A novel approach to improve corrosion resistance of Mg alloy by co-extrusion

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Abstract

In this study, Mg/Al composite rod with a thin 6082 aluminum alloy coated AZ31 magnesium alloy was successfully prepared by co-extrusion. Microstructure and texture of the extruded Mg/Al rod were systematically studied. A comparative study about electrochemical behavior of the Mg core and the Al sleeve was also addressed. Our results show that, co-extrusion can greatly refine grains of Mg alloy, but does not change the texture component. The 6082 sleeve exhibits a much better corrosion resistance than the AZ31 core. This study provided a novel approach to improve corrosion resistance of Mg alloy. The Al sleeve served as a barrier to protect the Mg alloy from corrosion.

1. Introduction

Magnesium alloys are desirable candidates as structural materials due to their low density and high specific strength [1–7]. Unfortunately, Mg has a quite low standard electrode potential \( E_{\text{Mg}}^0 = -2.37 \text{ V} \) and the naturally formed oxide or hydroxide layer on Mg alloys substrate is loose and porous, which leads to a poor corrosion resistance [8, 9]. The poor corrosion resistance has become one of the main limitations for their applications. Some efforts have been proposed for improving the corrosion resistance of Mg alloys, such as alloying [10, 11], physical vapor deposition (PVD) [12, 13], electroplating [14], anodic oxidation [15], micro arc oxidation [16] and chemical vapor deposition (CVD) [17].

Bimetal composites that combine the advantages of two metals are beneficial to improve mechanical properties [18–23]. Various laminated composites were prepared by accumulative roll bonding (ARB), which can effectively refine grain size and improve mechanical properties [24–29]. Besides ARB processing, co-extrusion processing is widely used to prepare bimetal composite [30–34]. Generally, the constructions and fractions of each component in composite can be accurately regulated by co-extrusion.

Aluminum alloys also have low density, excellent performances as well as a desirable corrosion resistance [35]. The naturally formed Al oxide layer is compact and capable of self-healing, which can effectively improve the corrosion resistance of Al alloys [36]. Therefore, if Mg alloy product can wear a thin Al alloy coat, the corrosion resistance of the Mg products can be effectively improved without an obvious increase in weight. In this study, we provide a new type of surface treatment for extruded Mg alloy rod using plastic processing. A thin Al sleeve coated Mg alloy was prepared by co-extrusion. The thin Al sleeve served as a barrier to protect Mg alloy from corrosion. The influence of co-extrusion processing on the microstructure and texture of Mg/Al rod were systematically studied. A comparative study about electrochemical behavior of the Al sleeve and the Mg core was also addressed and discussed.
2. Materials and methods

2.1. Fabrication of Mg/Al bimetal rod

As-cast AZ31 Mg alloy and 6082 Al alloy were used for preparation of Mg/Al bimetal rod. The as-received 6082 was machined into hollow cup with an inner diameter of 62 mm and an outer diameter of 80 mm. A Mg cylinder (62 mm in diameter) was cut and filled into Al hollow cup as demonstrated in figure 1. Mg/Al billet was annealed at 450 °C for 1.5 h and immediately extruded at 450 °C using an extrusion ratio 79:1 and an extrusion rate of 0.8 m min⁻¹. The final Mg/Al rod was a diameter of 9 mm and a core about 7 mm in diameter. For a comparison, a monolithic AZ31 extruded rod was also prepared using the same extrusion conditions. The final AZ31 extruded rod was a diameter of 9 mm.

2.2. Microstructure and electrochemical analyses

Microstructure of Mg/Al rod was examined using a scanning electron microscope (SEM) and electron back scattering diffraction (EBSD) mapping. Elemental distribution near the interface was identified using energy-dispersive spectroscopy (EDS) mapping. Samples for EBSD mapping were mechanically ground and electrochemically polished in AC2 electrolyte for Mg alloy and perchloric acid solution for Al alloy. EBSD mapping were conducted on a SEM (FEI Nova 400) equipped with a HKL-EBSD system. The step size for EBSD mapping was 0.4 μm. All EBSD date were analyzed through Channel 5 software.

Electrochemical behavior of the Al sleeve and the Mg rod in a 3% NaCl solution was investigated using a potential dynamic polarization test and an electrochemical impedance spectroscopy (EIS) analysis. The specimens for electrochemical tests of Al sleeve were cut from Mg/Al rod. The ends of specimens were sealed using resins to avoid the contact between Mg core and electrolyte. Samples for electrochemical tests of Mg core were cut from the monolithic extruded Mg rod. A scanning rate of 0.5 mV s⁻¹ was used in potentiodynamic polarization test and the impedance data were recorded from 100 kHz to 10 mHz using a 10 mV sinusoidal perturbing signal at open circuit potential. EIS data recording started after the samples was exposed to the test solution for 0.5 h. Equivalent circuits (EC) that not only match the physical structure of electrode system, but also generate similar impedance responses were used to analyze the EIS spectra.

3. Results and discussion

3.1. Microstructure and texture

Cross sectional SEM micrographs acquired from a region near the interface of Mg/Al rod were present in figure 2. The Al sleeve and Mg rod exhibit a good bonding condition. A uniform diffusion layer with a thickness of about 5 μm is discerned. It is also noticed that this diffusion layer contains two sublayers (figure 2(b)). The EDX mapping results further show that the sublayer adjacent to Al sleeve contains a higher Al content and a lower Mg content, while a lower Al content and a higher Mg content exist in the sublayer close to Mg core (figure 2(c)). This reaction layer often appears in Mg/Al bimetallic constructions [30, 32, 37–39]. It has been found that the sublayer close to Mg contains much Mg₁₇Al₁₂ and Mg₂Al₃ generally forms in the sublayer adjacent to Al [32].

Microstructure and texture of the monolithic Mg rod, Mg core and Al sleeve in Mg/Al rod were shown in figures 3–5, respectively. Grain size distribution of the Mg rod, Mg core and Al sleeve were also given in figure 6. As seen in figures 3 and 6(a), the average grain size of Mg rod is about 20 μm. The Mg rod exhibits a typical extrusion texture with basal pole largely perpendicular to the extrusion direction (ED) [40]. Microstructure and texture of Mg core in Mg/Al rod were presented in figure 4. The average grain size of Mg core is about 3 μm (figure 6(b)). Obviously, the grain size of Mg alloys can be greatly refined by Mg/Al composite structure. It should be pointed out that the Mg core also exhibits a typical extrusion texture (figure 4(b)). Therefore, the
Figure 2. Cross sectional SEM micrographs of Mg/Al rod: (a) low magnification, (b) high magnification, (c) EDS mapping.

Figure 3. (a) Inverse pole figure, (b) pole figure and (c) misorientation angle distribution of the monolithic extruded Mg rod. ED and RD refer to the extrusion direction and radial direction, respectively.

Figure 4. (a) Inverse pole figure, (b) pole figure and (c) misorientation angle distribution of Mg core in Mg/Al rod. ED and RD refer to the extrusion direction and radial direction, respectively.
co-extrusion processing hardly changes the texture component of Mg alloy. Microstructure and texture of Al sleeve were also given in figure 5. The Al sleeve has a fully recrystallized grain structure containing an average grain size of about 2 \( \mu \text{m} \) (figure 6(c)). The Al sleeve exhibits a typical double fiber texture, with \( \langle 100 \rangle \) and \( \langle 111 \rangle \) parallel to the ED.

As seen in figures 3 and 4, the EBSD results show that the co-extrusion can greatly refine the grain size of Mg alloys. Generally, a strong friction shear exists at the interface during co-extrusion\(^{41}\). A higher shear strain is often beneficial for grain refinement during dynamic recrystallization\(^{42}\). Therefore, the friction shear at interface contributes to refine grains of Mg alloy. In our previous study, it was found that the difference in thermal conductivity between sleeve and core could greatly affect the microstructure of composite rods\(^\text{43, 44}\). In this study, the thermal conductivity of 6082 Al alloy (200 W m\(^{-1}\) K\(^{-1}\)) are about two times higher than that of AZ31 Mg alloy (70 W m\(^{-1}\) K\(^{-1}\))\(^\text{45, 46}\). Therefore, the heating during extrusion process is largely conducted by Al sleeve, which contributes to refine grains of Mg core. In addition, both the Mg rod and Mg core in Mg/Al rod exhibit a typical extrusion texture with basal pole largely perpendicular to the ED (figures 3 and 4). The results show that co-extrusion does not change the texture types of Mg alloy, which is mostly caused by the shear stress is generally axisymmetrical around the ED. The similar results were reported in our recent publications\(^\text{30–32}\).

### 3.2. Electrochemical behavior

Potentiodynamic polarization curves of the Mg core and the Al sleeve in a 3% NaCl solution were given in figure 7. Corrosion potential and corrosion current density derived from the polarization curves are listed in table 1. Corrosion potential of Al sleeve (about \(-0.654 \text{ V}\)) is about 0.85 V more positive than that of Mg core (about \(-1.503 \text{ V}\)). Al sleeve exhibits a corrosion current density of about 2 orders of magnitude lower than
Mg core. The lower corrosion current density value indicated that the Al sleeve exhibited a much better corrosion resistance than the Mg core [47].

Electrochemical behavior of Mg core and Al sleeve is further analyzed using EIS and the results are given in figure 8. The Nyquist plot of Mg core is composed of two loops, one high frequency capacitive loop and another one low frequency inductive loop. An enlarged capacitive loop at high frequency region and a Warburg impedance at low frequency region are seen in the spectrum of Al sleeve. The high frequency behavior of EIS was related to electrolyte penetration, including water uptake and electrolyte intrusion [48]. Usually, the low frequency region of EIS has an electrode control process combined with the contribution from localized defects to overall impedance [49].

The EIS plot for the Mg core and Al sleeve is further analyzed using equivalent circuit (EC). EC that considers both the physical structure of electrode system and the impedance response are proposed and given in figure 9. Rs was the solution resistance. Rct and CPEAl described the high frequency capacitive loop. Rct was the charge transfer resistance and CPEAl represented the electric double layer capacity. Rs and L described the low frequency inductive loop. W is Warburg impedance component that is linked up with the concentration and diffusion related processes [47]. The fitted parameters of EC components are listed in table 2. The charge transfer resistance (Rct) of Al sleeve (1.49E4 Ω·cm²) is about 2 orders of magnitude higher than that of Mg core (126 Ω·cm²), indicating a much better corrosion resistance.

Various adhesive and corrosion resistant coatings, e.g. anodic coatings, chemical conversion coatings and ceramic coatings prepared by physical vapor deposition, have been successfully fabricated on Mg alloy to improve corrosion resistance [12, 13]. However, the coatings on Mg alloy are vulnerable to pitting corrosion, as these corrosion pits generally do not possess self-healing ability [9]. Once corrosion pits appear, the whole protective coating of Mg alloy will result in a fast failure. Although micro arc oxidation can greatly improve the corrosion resistance of Mg alloy, a high cost greatly limits its range of applications [16]. Unlike Mg alloy, the naturally formed Al oxide layer on Al alloy is compact and capable of self-healing [36]. Thus, Al alloy product generally has much better corrosion performance than Mg alloy. The electrochemical tests in the present study strongly show that the as-used Al sleeve has much better corrosion resistance than Mg core. In this study, Mg

Table 1. Electrochemical properties of Mg core and Al sleeve in a 3% NaCl solution.

| Sample     | Corrosion potential/V | Corrosion current density/(A·cm⁻²) |
|------------|-----------------------|-----------------------------------|
| Mg core    | −1.503 ± 0.01         | (1.93 ± 0.03) × 10⁻⁴             |
| Al sleeve  | −0.654 ± 0.02         | (1.81 ± 0.05) × 10⁻⁶             |

Figure 7. Potentiodynamic polarization curves of Mg core and Al sleeve.
AZ31 rod with a thin Al 6082 sleeve was prepared by co-extrusion. If the thin Al sleeve totally coated Mg alloy by co-extrusion, the corrosion resistance of Mg/Al rod is equivalent to the Al sleeve, which can greatly improve the corrosion resistance of Mg alloy. Therefore, this study provides a novel approach to improve corrosion resistance of Mg alloy by co-extrusion.

4. Conclusions

In this study, Mg/Al rod with a thin 6082 Al alloy coated AZ31 Mg alloy was successfully prepared by co-extrusion. Microstructure and texture of the extruded Mg/Al rod were systematically studied. A comparative study about electrochemical behavior of the Mg core and the Al sleeve was also addressed. This study provided a novel approach to improve corrosion resistance of Mg alloy. The conclusions are as follows:

(1) Co-extrusion can greatly refine grains of Mg alloy, but does not change the typical extrusion texture.
(2) The Al 6082 sleeve exhibits a much better corrosion resistance than the Mg AZ31 core.

Table 2. Parameters of EC components derived from EIS fitting.

| Sample  | $R_s/\Omega \cdot cm^2$ | $Y_{dl}/\Omega^{-1} \cdot cm^{-2} \cdot s^n$ | $n_{al}$ | $R_c/\Omega \cdot cm^2$ | $L/H \cdot cm^2$ | $R_L/\Omega \cdot cm^2$ |
|---------|------------------------|---------------------------------|----------|------------------------|-----------------|------------------------|
| Mg core | 3.93 ± 0.5             | (4.78 ± 0.2)E-5                | 0.89 ± 0.02 | 126 ± 11              | 71.12 ± 5.21  | 137.60 ± 15.3          |
| Al sleeve | 5.25 ± 0.6             | (5.73 ± 0.4)E-6               | 0.92 ± 0.03 | (1.49 ± 0.1)E4        | —               | —                      |

Figure 8. EIS spectra of (a) Mg core and (b) Al sleeve in a 3% NaCl solution.

Figure 9. Equivalent circuits for analysis of EIS spectra: (a) Al sleeve and (b) Mg core.

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