Effect of texture evolution on corrosion resistance of AZ80 magnesium alloy subjected to applied force in simulated body fluid

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Abstract
The electrochemical corrosion behavior of a rolled AZ80 magnesium alloy after pre-compressive strain along different orientation in a simulated body fluid (SBF) was investigated by immersion test for 48 h. The results showed that the corrosion resistance is related to the crystal texture of the material. The texture evolution induced by plastic deformation changed the corrosion behavior of the magnesium alloy. Slip and twin associated with plastic deformation changed the orientation of crystallographic planes. The surface containing highly concentrated orientation of (0002) basal plane has higher corrosion resistance. The appearance of {10 \overline{1} 2} tension twinning could improve the corrosion resistance of magnesium alloy with specific orientation. However, the coexistence of {10 \overline{1} 2} tension twinning with high volume fraction and slip with high density dislocation reduced the corrosion resistance and corrosion anisotropy of the magnesium alloy.

1. Introduction

The mechanical properties (density, elasticity modulus and compressive strength, etc) of degradable magnesium alloys are similar to those of human bone, which is suitable for load bearing bone implants [1]. Unlike traditional stainless steel and titanium alloy implants, magnesium alloys effectively minimize stress shielding effect and avoid secondary surgery due to they can gradually dissolve in physiological environment [2]. However, the rapid degradation rate of magnesium alloy resulted in the loss of mechanical integrity of the implant before required function is finalized [3].

The bone implants are often subjected to the interaction of mechanical stress and corrosion medium when they are in service in human body. However, most previous studies on biomedical magnesium alloys focused on biodegradation behavior investigated by immersion test in SBF [4–9]. Recently, Zhang et al [10] investigated the corrosion behavior of bioglass-ceramic coated magnesium alloys under different compressive stress in SBF by in vitro immersion test. The results showed that the corrosion rate of the sample under different applied forces is very different. It is well know that the deformation texture have a typical feature in the forging magnesium alloy due to their processing method. Basal texture can be formed by rolling and fiber texture by extrusion. There are two inevitable problems when magnesium alloy is used as implanted device. One is the corrosion anisotropy caused by texture orientation of material. The difference of corrosion rate in the different oriented materials is due to the different corrosion sensitivity of crystallographic planes [11–13]. Other is the corrosion effect induced by applied force related to plastic deformation. Plastic deformation process affected the microstructure (such as grain size and dislocation density) and thus changed the corrosion resistance of magnesium alloy [14, 15]. The previous work of the author showed that the crystal texture of magnesium alloy was changed when the rolled or extruded magnesium alloy was subjected to applied forces along different material orientations [16, 17]. It can be inferred that the corrosion resistance of magnesium alloy may be changed with the evolution of crystal texture caused by applied force. The effect of grain orientation or plastic deformation on the corrosion resistance of...
magnesium alloys in NaCl solution has been reported by some researchers [11–15]. However, the texture effect of applied force on corrosion resistance of magnesium alloys in physiological environment has not been reported.

The purpose of this work is to investigate the corrosion behavior of rolled AZ80 magnesium alloy with different crystal textures, which is obtained at different loading levels along different material orientation. The effect of crystal texture on corrosion resistance of AZ80 magnesium alloys is discussed.

2. Experimental

2.1. Material and sample
The material used in the study is a hot-rolled AZ80 magnesium alloy plate (7.8 wt% Al, 0.36 wt% Zn, 0.04 wt% Mn, Mg balance) with a thickness of 60 mm. The original microstructures and mechanical properties of the material were reported in earlier publication [17]. The result observed by electron back scattered diffraction (EBSD) indicated that the material exhibits a typical basal texture, and the basal planes are parallel to the rolled plane [17]. The sample diagram for this study is shown in figure 1. To visualize the initial texture of material, a schematic of hexagonal close packed (hcp) unit cell is embedded in figure 1, their c-axes approximately perpendicular to the rolled plane. The typical three directions are denoted as rolling direction (RD), normal direction (ND) and tangential direction (TD). Dog-bone shaped specimens were machined from the rolled plate with the loading axes of the testing specimens along 0° (ND) and 30° (ND30) with respect to the ND. Each specimen has a gage length of 15 mm and a diameter of 10 mm within the gage section. The two orientation specimens were compressed to different strain level along the cylinder axis of the specimen. The compression tests were conducted using an INSTRON testing machine at room temperature and a strain rate of $6.0 \times 10^{-3}$/s. For the ND specimens, tests were interrupted at strains of 2%, 5% and 7%. For the ND30 specimens, tests were stopped at strains of 3%, 6% and 9%. In order to investigate the effect of texture evolution on corrosion resistance under different applied force, these pre-compressed specimens were re-machined to cylindrical samples with the size of Ø10 mm × 2 mm. The plane of diameter 10 mm parallel to rolled plane. Prior to the experiment the samples were ground up to 1200 grit silicon carbide paper, followed by washing with distilled water and cleaned ultrasonically in acetone. The sample was embedded in the resin, and the circular surface with an area of 0.785 cm² was exposed to SBF solution.

2.2. Electrochemical test
The SBF solution performed in immersion experiment is composed of $6.8 \text{ g L}^{-1} \text{ NaCl, 0.1 g L}^{-1} \text{ MgSO}_4, 0.2 \text{ g L}^{-1} \text{ CaCl}_2, 2.2 \text{ g L}^{-1} \text{ NaHCO}_3, 0.126 \text{ g L}^{-1} \text{ NaH}_2\text{PO}_4, 0.026 \text{ g L}^{-1} \text{ Na}_2\text{HPO}_4, 0.4 \text{ g L}^{-1} \text{ KCl, pH 7.4}$ [18]. EIS tests were performed on an electrochemical workstation with a standard three-electrode cell. Saturated calomel electrode (SCE) was used as a reference electrode, platinum electrode as the counter electrode and the sample as the working electrode. The ratio of the test solution volume (mL) to the specimen surface area (cm²) was 300 mL cm⁻². Three times electrochemical test is performed for each sample. Electrochemical impedance spectroscopy (EIS) measurement were collected in a range of 0.01 Hz to 100k Hz with AC voltage amplitude of
10 mV, and carried out after the samples were immersed for 1, 4, 8, 24 and 48 h, respectively. Before the beginning electrochemical EIS tests, the samples were hold at the open circuit potential (OCP) for 30 min. EIS data was analyzed using the ZSimpWin software.

2.3. Microstructural characterization
The texture components of the samples were identified by x-ray diffractometer (XRD) using Cu Ka radiation at the 20–60° (in 2θ) and steps of 0.02°. The surface morphologies of the corroded samples were observed by scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS) system.

3. Results
3.1. XRD analysis
Figure 2 shows the XRD scans at various compressive strains for different orientation samples. The XRD scans observed on the RD-TD plane is a surface exposed to the SBF solution. For the ND series samples (figure 2(a)), the values of (0002), (1010), and (1120) diffraction peaks hardly change with the increase of compression strain. The reason is that the compression stress is parallel to the c-axes of grains, and (1012) tension twinning is difficult to be activated. Therefore, the crystallographic planes of the sample exposed to SBF solution mainly are the (0002) basal plane. For the ND30 series samples (figure 2(b)), (0002) peak decreases rapidly and (1010) peak rises at the compressive strain of 3% compared to as-rolled material. As the compressive strain increases from 3% to 9%, (0002) peak increases significantly and (1010) peak increases slightly. Others, (1012) peaks in ND30 pre-compressed samples (ND30%-3%, ND30%-6% and ND30%-9%) are higher than that of ND pre-compressed samples (ND-2%, ND-5% and ND-7%). The previous research results show that the basal (a) slip dominant deformation in the ND30 orientation sample before the compressive strain of 2%, after that the (1012) tension twinning nucleates gradually with the strain increasing [19]. In a word, the RD–TD plane in ND series samples consists mainly of the (0002) basal plane, whereas the one plane in ND30 series pre-compressed samples consists mainly of the (0002) basal plane, the (1010) pyramidal plane and the (1012) plane.
3.2. EIS measurement

EIS is an effective method to evaluate the degradation behavior of materials. In order to obtain information of corrosion behavior during immersion, the EIS spectra of all the samples immersed in SBF were recorded for prolonged duration up to 48 h. Figure 3 shows EIS plots of different samples at various immersion times. It can be clearly seen that the EIS plots of all the samples show similar characteristics. Nyquist plots consist of a capacitive semicircle in the high frequency region and a relatively small capacitive semicircle in the medium frequency region, and the phase angles present two peaks in Bode plots. According to the EIS test results, an equivalent circuit for fitting EIS plots of these samples is shown in figure 4. In the equivalent circuit, the constant phase element (CPE) is used instead of a pure capacitance (C) to compensate the error caused by the non-

Figure 3. EIS plots (Nyquist, Bode-resistance and Bode-theta) and fitting results at various immersion times for all the samples tested in SBF.
homogeneity in the system [20–22]. $R_s$ is the solution resistance. The medium-frequency capacitive loop is described by the film capacity (CPE$_1$) and the film resistance ($R_f$). The high-frequency capacitive loop can be characterized by the electric double-layer capacity (CPE$_2$) and the charge transfer resistance ($R_t$). The impedance plots are fitted with this equivalent circuit and the fitting curves also are shown in figure 3. The values of circuit elements calculated from the fitting results of all the samples are listed in table 1.

According to the variation of impedance response fitting parameters with time, the corrosion kinetics can be better understood. The variation of fitting parameters ($R_s$ and $R_t$) of ND and ND30 series samples with immersion time are shown in figures 5(a) and (b), respectively. It is worth noting that the $R_s$ values of all the samples suddenly increased in the initial stage of immersion (0–1 h). This indicates that the original MgO film is permeated rapidly in the SBF solution in the period. Because the MgO passivation film formed on the surface of
magnesium alloy is unstable in SBF solution, and hydration of MgO leading to formation of Mg(OH)_2

\[ \text{MgO} + \text{H}_2\text{O} \rightarrow \text{Mg(OH)}_2 \]

in the early stage of immersion [23, 24]. Newly formed Mg(OH)_2 film provides a barrier effect and has a high resistance to resisting the localized damage [25, 26]. With the immersion time increases from 1 h to 4 h, PO_4^{3-} will preferentially bond with Mg^{2+} and Ca^{2+} to form corrosion compound such as phosphate, and deposited onto the Mg(OH)_2 film [9]. The compound layer reduces the porosity of Mg(OH)_2 film and thus increases the charge transfer resistance (R_2). However, the corrosion behavior of ND series and ND30 series samples is different during the subsequent immersion period (4–48 h). For the ND pre-compressed samples (ND-2%, ND-5% and ND-7%), the values of R_2 decrease slightly when immersion time increases from 4 h to 8 h, and then maintained relatively stable after 8 h until 48 h. For the ND30 pre-compressed samples (ND30%-3%, ND30%-6% and ND30%-9%), the values of R_2 increase first and then decrease with immersion time. However, the maximum values of R_2 corresponded to different immersion time. The R_2 peaks corresponding to ND-3%, ND-6% and ND-9% samples are 8 h, 24 h and 4 h. Over the peaks, the R_2 value decrease gradually with the prolongation of immersion time. The increase in R_2 value before the peak is due to the blocking effect of corrosion products on the porous layer of Mg(OH)_2 film, which prevents the permeation of SBF solution. The decrease in R_2 value after the peaks is attributed to the Cl^- ions in the corrosion solution attack the Mg(OH)_2, which results in the partial dissolution of the corrosion products layer and the formation of diffusion channels (Mg(OH)_2 + 2Cl^- MgCl_2 + 2OH^-). For the ND pre-compressed samples, the SBF solution permeates into the film/matrix interface at a steady rate. However, the permeation rate of ND30 pre-compressed samples is faster than that of ND pre-compressed samples, which should be attributed to the increase in the diffusion channels.

The smaller R_2 values observed from ND30%-9% and ND samples can be related to the occurrence of a localized corrosion process, i.e., pitting [4]. The surface corrosion morphologies of different samples observed at various immersion times are shown in figure 6. The ND, ND-2% and ND30%-9% samples show obvious local corrosion characteristics. The surface morphology of other samples is similar, that showed relatively uniform corrosion characteristics. The surface morphologies and the elemental analysis on the ND-2% and ND30%-3% samples are depicted in figures 7 and 8. Figures 7(a) and 8(a) show surface morphology of the both samples.
observed by SEM after immersing for 48 h. It can be observed that there are many cracks and accumulation on
the surface of sample. The EDS spectrums evidenced in the localized degraded areas marked by the red square
are shown in figures 7(b) and 8(b). The results indicates that the chemical elements of this area are mainly
composed of C, O, Na, Mg, P, Cl and Ca. It is confirmed that the corrosion products such as magnesium
hydroxide, carbonates and phosphates are formed on the surface of sample during SBF immersion [27]. The
presence of Cl⁻ ions in the localized corrosion site reveals its contribution to pitting corrosion [4, 9]. Ca/Mg-
phosphates can effectively improve corrosion resistance of magnesium alloys [28, 29]. The content of Ca, P elements in ND30%-6% sample is higher than that in ND-2% samples, which indicates that the number of phosphate compound layers formed on the former is higher than that on the latter. This result further confirms that the corrosion resistance \( R_2 \) of ND30%-6% sample is higher than that of ND-2% sample.

Although SBF solution is penetrated into the interface between film layer and the substrate after immersing for 48 h, the penetration to the substrate may not occurred yet. It is proved by the fact that the \( R_z \) values are higher than \( R_c \) values in the tested all samples during the whole immersion process. Therefore, the charge transfer resistance \( R_z \) indirectly reflects the deterioration process and corrosion rate of the material in the SBF medium. The higher \( R_z \) value means that the samples have a greater corrosion resistance or lower corrosion rate. The results above show that the corrosion rate of ND-7% is the lowest and that of ND-2% is the highest among ND pre-compressed samples. The corrosion rate of ND30%-6% is the lowest and that of ND-9% is the highest among ND30 pre-compressed samples. The corrosion rate of ND substrate is similar to that of ND-9% sample. The corrosion rate is related to the number of diffusion channels, which would be attributed to the evolution of crystal texture after pre-compressive train. This will be discussed in the next section.

4. Discussion

The experimental results show that the corrosion rate is impacted by initial crystallographic orientation. Song et al [12] and Liu et al [30] reported that the theoretical dissolution rates of (10\( \bar{1} \)0) and (11\( \bar{2} \)0) planes are around 18–20 times higher than that of the (0002) basal plane, therefore, the corrosion resistance of the basal plane is much better than that of other crystallographic planes. In order to find out the relationship between corrosion rate and crystal texture, two parameters are defined. The orientation concentration parameter of crystallographic planes, \( \gamma \) is related to the crystal texture. The corrosion resistance parameter, \( \psi \) is related to the corrosion rate. According to the peak intensity of crystallographic planes measured by XRD (figure 2),

\[
\beta = \frac{ \gamma(10\bar{1}0) + (11\bar{2}0) }{ \gamma(0002) } \]

is calculated, and then \( \beta \) of ND sample is taken as denominator and \( \beta \) of pre-compressed samples is used as molecule to calculate the parameter \( \gamma \). The charge transfer resistance \( R_z \) of ND sample as denominator and \( R_z \) of pre-compressed samples as molecules to calculate the parameter \( \psi \). Figure 9 shows the relationship between \( \gamma \) and \( \psi \) at various immersion times for different samples. According to the previous results [12, 30], the larger the crystal texture parameter \( \gamma \), the higher the corrosion resistance parameter \( \psi \). It can be seen that ND series samples (ND-2%, ND-5% and ND-7%) conform to this principle, but ND30 series (ND30%, ND30%-6% and ND30%-9%) samples do not conform to it. The reason is that the evolution of crystal texture after pre-compression leads to the change of corrosion resistance.

For the ND pre-compressed samples, the basal \{a\} slip and \{10\( \bar{1} \)2\} tension twinning are difficult to be activated when compressive loading along the ND, and the deformation mechanism is mainly non-base slip mode [19]. The RD-TD plane exposure to SBF solution mainly contains (0002) basal planes (figure 2(a)). With the strain increasing, \((c+a)\) slip may be activated, resulting in a change in the crystal orientation of some grains. This also changes the peak values of (10\( \bar{1} \)2) (10\( \bar{1} \)0) (10\( \bar{1} \)) planes in RD-TD plane (figure 2(a)). The result is that the texture parameter \( \gamma \) increases with increasing strain. Therefore, the corrosion resistance of ND-7% sample with higher value of \( \gamma \) is the highest among the ND pre-compressed samples.

For the ND30 pre-compressed samples, the deformation behavior depends on the competition between slip and twinning [19]. At the compression strain of 3%, the deformation mechanism of ND30%-3% sample is mainly the basal \{a\} slip. With the compression strain increases from 3% to 6%, the volume fraction of \{10\( \bar{1} \)2\}
tension twinning increases evidently [19]. Because of the lower coordination number, the surface energy of atoms in \{10\bar{1}2\} tension twinning plane is higher than that in (0002) basal plane [31]. It indicates that the oxide film can be preferentially formed in twinning plane with higher oxidation activity, and thus increased the charge transfer resistance \( R_2 \) [32]. Compared with ND30%-3% sample, ND30%-6% sample has both high twinning fraction and high texture parameter, which enhanced the growth of oxide film and thus improved corrosion resistance of the material. When the compression strain is increases to 9%, the basal \(<a>+\) and pyramidal \(<c+a>\) slips participate in the plastic deformation in addition to the \{10\bar{1}2\} tension twinning [19]. The interaction between slip and twinning may affect the integrity of oxide film. The reason is that the higher dislocation density and twins decreased the electrochemical potential of AZ80 matrix and increased anodic dissolution [14, 33]. In this case, the corrosion product film degenerated widely, which further reduced the charge transfer resistance \( R_2 \). Therefore, the corrosion resistance of ND30%-9% sample with higher value \( \gamma \) is lower than that of ND30%-3% and ND30%-6% samples.

Comparing ND series with ND30 series samples, it can be seen that the corrosion resistance \( (\psi) \) of ND30%-3% and ND30%-6% samples with twins is higher than that of ND series samples without twins during SBF immersion. This shows that the corrosion resistance of specific oriented alloys can be improved by containing a certain number of twins. However, when the material is subjected to large compression deformation, the corrosion resistance of ND30%-9% sample with high twin volume fraction is lower than that of ND-7% sample without twins at each immersion time. This result further confirms that the combination of higher fraction twinning and higher density dislocations reduces the corrosion resistance of orientation sample, and thus weakens the corrosion anisotropy caused by the initial crystal texture.

The experimental results show that the corrosion rate of magnesium alloy in SBF solution can be controlled by adjusting the crystal texture. Additional, a magnesium-containing calcium phosphate layer can be formed on the surface of magnesium alloy during biodegradation, which is beneficial to the rapid formation of new bone around magnesium implant [34]. Therefore, adjusting the crystal texture of magnesium alloy is expected to meet
the medical requirements for the mechanical integrity of magnesium alloys as implanted devices in a certain period.

5. Conclusion

In this work, the Effect of texture evolution on corrosion resistance of a rolled AZ80 magnesium alloy in SBF is investigated. The pre-compression strain changed the crystal texture of the alloy and resulted in the change of corrosion behavior.

(1) At the early stage of immersion, a corrosion production layer formed on the sample surfaces exposed to the SBF solution. With the increase of immersion time, the corrosion layer degraded gradually.

(2) Corrosion resistance is sensitive to the composed crystallographic planes in the surface of sample exposed to the SBF solution. The surface containing highly concentrated orientation of (0002) basal plane has high corrosion resistance.

(3) A certain number of {1012} tension twinning could improve the corrosion resistance of magnesium alloy with specific orientation. Because the {1012} twinning planes with high atomic surface energy promotes the formation of oxide film, it effectively prevents the contact between the solution medium and the alloy surface.

(4) The coexistence of high volume fraction {1012} tension twinning and high density dislocation slip reduced the corrosion resistance and corrosion anisotropy of magnesium alloy. Because a large number of dislocations and twins lead to the decrease of electrochemical potential, which promoted anodic dissolution.

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