Microstructure and corrosion behaviors of AZ63 magnesium alloy fabricated by accumulative roll bonding process

Yanbo Pei1,2,3, Yunwei Gui1,2,3, Tao Huang1,2,3, Fuxiao Chen1,2,3, Junqing Guo1,2,3, Shiyu Zhong4 and Zhuo Song1,2,3

1 School of Materials Science and Engineering, Henan University of Science and Technology, Luoyang 471023, People’s Republic of China
2 Collaborative Innovation Center of Nonferrous Metals, Luoyang 471023, People’s Republic of China
3 Key Laboratory of Materials Science & Processing Technology for Non-Ferrous Metals of Henan, Luoyang 471023, People’s Republic of China
4 Chengdu Galaxy Power Co. Ltd, Chengdu 610000, People’s Republic of China
5 Authors to whom any correspondence should be addressed. E-mail: huangtao@haust.edu.cn and chenfx126@126.com

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Abstract

AZ63 magnesium alloy sheets were fabricated by accumulative roll bonding (ARB) process at 350 °C up to 5 cycles. The aim of this work is to study the effect of ARB process on the microstructure and corrosion behavior of AZ63 magnesium alloy. The ARBed sheets were investigated via microstructure observations, electrochemical tests, hydrogen evolution measurements and immersion tests. After ARB process, the average grain size decreased. The coarse Mg17Al12 phase was broken under the action of rolling force and it distributed more dispersed in the magnesium matrix. The corrosion resistance of samples increased significantly with increasing the number of ARB cycles. The influence of microstructure on the corrosion behaviors was discussed. Fine grain produced more passive oxide layers which acted as a corrosion barrier. The Mg17Al12 phase acted primarily as a micro-galvanic cathode during the corrosion process because of its low volume fraction and isolated distribution. The volume fraction of Mg17Al12 phase decreased after ARB process, which results in the reduction of the effect of galvanic corrosion.

1. Introduction

Magnesium alloys are increasingly used for light weight structures in the fuel vehicle and aerospace industries due to their low density, high specific stiffness and outstanding damping capacity [1–3]. However, the poor corrosion resistance of magnesium alloys is a major limitation to their wider application [4–6]. Over the last few years, the corrosion behavior of magnesium alloys has been researched to comprehend the factors that affect their corrosion behavior and achieve adequate corrosion resistance [7, 8].

One way to overcome this main obstacle of magnesium alloys is modifying their microstructure. Grain size plays an important role in the corrosion resistance. Aung et al investigated the effect of grain size on the corrosion behavior of AZ31 magnesium alloy and found that the corrosion rate decreased as the grain size decreased [9]. Birbilis et al investigated the effect of grain refinement on the corrosion resistance of magnesium alloy with the grain sizes in the range of 2–100 μm. It was found that the corrosion resistance improved as the grain refined, which could be attributed to a more denser passive oxide layers of MgO and Mg(OH)2 forming on the upper surface of the magnesium alloy [10]. In the Mg-Al-Zn alloys, the most common second phase is intermetallic Mg17Al12, which also play important role in the corrosion process. Therefore, in order to understand the corrosion mechanism of AZ63 magnesium alloy, it is necessary to understand the role of the Mg17Al12 phase in the corrosion. Song et al found that the free corrosion potential of the Mg17Al12 phase was more positive than that of the magnesium matrix in 3.5 wt.% NaCl solutions [11]. Zhao et al researched the effect of the Mg17Al12 phase on the corrosion behavior of AZ91 magnesium alloy. The results showed that the
The grain evolution in the rolling direction was observed by Olympus-BX51M OM. Samples were ground and polished, then etched in a solution of 2.5 g picric acid, 2.5 ml acetic acid, 5 ml H2O and 50 ml ethanol. The phase composition of the samples was analyzed using D8 ADVANCE XRD with a scanning rate of 8°/min. The microstructure correlated to the corrosion behavior after ARB process has become an important subject over the past decades. Babaiee et al evaluated the microstructure and electrochemical properties of ARBed Al/nanosilica sheets. The UFG microstructure was formed after ARB process, resulting in the formation of uniform and dense passive oxide layer on the samples surface, which improved the corrosion resistance [19]. Nikfahm et al studied the corrosion behavior of the pure copper sheets fabricated by ARB process. The microstructure became more stable after ARB process and the corrosion current of the ARBed sample decreased due to the formation of UFG microstructure [20]. Darimi et al investigated the corrosion behavior of Al/SiC composite sheets produced by ARB process and concluded that the corrosion resistance increased with increasing the number of ARB cycles because of the microstructure refinement [21]. However, up to now, there are few systematic works on the effect of ARB process on the corrosion behavior of AZ63 magnesium alloy. Thus, this paper has given eyes on these aspects, could be seen a supplement for this field.

In this work, the microstructure evolution of the AZ63 magnesium alloy after ARB process was systematically examined using optical microscope (OM), x-ray diffractometer (XRD), transmission electron microscopy (TEM) and scanning electron microscopy (SEM). Meanwhile, the corrosion behaviors of the samples were investigated by electrochemical experiments, immersion tests and hydrogen evolution measurements. Finally, the influence of microstructure on the corrosion behaviors following ARB process were discussed. The results of this research have certain guiding significance for the practical application of AZ63 magnesium alloy.

### 2. Materials and methods

#### 2.1. Materials

Commercial AZ63 magnesium alloy sheets were used in this research. Table 1 lists the detailed chemical composition of the sheets. The original sheets have been cut with dimensions 100 mm × 60 mm × 2 mm for ARB process. The surface of the sheets was cleaned by acetone and wire-brushed to remove some of the pre-existing oxide films. After surface treatment, two stacks of original sheet were kept at 350 °C for 30 min to reach the rolling temperature. After roll bonding with 50% reduction, samples were air-cooled. Then the sheets were halved and the process was repeated for 5 times. All the ARB processes were carried out on a 180 mm diameter roll mill at a rolling speed of 18 m min⁻¹ without lubrication. The schematic illustration of the ARB process is shown in figure 1.

| Element | Al  | Zn  | Mn  | Si  | Cu  | Ni  | Fe  | Mg  |
|---------|-----|-----|-----|-----|-----|-----|-----|-----|
| Content | 6.37 | 2.96 | 0.44 | 0.007 | 0.005 | 0.002 | 0.001 | Balance |

#### 2.2. Microstructural studies

The grain evolution in the rolling direction was observed by Olympus–BX51M OM. Samples were ground and polished, then etched in a solution of 2.5 g picric acid, 2.5 ml acetic acid, 5 ml H2O and 50 ml ethanol. The phase composition of the samples was analyzed using D8 ADVANCE XRD with a scanning rate of 8° min⁻¹ and a step size of 0.02. The morphology evolution of the second phase after ARB process was observed by TEM. JSM-5610LV SEM was used to observe the distribution of the second phase after ARB process.

#### 2.3. Electrochemical experiments

Electrochemical experiments were performed using CHI660D electrochemical workstation. Samples with dimensions of 10 mm × 10 mm were prepared from the original sheet and the ARBed sheets. Both samples were covered by the epoxy resin leaving only 1 cm² surface, then mechanically polished by abrasive papers 1000,
1200, 1500 and 2000 grits. Electrochemical measurements were conducted on using a conventional three-electrode glass cell with an auxiliary electrode of graphite, a reference electrode of saturated KCl/calomel and a working electrode of AZ63 magnesium alloy samples. Samples were first immersed in 3.5 wt.% NaCl solutions at room temperature for 60 min to record the open circuit potential (OCP) values. Electrochemical impedance spectroscopy (EIS) measurements were performed at OCP with the amplitude of 5 mV and a frequency interval of 10⁵ Hz to 0.1 Hz. Polarization tests were carried out after the EIS studies with a scanning rate of 0.5 mV s⁻¹. The measurements were duplicated at three times to confirm good reproducibility.

2.4. Hydrogen evolution measurement and immersion test
The hydrogen gas evolution rates of the samples were measured in 3.5 wt.% NaCl solution for 10 h at the room temperature. Samples were ground and polished, and hydrogen gas was collected using an inverted burette and funnel above the immersed samples.

Based on ASTM-G31-72 [22], the immersion tests were performed by immersing samples (10 mm × 10 mm × 2 mm) in 3.5 wt.% NaCl solutions for 24 h. Samples were grinded using progressively finer abrasive papers up to 2000 grits and then weighed. After immersion tests, the corrosion products were removed by immersing in boiling chromic acid (20 mg CrO₃ + 10 mg AgNO₃). Then samples were washed with deionised water, dried and reweighed to calculate weight loss. The morphologies of the corroded surfaces after weight loss were observed using JSM-5610LV SEM.

3. Results and discussion

3.1. Microstructural evolution
Figure 2 shows the microstructural evolution of AZ63 magnesium alloy after ARB process. The related average grain sizes are measured, as shown in figure 3. The average grain size of the original sheet is 30.46 μm and typical deformation twins are observed in the microstructure. After 1 ARB cycle, the grain is refined because that large strain imposed on the sheet, as shown in figure 2(b). The refinement of grains is not homogeneous initially, with fine grains of less than 10 μm as well as some coarse grains of greater than 40 μm. Since the slip systems of magnesium alloys are very limited, the grains with more favorable orientations are refined and deformed first, leaving islands of less deformed and coarser grains. This ‘bimodal’ microstructure phenomenon was also observed by Chang et al [23]. As the strain increases, the dislocation density and the stored energy inside the grain increases. When the energy stored in the materials increase to the activation energy of recrystallization, dynamic recrystallization will occur inside the sheet during ARB process. According to figures 2(b)–(e), the original coarse grains are surrounded by many fine recrystallized grains. After the subsequent ARB process, deformation spread to all the grains and the homogeneity of the microstructure is improved obviously. After 5 ARB cycle, the large grains are expensed by the increasing fraction of fine grains, and new recrystallized grains fill the entire volume of the samples. The average grain sizes of these recrystallized grains are reduced to 4.67 μm and some UFG with grain size lower than 1 μm are observed in figure 2(f).
Figure 4 shows the XRD pattern of the original sheet and the ARBed samples, it can be seen that all of the peaks corresponded to either magnesium or Mg17Al12. It identifies that the second phase particle is Mg17Al12. The AZ63 magnesium alloy exhibits a microstructure of primary magnesium grains with intermetallic Mg17Al12 dispersed in the matrix, which is consistent with the description of other reports \[8, 24\]. The peak intensity of the Mg17Al12 decreases with increasing the number of the ARB cycles. It indicates that the volume fraction of the Mg17Al12 phase is reduced.

TEM was used to investigate the microstructures of magnesium matrix and morphology evolution of the Mg17Al12 phase after ARB process. As shown in figure 5(a), the majority of dislocations are formed inside the grains and twins are observed at the initial period of deformation. As strain increases, the distribution of dislocations become heterogeneous and easily entangled together (figure 5(b)). These structural features make the ARBed samples have relatively higher deformation energy, which could lead to dynamic recrystallization. Further interaction between dislocations promotes the formation of cell structures (figure 5(c)). As the strain
increases, the cell structure gradually transforms into subgrains, which led to the reduction of the grain size. The close-up micrograph shows the morphology evolution of the Mg17Al12 phase after ARB process. The Mg17Al12 phase in the original sheet was in the form of lath. As shown in figures 5(e)–(f), the brittle lath Mg17Al12 phase breaks into round or short rod shape after ARB process. Small equiaxed Mg17Al12 phase with an average size of about 50 nm are formed. The distribution of the Mg17Al12 phase from different cycles of ARB process are shown in figure 6. The white particles in the figure represent Mg17Al12 phase. As shown in figure 6(a), the Mg17Al12 phase in the original sheet is coarse and not homogeneous. As the ARB cycles increases, the Mg17Al12 phase becomes finer and more dispersed, which improves the homogeneity of microstructure. Meanwhile, a solid solution effect will occur, the justification for the observed microstructural changes in this study can be partial dissolution of Mg17Al12 phase during solutionizing at 350 °C. As a consequence, the volume fraction of the Mg17Al12 phase is reduced, which is consistent with the XRD result.
3.2. Electrochemical experiments

OCP variations for AZ63 magnesium alloy from different cycles of ARB process are determined by immersing in 3.5 wt.% NaCl solution for 3600 s. As shown in figure 7, both samples show variations with time for OCPs. The OCP of the sample was initially $-1.60$ V. Eventually, the OCP of the sample was stabilized to $-1.564$ V.

After OCP was determined, EIS measurements were carried out to further clarify the extent and type of the corrosion. EIS diagram for samples are illustrated in figure 8. It is obvious that the shapes of the impedance diagram are identical. Each sample has a capacitive loop at high frequency and an inductive loop at low frequency, indicating the same corrosion mechanism. The high frequency loop is attributed to the dissolution of magnesium matrix and the formation of oxide film on the surface, while the low frequency loop is ascribed to the desorption of corrosion products and the reaction of $\text{Mg}^{2+}$ with $\text{H}_2\text{O}$ at the damaged areas of the corrosion products film [24, 25]. The diameter of the loops increases with increasing the number of the ARB cycle, indicating that the corrosion resistance is enhanced.

Figure 9 shows the potentiodynamic polarization curves of samples after different cycles of ARB process. The anode curve represents the anodic oxidation of the magnesium alloy, while the cathode curve represents the hydrogen produced by the reduction of water. Each anodic curve shows an inflection point, which is characterized by two different slopes. This indicates the existence of a kinetic barrier effect, which is caused by the deposition of $\text{MgO}$ and $\text{Mg(OH)_2}$ surface film and its subsequently dissolution at higher anodic potential.

![Figure 6. The SEM micrographs of AZ63 magnesium alloy from different cycles of ARB process: (a) original sheet, (b) cycle 1, (c) cycle 2, (d) cycle 3, (e) cycle 4, (e) cycle 5.](image)

![Figure 7. The OCP curves of AZ63 magnesium alloy from different cycles of ARB process.](image)
The overall shapes of the polarization curves are not changing significantly, indicating that ARB process only changes the corrosion rate, without changing the corrosion mechanism. To analyze the electrochemical polarization curves, corrosion parameters including corrosion current density ($I_{\text{corr}}$) and corrosion potential ($E_{\text{corr}}$) are obtained using the Tafel extrapolation technique. Table 2 show that $I_{\text{corr}}$ and $E_{\text{corr}}$ decreased with increasing the number of ARB cycles. It can be clearly seen that the $E_{\text{corr}}$ of the sample after 5 ARB cycles is approximately 78 mV lower than that of the original sheet. The $I_{\text{corr}}$ of the sample after 5 ARB cycle is lower than that of the original sheet by approximately an order of magnitude. The results reveal the corrosion rate decrease with increasing the number of ARB cycles.

![Figure 8. The electrochemical impedance spectroscopy diagram of AZ63 magnesium alloy from different cycles of ARB process.](image1)

![Figure 9. The polarization curves of AZ63 magnesium alloy from different cycles of ARB process.](image2)

**Table 2.** Calculated parameters for polarisation diagram of AZ63 magnesium alloy from different cycles of ARB process.

| Sample     | $I_{\text{corr}}$ (μA cm$^{-2}$) | $E_{\text{corr}}$ (V) |
|------------|----------------------------------|-----------------------|
| Original sheet | 84.6                            | 1.24                  |
| Cycle 1    | 77.4                            | 1.25                  |
| Cycle 2    | 65.6                            | 1.28                  |
| Cycle 3    | 43.9                            | 1.28                  |
| Cycle 4    | 17.3                            | 1.30                  |
| Cycle 5    | 9.30                            | 1.32                  |
3.3. Weight loss and hydrogen evolution rate

The generation of hydrogen gas is accompanied by the dissolution of the magnesium matrix and it could directly reflect the corrosion rate. Figure 10 presents a cross plot of the weight loss measurements and hydrogen evolution data. The hydrogen evolution rate of the original sheet reaches to about 8.93 ml·cm⁻²·h⁻¹, while the hydrogen evolution rate of the sample after 5 ARB cycle is the lowest value of 1.44 ml·cm⁻²·h⁻¹. The weight loss rate of the original sheet is almost six times than the weight loss of the sample after 5 ARB cycle. The direct correlation can be obtained from the overall magnesium corrosion reaction (1) and equation (2) that 1 mol hydrogen gas released corresponds to 1 mol Mg corroded. Therefore, the trends revealed in the hydrogen evolution data is equivalent to in the weight loss measurements. After ARB process, the weight loss and hydrogen evolution rate reduced significantly, indicating that the corrosion resistance of AZ63 magnesium alloy increased with increasing the number of ARB cycles.

\[
\text{Mg} + \text{H}^+ + \text{H}_2\text{O} = \text{Mg}^{2+} + \text{OH}^- + \text{H}_2 \quad (1)
\]

\[
\text{Hydrogen evolution rate} / \text{ml} \cdot \text{cm}^{-2} \cdot \text{h}^{-1} = 0.922 \times \left( \text{Weight loss rate} / \text{mg} \cdot \text{cm}^{-2} \cdot \text{h}^{-1} \right) \quad (2)
\]

3.4. Characterization of corrosion morphology

After corrosion products were removed, the corrosion morphology of the sample surfaces was observed using SEM. An obvious pitting corrosion phenomenon can be seen in figure 11. The porous oxide films MgO and Mg(OH)₂, which are usually formed on the surface of magnesium alloys, are prone to induce the pitting corrosion in non-oxidizing media containing chloride ions [8, 24]. The exposed magnesium matrix via the porous oxide films would dissolve preferentially, then the corrosion would extend to the adjacent matrix. The denser the oxide film formed on the surface, the better the corrosion resistance of the magnesium alloy. As shown figure 11(a), the surface of the original sheet shows severe damage and many deep corrosion pits are observed. In comparison, the size and depth of the corrosion pits of samples after ARB process are smaller than those of the original sheet. The results show that the original sheet corrode more seriously than the ARBed sheet, which is consistent with the weight loss and hydrogen evolution rate.

3.5. Corrosion mechanism

The corrosion behavior of AZ63 magnesium alloy depends on its microstructure. There is a large variation in the grain size and the second phase distribution of AZ63 magnesium alloy after ARB process, which results in different corrosion rates and corroded areas.

It has been recognized that grain sizes play a primary role in the corrosion behavior and fine grains will create more grain boundaries. Since grain boundary is more active situation than the grain, it is the place where corrosion preferentially initiated. However, uniform and denser passive oxide layers will be deposited on the sample surface, and the stability of the oxide film improved when the grains are refined and homogenized [27, 28]. During the corrosion process, grain boundary is blocked by the stable passive oxide layers, so it become not relative active site compared with the grain. As is seen from figures 2 and 3, significant decreases in the grain size is achieved through application of the ARB. Meanwhile, some UFG are formed in microstructures, the formation of UFG microstructures makes the passive layer grow more rapidly [19, 29]. Therefore, the corrosion resistance is improved by increasing the number of ARB cycles.
AZ63 magnesium alloy is a two-phase alloy whose second phase plays two functions during corrosion. Namely, it can act as a galvanic cathode or as a corrosion barrier. Mg17Al12 phase is relatively stable and has a lower corrosion rate in the 3.5 wt.% NaCl solution. However, the corrosion potential of Mg17Al12 phase is higher than that of the magnesium matrix, so it can also serve as the micro-galvanic cathode. The corrosion barrier effect and the micro-galvanic cathode effect have a competitive relationship during the corrosion process. If the Mg17Al12 phase particles are isolated distributed in the magnesium alloy and have a small volume fraction, it will primarily act as a galvanic cathode to accelerate corrosion. On the other hand, when the Mg17Al12 phase is in a continuous network form, it will mainly act as a corrosion barrier and hinder the corrosion propagation in the magnesium matrix [12]. As shown in figure 6, isolated Mg17Al12 phase distributed in the whole microstructure in the original sheet and ARBed samples. The distance between them is large, so corrosion cannot be effectively hindered by the Mg17Al12 phase. Due to the isolated distribution of the Mg17Al12 phase, it primarily acts as the galvanic cathode in the corrosion process. In addition to the spontaneous dissolution of magnesium matrix, the micro-galvanic effect can accelerate corrosion rate. The main factor for the galvanic corrosion rate is the anode-to-cathode surface ratios. During the ARB process, the coarse brittle Mg17Al12 phase will break and form a fine equiaxed shape. In addition, some Mg17Al12 phase will dissolve into the magnesium matrix after ARB cycles, leading to a decrease in the volume fraction of Mg17Al12 phase. The surface of cathode reduced, so the effect of the galvanic corrosion was decreased. After 5 ARB cycles, there are only a few fine Mg17Al12 particles in the microstructure, such that the smaller cathode area causes less galvanic corrosion to accelerate the corrosion rates of the AZ63 magnesium alloy. This is because these particles act as a small cathode connected to a large anode.

4. Conclusion

In this work, AZ63 magnesium alloy sheets were fabricated by ARB process at 350 °C up to 5 cycles. The microstructure and the corrosion behavior of the ARBed sheets were investigated. The following results were obtained:

1. After the ARB process, the grain is refined and homogenized, some UFG with grain size lower than 1 µm are formed. By increasing ARB cycles, the Mg17Al12 phase becomes finer and the volume fraction of Mg17Al12 phase is reduced.

2. The ARB process has a significant influence on the corrosion behavior of AZ63 magnesium alloy. The corrosion resistance of AZ63 magnesium alloy increases with increasing the number of ARB cycles.

3. Fine grain produces more passive oxide layers which act as a corrosion barrier. The Mg17Al12 phase acts primarily as a micro-galvanic cathode to accelerate corrosion because of its low fraction and isolated distribution. After ARB process, the effect of galvanic corrosion decreases due to the volume fraction of Mg17Al12 phase decreases.
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ORCID IDs

Tao Huang @ https://orcid.org/0000-0002-2237-4607

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