-term Electrochemical Impedance Spectroscopy (EIS) will be essential to dynamically monitor charge transfer kinetics and interface degradation over extended immersion durations.

4. Conclusions

This study investigated the influence of magnetron sputtering deposition time on structural, mechanical, and electrochemical properties of Ti-coated WE 43 magnesium alloy. The following key observations are drawn.

- i. FESEM analysis confirmed a monotonic increase in coating thickness with varying Titanium deposition time, ranging from 500 nm at 1 h to 1400 nm at 3 h. Also, it confirms that the morphological transition from fine to dense, and eventually, coarse structures occurred as grains coalesced with increasing deposition time.
- ii. GIXRD analysis revealed that the 2 h deposition yielded the optimal structural condition, with a crystallite size of 27.17 ± 3.42 nm. This indicates a highly ordered crystalline state before the onset of extreme strain-induced refinement observed at 3 h.
- iii. Residual stress analysis revealed a minor tensile stress in the uncoated WE43 substrate and significant compressive residual stresses in all Ti-coated samples. The 2 h coating exhibited the most stable residual stress state, with a compressive residual stress of 605.3 ± 15.11 MPa, whereas the 3 h coating reached a compressive residual stress of 992.4 ± 21.7 MPa, which is likely to promote microstructural defects and compromise coating integrity.
- iv. The wettability study clearly indicated that Ti deposition modified the surface energy, increasing the contact angle from $75^\circ \pm 2.1^\circ$ to $83^\circ \pm 1.8^\circ$. It shifted towards a hydrophobic behaviour, providing an initial physical barrier against the SBF electrolyte in physiological environments.
- v. Electrochemical testing revealed that 2 h deposition provides the most effective corrosion protection, achieving the lowest corrosion rate of 2.93 mm/yr and noble shift in corrosion potential of -1.84 V, while 3 h deposition showed a thicker film and higher corrosion rate of 4.81 mm/yr, confirming that film density and residual stress are more critical than the thickness alone for the barrier integrity.
- vi. Post-corrosion EDX mapping confirms that thickness alone is not a primary factor for corrosion resistance, but also that a combination of film density and residual stress is critical to maintaining a good electrochemical barrier on reactive WE 43.

The present study demonstrates that corrosion protection of magnetron-sputtered Ti-coated WE43 alloy is governed by an optimum balance between coating thickness, microstructural evolution and residual compressive stress, rather than by coating thickness alone. Under the present deposition conditions, the 2 h sputtered Ti coating (~1000 nm) exhibited the most favourable combination of dense microstructure, maximum crystallite size and moderate compressive residual stress, resulting in a 67% reduction in corrosion rate compared with the uncoated WE43 alloy. These findings establish a clear processing–microstructure–corrosion relationship and provide practical guidance for optimising titanium coating thickness to improve the durability and controlled degradation of biodegradable magnesium implants intended for temporary orthopaedic applications. The findings also provide a transferable methodology for optimising sputtered metallic coatings in which coating thickness, residual stress and microstructural evolution are simultaneously controlled to maximise corrosion protection.

While the present study demonstrates that optimising Ti coating thickness significantly improves the initial corrosion resistance of WE43 magnesium alloy, the findings should be interpreted within the scope of the experimental investigation. The electrochemical evaluation was limited to potentiodynamic polarisation measurements in simulated body fluid, which represent the initial corrosion response and do not fully reproduce the complex physiological conditions encountered *in vivo*. Accordingly, long-term immersion behaviour, hydrogen evolution, electrochemical impedance spectroscopy (EIS), coating adhesion after prolonged degradation, and comprehensive biological evaluations were

beyond the defined scope of this investigation. These aspects may be considered in future studies to further extend the present findings and support the comprehensive assessment of Ti-coated WE43 magnesium alloys for biomedical applications.

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Abbreviations

AFM	Atomic Force Microscopy
ASTM	American Society for Testing and Materials
DC	Direct Current
EDX	Energy-Dispersive X-ray Spectroscopy
E_{corr}	Corrosion Potential
FESEM	Field-Emission Scanning Electron Microscopy
GIXRD	Grazing-Incidence X-ray Diffraction
i_{corr}	Corrosion Current Density
PDP	Potentiodynamic Polarisation
PECVD	Plasma-Enhanced Chemical Vapour Deposition
PVD	Physical Vapour Deposition
Ra	Average Surface Roughness
SBF	Simulated Body Fluid
Ti	Titanium
WE43	WE43 Magnesium Alloy
XRD	X-ray Diffraction

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