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Microstructural Evolution and Electrochemical Behavior of Solution Treated, Hot Rolled and Aged MgDyZnZr Alloy

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Abstract: In order to develop a potential route to fabricate plates and clips for orthopedic applications, a Mg–3.4Dy–0.2Zn–0.4Zr (wt.%) alloy was produced and analyzed in different conditions: solution treated at 525 °C for 3 h, hot rolled and hot rolled and aged at 250 °C. The aging behavior of the rolled alloy was investigated during isothermal aging at 250 °C, and a significant peak was observed at 10 h. The electrochemical behavior was evaluated in 0.9 wt.% NaCl solution at 37 ± 0.5 °C by potentiodynamic polarization and electrochemical impedance spectroscopy. The 525 °C–3 h and hot rolled specimens exhibited corrosion rates of 2.0 and 1.7 mm/year, respectively. The hot rolled and aged at 250 °C for 10 h specimen presented a grain size of 11.8 ± 1.7 µm with an intense microtexture of the basal {0002} plane, hardness of 73 ± 3 HV and higher impedance modulus and obtained the highest corrosion resistance with a corrosion rate of 0.9 mm/year.

Keywords: Mg alloys; MgDy alloy; MgDyZnZr hot rolled; aging hardening; electrochemical

1. Introduction

Studies on magnesium (Mg) alloys containing zinc (Zn) and rare earth elements (REEs) are important for several applications, including bioabsorbable implants, due to their biocorrosion and biocompatibility properties [1,2]. It is well described in the literature that the presence of Mg²⁺ and Zn²⁺ cations are essential to the vital functions of the human body [3], and the normal Mg content in the blood is in the range of 1.7–2.22 mg/dL [4]. When these elements are in excess, they can be excreted out through the urinal route [3,5].

It has been reported that REEs should be used with caution in the human body [6]. However, investigations have shown that the dysprosium cation (Dy³⁺) exhibits good cytocompatibility according to an in vitro study of cytotoxicity as well as an acceptable inflammatory response [7] with a half-lethal dose (LD₅₀) value of dysprosium chloride at 585 mg/kg [6,8]. Moreover, Dy³⁺ may replace calcium (Ca) in hydroxyapatite, and Dy phosphate can be formed as a degradation product [9–11]. Another aspect is that Dy compounds might be used as negative contrast agents for high field magnetic resonance imaging [12], which may help to monitor the implant degradation in the human body.

According to the binary Mg–Dy phase diagram, Dy has an important solubility in α-Mg, with a maximum of 6.16 at.% reached at 560 °C, the highest of all REEs. How-

ever, it presents low solubility at room temperature [13]. Thus, Mg–Dy alloys are susceptible to microstructural modifications by thermomechanical processes. It has been reported that microalloying with Dy has positive effects in both mechanical and corrosion properties [6,14,15], and the control of second phases precipitation is one of the critical steps during thermomechanical processing and heat treatments of these alloys.

Generally, for Mg–REEs alloys, the solid solution decomposition during aging heat treatments at low temperatures proceeds in the following sequence: Mg supersaturated solution $\rightarrow \beta'' \rightarrow \beta' \rightarrow \beta_1 \rightarrow \beta$, where β'' , β' and β_1 are metastable phases and β is only the stable one, with stoichiometry of $\text{Mg}_{24}\text{Dy}_5$ [16,17]. In addition, during the early stages of aging heat treatments, the β'' -phase precipitation is common and is characterized by a D0_{19} -type structure [18]. The β' precipitation in the α -Mg matrix is responsible for the peak of hardness of aged alloys [19] and presents an orthorhombic unit cell [20] with Mg_7Dy stoichiometry.

Regarding the corrosion process of Mg–Dy alloys, a pitting corrosion mode takes place when the Dy content is increased [6], which is generally caused by galvanic corrosion between α -Mg matrix and intermetallic phases [21]. Previous studies involving a Mg–5Dy (wt.%) alloy demonstrated a filiform corrosion mode with a corrosion rate of 0.5 mm/year in 0.9 wt.% NaCl aqueous solution [6]. During the corrosion process, Dy is oxidized and incorporated into corrosion films [6,11]. Dy_2O_3 and $\text{Dy}(\text{OH})_3$ have been observed as corrosion products in a Mg–10Dy (wt.%) alloy [11], where $\text{Dy}(\text{OH})_3$ is mainly distributed at the outer and Dy_2O_3 at the inner corrosion layer.

In this work, a MgDyZnZr alloy was produced from pure elements by a melting/casting process and a plate was manufactured by hot rolling. Possible precipitation strengthening and the electrochemical behavior were investigated in order to evaluate the potential of the material as plates and clips for orthopedic applications or as a substrate for drug delivery devices.

2. Materials and Methods

2.1. Alloy Fabrication and Heat Treatments

A Mg–3.4Dy–0.2Zn–0.4Zr (wt.%) (named MgDyZnZr) alloy was produced using high-purity Mg (min. 99.97%), Dy (min. 99.9%), Zn (min. 99.9%) and Zr (min. 99.8%). The raw materials were induction melted in a water-cooled copper crucible under argon atmosphere, producing an ingot of dimensions near 30 mm diameter by 30 mm length. The as-cast ingot was solution treated at 525 °C for 3 h (named as “H525 °C-3 h”) under argon flux and then slowly furnace cooled to room temperature.

A small plate of 14 mm thickness was removed longitudinally from the ingot, pre-heated at 450 °C for 0.5 h and then hot rolled to a thickness of 2.2 mm (84.2% of reduction) after multiple passes. Between rolling passes, the material was heated at 450 °C for 5 min. The hot rolled specimens are named “rolled” hereafter. Specimens cut from the final hot rolled plate were aged at 250 °C under an argon atmosphere for different times and named, e.g., “aged-10 h”, for a specimen aged for 10 h.

2.2. Vickers Hardness

The hardness values of the different specimens were determined using a Vickers hardness tester (Buehler MicroMet 6020) with load of 100 gf and holding time of 15 s. For each specimen 20 points were randomly measured.

2.3. Microstructural Characterizations

The different MgDyZnZr alloy specimens were characterized in terms of chemical composition, microstructure and macro-texture. For chemical composition analysis, scanning electron microscopy (SEM) (Hitachi TM3000) in the backscattered electron mode (BSE) equipped with X-ray energy-dispersive spectrometer (EDS) was used. X-ray diffraction (XRD) (PANalytical, Empyrean) was performed with Cu-K_α radiation, at 40 kV and 30 mA; 2θ ranging from 20° to 60°, step size of 0.02° and counting time of 150 s per step. The

macro-texture of aged-10 h specimens was determined by measuring pole figures of $\{10\bar{1}0\}$, $\{0002\}$ and $\{10\bar{1}1\}$ using XRD (PANalytical, Empyrean) with Mo- K_α radiation at 40 kV and 30 mA, and the data was compiled with ATEX software [22].

The grain sizes of the different specimens were determined using light optical microscopy (LOM) (Leica DM 4000 M) applying the line intercept method [23]. The specimens for LOM observations were ground, polished and etched in a 6 g picric acid, 100 mL ethanol and 10 mL H₂O solution.

2.4. Electrochemical Tests

Electrochemical measurements of the H525 °C 3 h, rolled and aged-10 h specimens were carried out in an aqueous solution of 0.9 wt.% NaCl at 37.0 ± 0.5 °C in order to simulate human body conditions. The tests were performed using a SOLARTRON 1287A potentiostat/galvanostat equipment model 1287A equipped with a SOLARTRON 12587S impedance analyzer module and a three-electrode cell formed by a saturated silver chloride Ag|AgCl (KCl sat.) reference electrode, platinum counter electrode and a working electrode with 1.5 cm² of exposed area. Prior to the tests, the specimens were ground through successive grades of silicon carbide (SiC) abrasive papers from 320 grit to 2400 grit using isopropyl alcohol as lubricating liquid.

The Open Circuit Potential (OCP) value was measured prior to each Electrochemical Impedance Spectroscopy (EIS) test. The EIS tests were performed at OCP, and they were carried out at specific moments (30 min, 50 min and 70 min) after electrolyte immersion. An AC amplitude of 10 mV over the frequency range of 100 kHz to 0.02 Hz was used. The ZView and ZPlot softwares were used for obtaining and fitting the impedance data, respectively.

Potentiodynamic polarization curves were obtained using a scan rate of 1 mV/s. The polarization started from cathodic potential of -0.5 V relative to the last OCP value and interrupted when the current reached 0.1 A. For every condition, a minimum of two trials were conducted.

3. Results and Discussion

3.1. Microstructural Characterization of the H525 °C-3 h Specimen

Figure 1a shows a SEM/BSE micrograph from the H525 °C-3 h specimen. Thin Mg_xDy precipitates were observed homogeneously distributed in the α -Mg matrix. According to the XRD pattern exhibited in Figure 1b, these precipitates were composed mostly of Mg₂Dy. The Mg₂Dy precipitate is thermodynamically stable as reported in the Mg–Dy phase diagram [13] and was found in a similar Mg–2.08Dy–2Sr–1Zr (wt.%) alloy [24]. LOM analyses indicated coarse equiaxed grains with an average grain size of 326 ± 91 μ m as shown in the micrograph of Figure 1c. The hardness value was 53 ± 3 HV.

3.2. Microstructural Characterization of Rolled Specimen

Figure 2 shows a schematic plate with indication of the rolling direction (denoted as RD), transversal rolling direction (denoted as TD) and the normal rolling direction (denoted as ND), which were used to refer to the different regions of the plate. The hot rolled process promoted thin precipitates randomly distributed along the microstructure (Figure 2a,c,e) and recrystallized grains, with an average grain size of 8.7 ± 1.5 μ m, approximately 40 times smaller than that of the H525 °C-3 h specimen. Furthermore, we observed similar grain shapes in different directions, indicating a homogeneous microstructure, as shown by the LOM micrographs in Figure 2b,d,f.

The XRD pattern (Figure 3) of the rolled specimen showed small peaks of β'' in addition to those of the Mg₂Dy and α -Mg matrix phase. This metastable β'' precipitation with a D0₁₉-type structure likely occurred during the heating period between the rolling passes. Additionally, an intensification of peaks associated with the (0002) and (10 $\bar{1}1$) planes has been observed, and this would have been caused by slip plane activation during

the hot rolling process. The plate in the as-rolled condition exhibited a hardness value of 60 ± 6 HV.

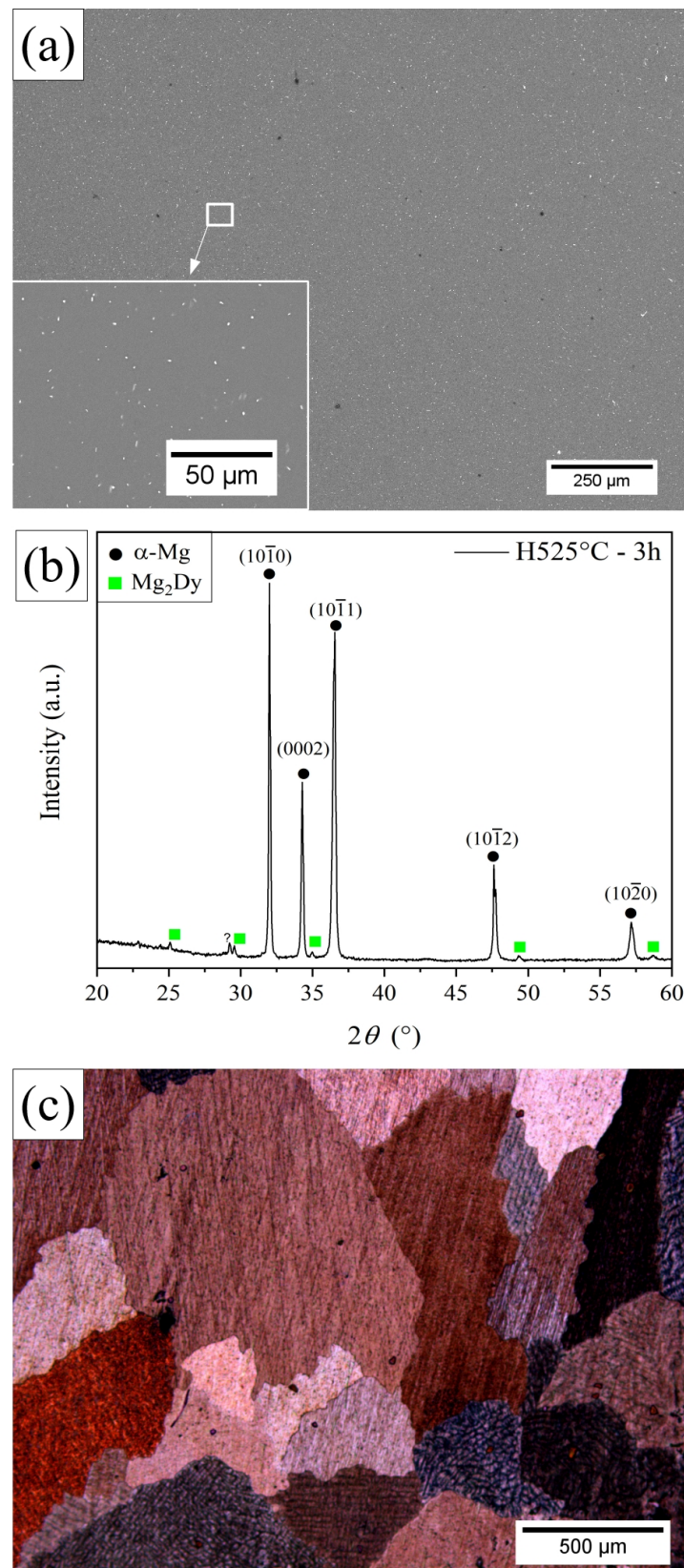


Figure 1. (a) SEM/BSE micrograph, (b) XRD pattern and (c) LOM micrograph of the H525 $^\circ\text{C}$ -3 h specimen.

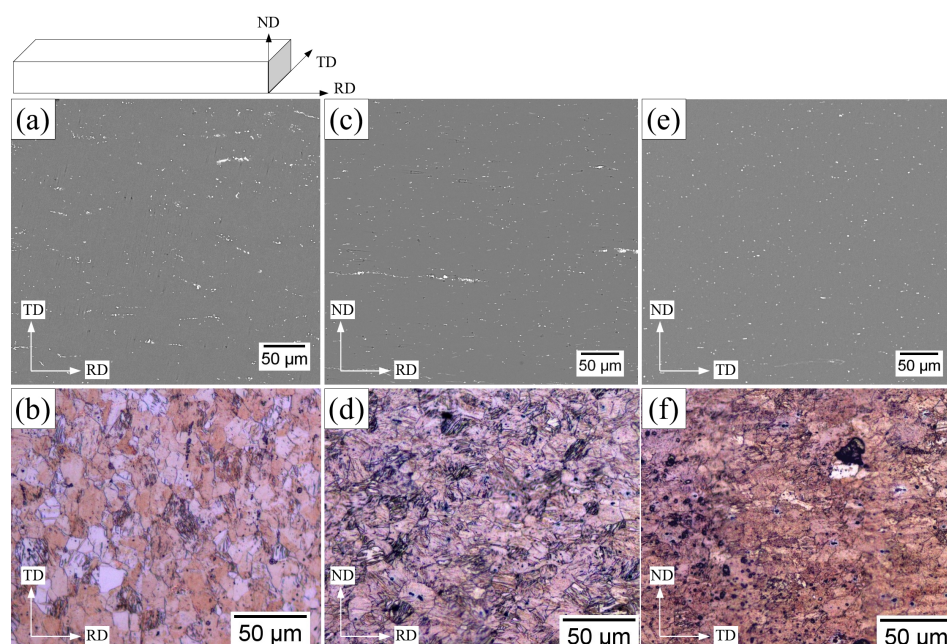


Figure 2. Schematic directions in the rolled plate and SEM/BSE micrographs (a,c,e) and LOM micrographs (b,d,f) of the rolled specimen.

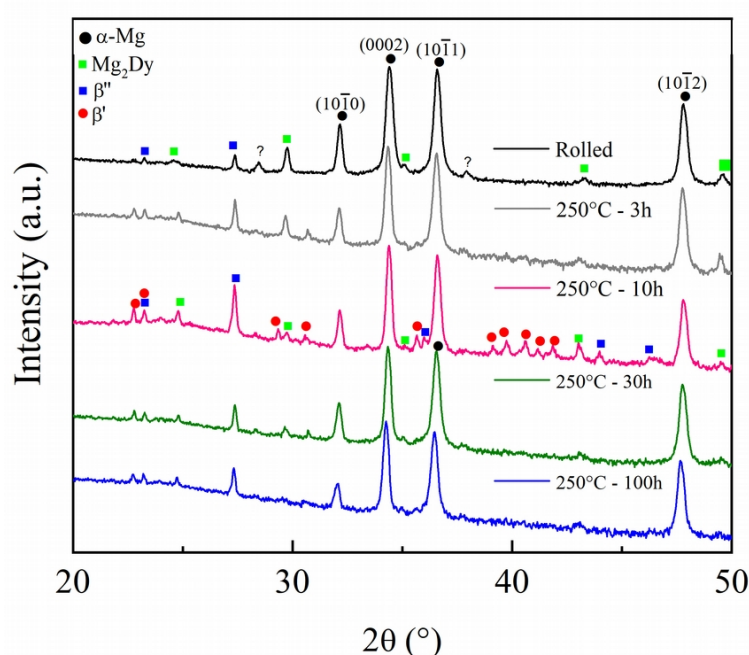


Figure 3. XRD diffractograms of rolled and aged specimens.

3.3. Aging Hardening Behavior of the Alloy

Figure 4 shows the age-hardening response of the alloy after hot rolling at 250 °C. The specimen aged for 10 h (named aged-10 h) had the maximum hardness recorded of 73 ± 3 HV, a 22% increase compared to that of the rolled specimen. Subsequently, the hardness values decreased with prolonged aged.

The XRD patterns of the aged specimens (Figure 3) clearly exhibited an intensification of the peaks related to β'' and the β' phase peaks increase in intensity up to 10 h of aging, then decreased with prolonged aged. These metastables β'' and β' precipitates are expected for Mg–REEs alloys when aged in range of 190–300 °C [17,25,26]. Generally, β'' phases may

be observed at the beginning of aging and further aging will result in the transformation of these β'' precipitates to β' precipitates [27].

Gao et al. [16] informed that after some aging hours the β' phase tends to transform to β_1 phase and takes more than 2000 h to transform β_1 to β when aged at 250 °C for Mg–15Gd–0.5Zr (wt.%) alloy. Although the peaks of β'' and β' phases became less intense for longer aging times, β_1 and β could not be identified in the XRD patterns (Figure 3). Likely, the small amount of Dy in the MgDyZnZr alloy contributed to a low volume fraction of β_1 phase. In addition, the aging times at 250 °C used in this work would likely have been very short to achieve precipitation of the β stable phase.

Apps et al. [17], who investigated the aging–hardening behavior of Mg–7Dy–2.25Nd–0.6Zr (wt.%) and Mg–7Gd–2.25Nd–0.6Zr (wt.%) alloys, found similar results to those obtained in this work. A significant aging response at 300 °C could be attributed to the rapid formation of β' and possibly β'' , precipitates during the early stages of aging for the alloy with Gd. Saito et al. [18] showed the aging–hardening response for Mg–21.79Dy (wt.%) alloy, and, as in cases of Mg–Gd and Mg–Y alloys, β' precipitation occurred and increased the age-hardening effects. In addition, for longer aging times, the β' precipitates became larger.

Therefore, it is suggested that a combination of β'' precipitation and the transformation of $\beta'' \rightarrow \beta'$ during the 10 h of aging promoted the age-hardening response for the alloy investigated in this work.

Figure 5a,c,e shows micrographs from different regions of the aged-10 h specimen, with a similar microstructure observed for all regions with an average grain size of $11.8 \pm 1.7 \mu\text{m}$ (Figure 5b,d,f) that are not much higher than that of the rolled specimen condition. Possibly, β''/β' precipitates may have restrained the grain growth so that the strengthening contribution from the grain refinement was preserved during the aging process [28].

Wan et al. [29] reported that a small Dy addition in Mg–1Zn–0.6Zr–0.6Dy (wt.%) alloy enhanced ductility promoted by a finer grain structure and weaker texture, where grain refinement caused the strengthening of the material. Regarding the macrotexture, the pole figures shown in Figure 5 exhibited a strong basal {0002} texture. However, an intense texture of the {10 $\bar{1}$ 0} planes was also observed, evidencing the activation of slip prismatic planes during the hot rolling process.

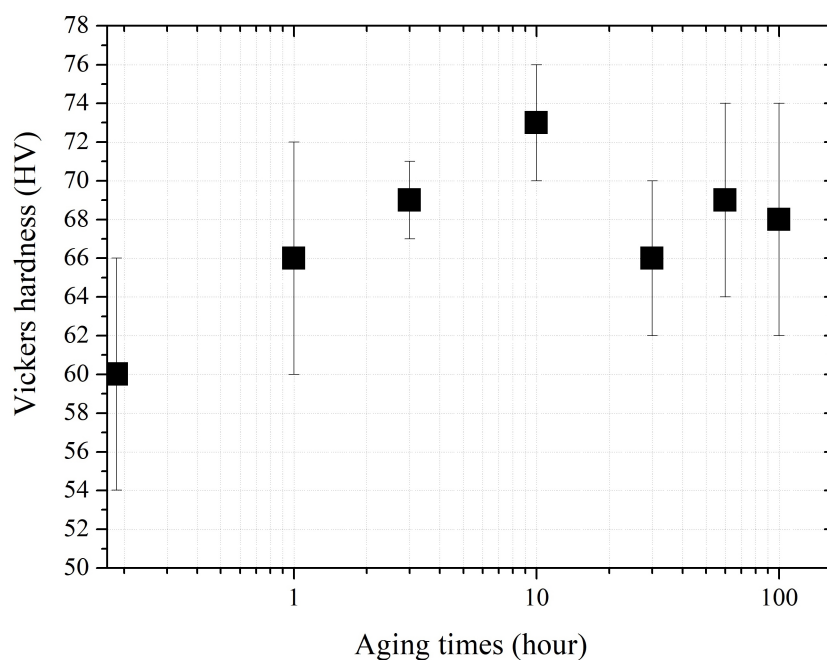


Figure 4. Aging hardening behavior at 250 °C after hot rolling.

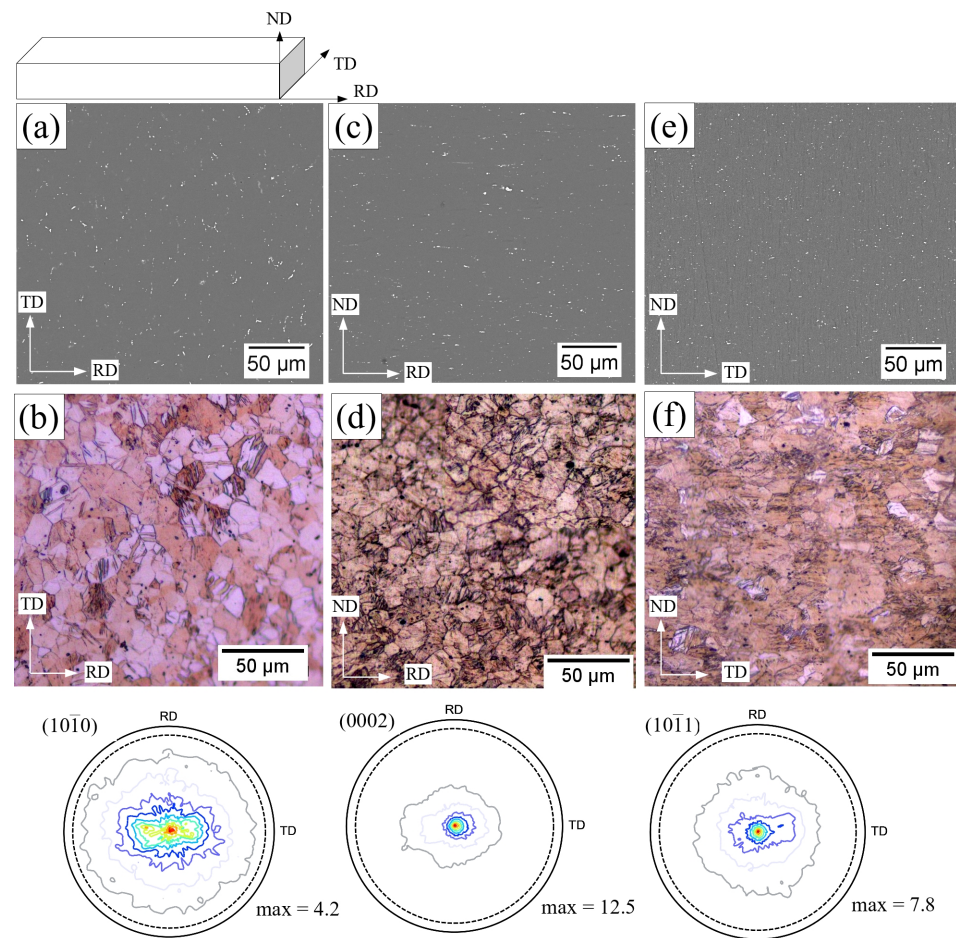


Figure 5. SEM/BSE (a,c,e) and LOM micrographs (b,d,f) of aged-10 h specimen; and pole figures of {10 $\bar{1}$ 0}, {0002} and {10 $\bar{1}$ 1} planes.

3.4. Electrochemical Characterization

Figure 6 depicts the curves of open-circuit potential for the electrochemical tests in 0.9 wt.% NaCl aqueous solution at 37.0 ± 0.5 °C. At the final stage of the test, the OCP data of the H525 °C-3 h, rolled and aged-10 h specimens stabilize on values of -1.568 V, -1.576 V and -1.588 V, respectively. Other Mg alloys, such as Mg-1Ca wt.% [30], WE43 [31] and Mg-6Al-1Zn 0.5Mn-1Ce wt.% [32], as shown in the literature, obtained an OCP stabilized within a small range of -1.62 , -1.62 and -1.59 V, respectively.

Figure 7 shows the potentiodynamic polarization curves of the H525 °C-3 h, rolled and aged-10 h specimens. The results revealed close values for E_{corr} for the H525 °C-3 h, rolled and aged-10 h specimens of -1.429 V/Ag | AgCl_{sat}, -1.475 V/Ag | AgCl_{sat} and -1.479 V/Ag | AgCl_{sat}, respectively. Table 1 presents the corrosion potential (E_{corr}), current density (i_{corr}) and the corrosion rate (CR), which was calculated using Equation (1).

$$CR = C \frac{M i_{corr}}{n \rho} \quad (1)$$

where C is a constant, which includes the Faraday constant and any other conversion factor for units = 0.00327 (mm year $^{-1}$), M is the standard atomic weight of Mg = 24.301 g mol $^{-1}$, ρ is the density of the alloy (g cm $^{-3}$), i_{corr} is the current density (μ A cm $^{-2}$), and n is the number of electrons involved in the corrosion reaction.

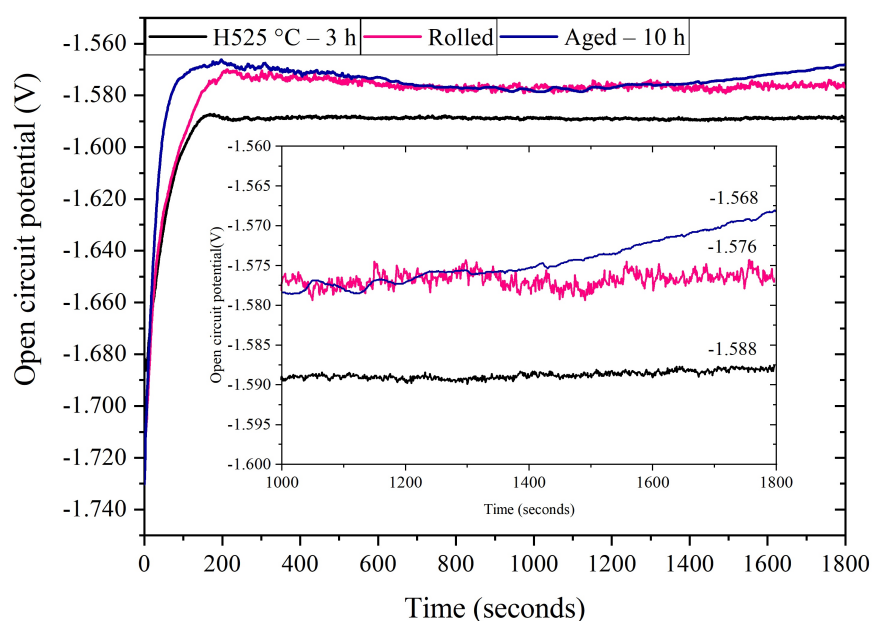


Figure 6. Open circuit potential curves of H525 °C-3 h, rolled and aged-10 h specimens in 0.9 wt.% NaCl aqueous solution at 37.0 ± 0.5 °C.

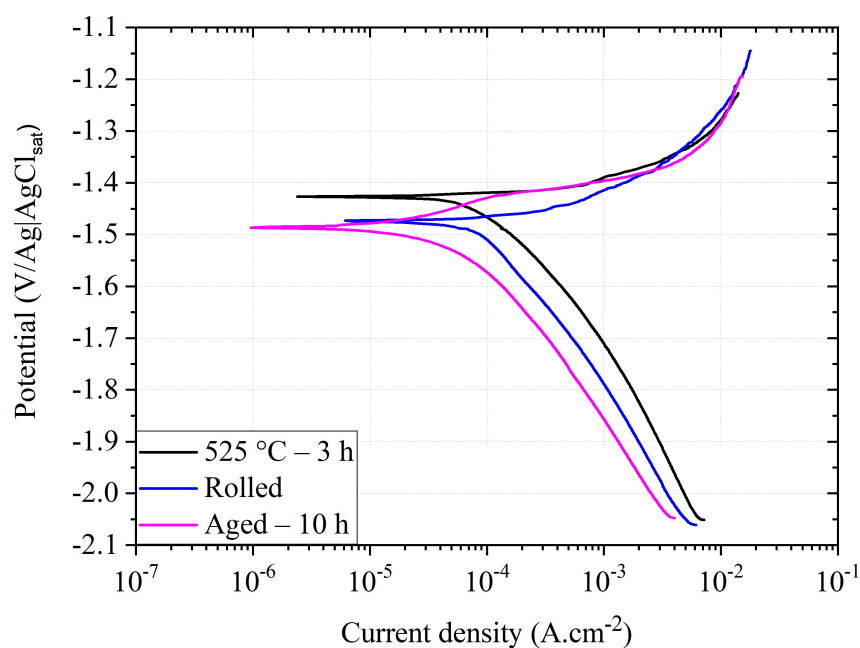


Figure 7. Potentiodynamic polarization curves of H525 °C-3 h, rolled and aged-10 h specimens in 0.9 wt.% NaCl aqueous solution at 37.0 ± 0.5 °C.

Table 1. Electrochemical parameters of H525 °C-3 h, rolled and aged-10 h specimens: corrosion potential (E_{corr}), current density (i_{corr}) and corrosion rate (CR).

Condition	E_{corr} (V/Ag AgCl _{sat})	i_{corr} ($\mu\text{A cm}^{-2}$)	CR (mm Year ⁻¹)
H525 °C-3 h	-1.423 ± 0.011	85.2 ± 3.0	2.0 ± 0.1
Rollled	-1.475 ± 0.020	74.4 ± 3.1	1.7 ± 0.1
Aged-10 h	-1.479 ± 0.021	35.4 ± 9.7	0.9 ± 0.2

The aged-10 h specimen exhibited the lowest corrosion rate of 0.9 mm/year against 2.0 and 1.7 mm/year for the H525 °C-3 h and rolled specimens, respectively. The combination of grain refinement and micrometric precipitates is suggested to be the cause of corrosion resistance improvement. Table 2 shows the corrosion rates of several Mg–REEs alloys for comparisons. Similar corrosion rates were observed when compared to as-cast, solution treated, hot rolled and extruded alloys. A Mg–1Y (wt.%) alloy in the rolled condition presented a corrosion rate of 1.65 mm/year in SBF [33]. Binaries Mg–*x*Dy (*x* = 5 and 10 wt.%) alloys exhibited after heat treatments at 520 °C–24 h a corrosion rate of 0.5 and 0.9 mm/year, respectively [27]. The JDBM (Mg–3.1Nd–0.2Zn–0.4Zr wt.%) alloy extruded and aged at 200 °C for 10 h showed a corrosion rate of 0.18 mm/year in SBF solution [5].

Table 2. Comparison of corrosion rate of H525 °C-3 h, rolled and aged-10 h specimens with data from the literature.

Alloy (wt.%)	Condition	Solution	CR (mm Year ^{−1})	Ref.
Mg–3.4Dy–0.2Zn–0.4Zr	525 °C-3 h	0.9 wt.% NaCl	2.0	This work
Mg–3.4Dy–0.2Zn–0.4Zr	Rolled	0.9 wt.% NaCl	1.7	This work
Mg–3.4Dy–0.2Zn–0.4Zr	Aged-10 h	0.9 wt.% NaCl	0.9	This work
Mg–3.1Nd–0.2Zn–0.4Zr	Extruded–Aged 10 h	SBF	0.18	[5]
WE43	Extruded	SBF	0.125	[34]
Mg–5Dy	520 °C–24 h	0.9 wt.% NaCl	0.5	[27]
Mg–10Dy	520 °C–24 h	0.9 wt.% NaCl	0.9	[27]
Mg–3Gd–1Y	As-cast	9 g L ^{−1} NaCl	3.16	[35]
Mg–4Nd	520 °C–20 h	9 g L ^{−1} NaCl	0.62	[35]
Mg–1Y	Rolled	SBF	1.65	[33]
Mg–2.13Nd	As-cast	SBF	1.25	[36]
Mg–1.27Ce	As-cast	SBF	1.84	[36]
Mg–0.69La	As-cast	SBF	2.15	[36]

Figures 8 and 9 show the results of the EIS tests (Nyquist, Bode and phase angle plots). From the Nyquist diagrams (Figure 8a) of the H525 °C-3 h specimen after 30, 50 and 70 min of immersion times revealed a non-monotonous variation with a decrease for 50 min and then an increase for 70 min of high-frequency capacitive loops, in addition to an inductive loop at low frequencies. For the rolled specimen (Figure 8b), a decrease of high-frequency capacitive loop results was observed with longer immersion times. These phenomena may be connected to an increase of the active surface area promoted by corrosion layer formation [37] during the 70 min of immersion time.

Figure 8c exhibits the aged-10 h specimen data with three loops on the first impedance measurements, two capacitive loops and one inductive loop. The high frequency capacitive loop is typically attributed to the charge transfer resistance [38]. The medium frequency capacitive loop may be related to the relaxation of mass transport in the solid phase [38,39]. This loop disappears after a long immersion time as shown by the second and third impedance curves. The inductive loop at the low frequency is attributed to the relaxation processes of adsorbed species, such as Mg(OH)⁺ and Mg(OH)₂ on the specimen surface [38,40] and also related to pit formation [40].

Figure 9 shows Bode plots and phase angles comparing data from the three conditions analyzed, and a qualitative analysis of these plots shows that the shape of experimental diagrams was quite similar for all conditions. In the range of the swept frequency, the impedance modulus, $|Z|$, at medium frequencies shifted towards higher positive values.

With further corrosion from 30 to 70 min, the impedance value reduced, manifesting that the corrosion resistance gradually worsened. The same behavior was observed for extruded Mg–6.0Zn–1.0Ca wt.% and Mg–6.0Zn–1.0La/Ce wt.% alloys tested in SBF for 30 h [41]. This behavior suggests that the aged-10 h specimen exhibited better corrosion resistance when compared to H525 °C-3 h and rolled specimens as also indicated by the potentiodynamic polarization data shown previously.

Figure 10 shows the equivalent electrical circuit used [31] to model the experimental impedance data, which includes, as the main components, the solution resistance (R_s), a constant phase element (CPE_1) and a resistor (R_1), associated with the double layer and charge transfer resistor (substrate/electrolyte interface) [42,43]. The (CPE_2) and the resistance (R_2) are characteristic of the corrosion process at the metal surface through the corrosion layer and the release of Mg ions.

The constant phase elements (CPE) have been used instead of a capacitance, due to the possible microscopic roughness or the heterogeneities of the alloy surfaces and is defined in terms of n where n is an empirical exponent that ranges from 0 to 1. A perfectly smooth surface corresponds to n equal to unity, which decreases with increasing surface roughness [44]. R_L and L_1 represent the pitting corrosion resistance [45] and inductive behavior associated with the anodic reaction of magnesium oxidation accompanied by local hydrogen evolution.

Table 3 showcases the electrical parameters of the various components of the fitted equivalent circuit based on the EIS data of the different condition of the MgDyZnZr alloys. The results evidently confirmed that the aged-10 h specimen exhibited much better corrosion performance than the H525 °C-3 h and rolled specimens since the polarization resistance ($R_p = R_1 + R_2$) and the pitting corrosion resistance (R_L) had significantly higher values.

Table 3. Fit of the equivalent circuit parameters of H525 °C-3 h, rolled and aged-10 h specimens.

Condition Ω	EIS	$R_s (\Omega \cdot \text{cm}^2)$	$R_1 (\Omega \cdot \text{cm}^2)$	$\text{CPE}_1 (\Omega^{-1} \cdot \text{s}^n \cdot \text{cm}^{-2})$	n_1	$R_2 (\Omega \cdot \text{cm}^2)$	$\text{CPE}_2 (\Omega^{-1} \cdot \text{s}^n \cdot \text{cm}^{-2})$	n_2	$R_L (\Omega \cdot \text{cm}^2)$
H525 °C-3 h	1°	15.99 ± 1.06	89.25 ± 2.13	$4.5 \times 10^{-5} \pm 3.89 \times 10^{-6}$	0.98 ± 0.01	9.18 ± 2.73	$1.88 \times 10^{-6} \pm 2.17 \times 10^{-7}$	0.60 ± 0.04	8.06 ± 0.02
H525 °C-3 h	2°	1.02 ± 0.08	84.86 ± 1.42	$7.17 \times 10^{-5} \pm 4.45 \times 10^{-6}$	0.93 ± 0.01	18.20 ± 0.20	$5.30 \times 10^{-9} \pm 4.75 \times 10^{-10}$	0.70 ± 0.03	12.99 ± 0.14
H525 °C-3 h	3°	0.52 ± 0.09	142.4 ± 1.30	$9.53 \times 10^{-5} \pm 2.98 \times 10^{-6}$	0.93 ± 0.01	18.62 ± 0.11	$2.59 \times 10^{-9} \pm 2.76 \times 10^{-10}$	0.70 ± 0.02	13.76 ± 0.12
Rolled	1°	12.79 ± 0.16	144.6 ± 1.21	$2.13 \times 10^{-5} \pm 4.07 \times 10^{-7}$	0.95 ± 0.02	8.95 ± 1.00	$6.86 \times 10^{-6} \pm 1.30 \times 10^{-7}$	0.74 ± 0.05	8.21 ± 0.83
Rolled	2°	12.87 ± 0.13	135.5 ± 0.73	$3.40 \times 10^{-5} \pm 3.26 \times 10^{-7}$	0.95 ± 0.02	6.46 ± 0.24	$2.82 \times 10^{-6} \pm 3.90 \times 10^{-7}$	0.60 ± 0.04	6.03 ± 0.20
Rolled	3°	12.27 ± 0.14	137.6 ± 0.94	$4.78 \times 10^{-5} \pm 1.24 \times 10^{-6}$	0.95 ± 0.01	4.32 ± 0.19	$2.28 \times 10^{-6} \pm 4.60 \times 10^{-7}$	0.60 ± 0.01	4.17 ± 0.17
Aged-10 h	1°	19.74 ± 0.11	$17,361.0 \pm 1607$	$3.66 \times 10^{-4} \pm 5.54 \times 10^{-5}$	0.95 ± 0.03	477.2 ± 7.86	$8.81 \times 10^{-6} \pm 2.08 \times 10^{-7}$	0.91 ± 0.01	462.4 ± 7.21
Aged-10 h	2°	21.25 ± 0.15	402.3 ± 20.0	$1.44 \times 10^{-5} \pm 6.15 \times 10^{-7}$	0.95 ± 0.02	106.9 ± 20.6	$2.58 \times 10^{-5} \pm 3.59 \times 10^{-6}$	0.70 ± 0.05	65.54 ± 5.97
Aged-10 h	3°	21.66 ± 0.20	335.3 ± 20.8	$2.28 \times 10^{-5} \pm 1.46 \times 10^{-6}$	0.95 ± 0.03	98.09 ± 23.7	$1.25 \times 10^{-5} \pm 1.80 \times 10^{-6}$	0.66 ± 0.04	61.66 ± 6.95

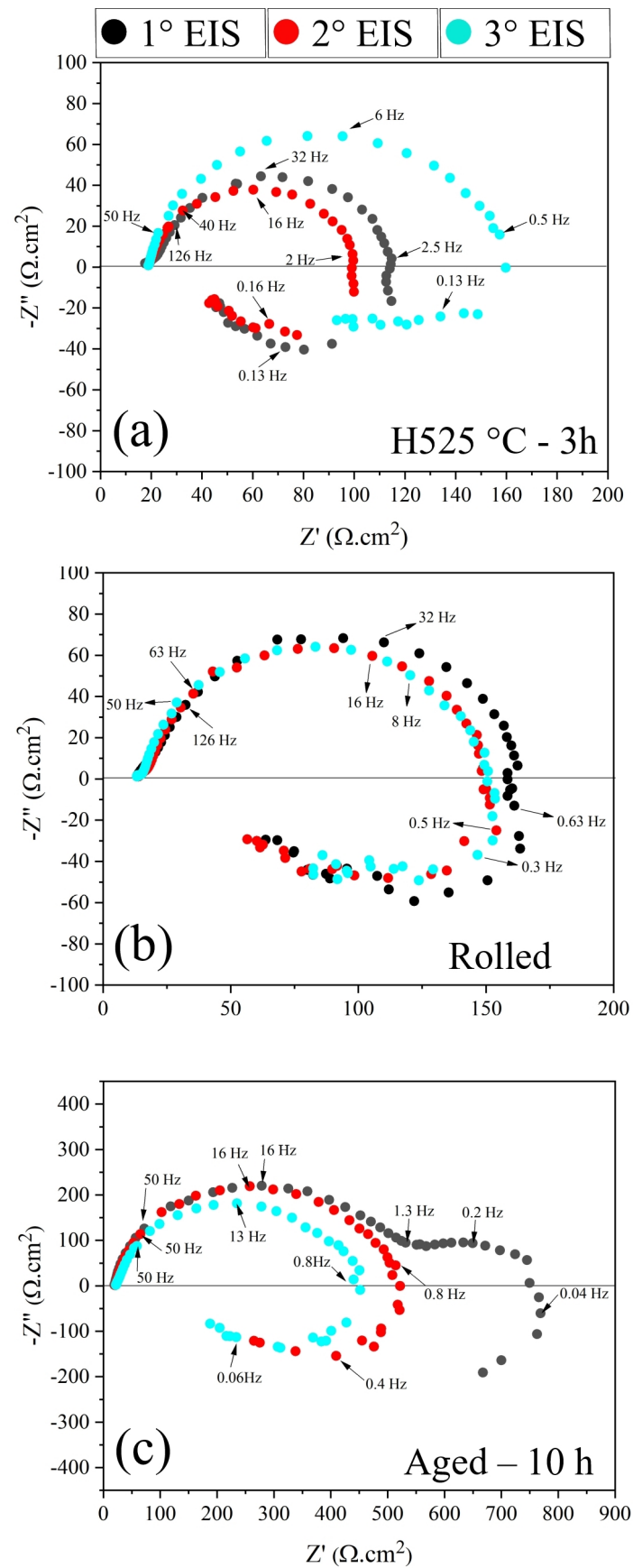


Figure 8. Nyquist curves after 30 min (1°EIS), 50 min (2°EIS) and 70 min (3°EIS) of immersion times (a) H525 °C-3 h, (b) rolled and (c) aged-10 h specimens in 0.9 wt.% NaCl aqueous solution at 37.0 ± 0.5 °C.

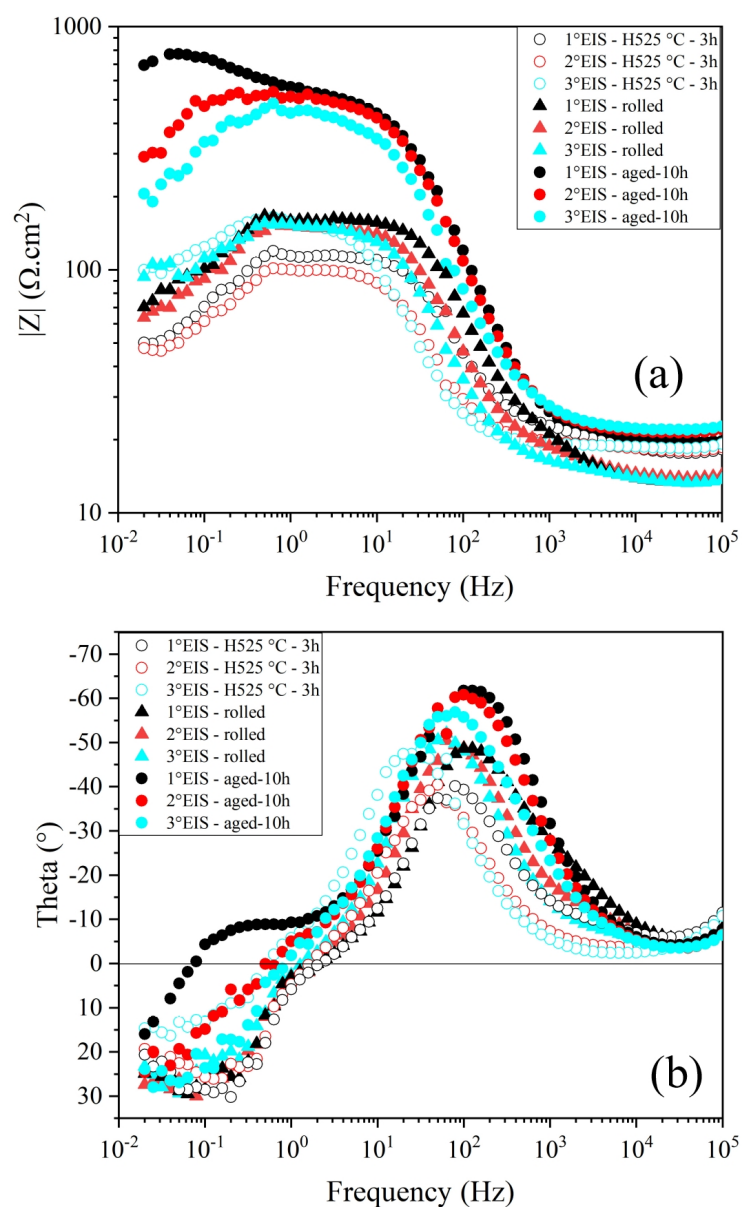


Figure 9. (a) Bode curves and (b) phase angle of H525 °C-3 h, rolled and aged-10 h specimens in 0.9 wt.% NaCl aqueous solution at 37.0 ± 0.5 °C.

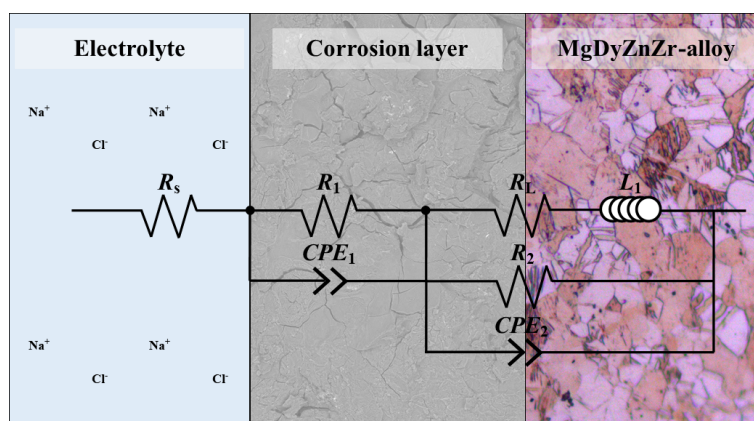


Figure 10. Equivalent electric circuit used to model the experimental impedance data of MgDyZnZr alloy specimens immersed in 0.9 wt.% NaCl solution at 37 ± 0.5 °C

4. Conclusions

In the present work, through observing the microstructure and investigating the corrosion behavior of Mg–3.4Dy–0.2Zn–0.4Zr (wt.%) alloys in 0.9% NaCl solution at 37 ± 0.5 °C after homogenization heat treatment, hot rolling and aging. The following conclusions can be drawn:

- Homogenization heat treatments at 525 °C for 3 h successfully avoided any inhomogeneity that otherwise occurred during the melting process.
- The hot rolling process with multiple passes promoted a homogeneous grain-refined microstructure for all rolling directions with a basal {0002} texture.
- A combination of hot rolling and aging heat treatments at 250 °C for 10 h promoted an alloy with a small grain size with thin, dispersed precipitates.
- These precipitates dispersed after aging at 250 °C for 10 h collaborated for an increase in the strength of the alloy as shown by the hardness increase.
- These characteristics of grain refinement and thin, dispersed precipitates led to a better corrosion resistance of aging at 250 °C for 10 h specimens as shown by a lower corrosion rate.

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