



Lower temperature fabrication of continuous intermetallic coatings on AZ91D magnesium alloy in molten salts

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ARTICLE INFO

Article history:

Received 5 April 2010

Received in revised form 17 May 2010

Accepted 25 May 2010

Available online 4 June 2010

Keywords:

Magnesium alloy

Molten salt

Diffusion

Corrosion

Hardness

ABSTRACT

A continuous Mg–Al intermetallic coating was fabricated on an AZ91D Mg alloy in molten salts in the lower temperature range from 300 °C up to 400 °C. The coating consists of a single $Mg_{17}Al_{12}$ intermetallic layer or both $Mg_{17}Al_{12}$ and Mg_2Al_3 intermetallic layers, depending on the treatment temperature. In contrast to the conventional powder pack cementation process, active Al atoms can be formed at a lower temperature in molten salts, which contributes to the lower temperature formation of continuous intermetallic layers. The intermetallic layer markedly improves the hardness as well as the corrosion resistance of the AZ91D Mg alloy. Furthermore, the continuous intermetallic layer exhibits passive behaviour in 3.5 wt.% NaCl solution, which is attributed to the homogeneously distributed intermetallic phases.

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1. Introduction

Mg alloys are advanced light structural and functional materials being increasingly used in the automotive, aerospace, electronic and energy industries, owing to their high strength-to-weight ratio, high thermal conductivity and electrical conductivity and good recycling with low energy consumption. However, the low corrosion resistance of Mg alloys is currently limiting their further applications [1]. Various surface modification techniques have been developed to improve the poor corrosion resistance of Mg alloys [2–6]. Of these techniques, recently developed surface alloying of Mg alloys by diffusion coating is of great interest because of the following potential advantages. Firstly, the electrical conductivity of the coated Mg alloy can be maintained. Moreover, the diffusion coating has high adhesion strength since there is a strong metallurgical bond between the coating and substrate [7]. In addition, the alloyed layer comprises of intermetallic compounds, which can both improve the corrosion resistance [8] and the mechanical properties [9].

Based on the above reasons, considerable research has been done trying to achieve diffusion coatings on Mg alloys [7,10–13]. For example, Shigematsu et al. [10] have obtained an Al-enriched diffusion coating by covering Mg alloys with Al powders at 450 °C. Al, Zn powder pack cementation and vacuum Al powder pack cementation have also been applied to obtain a diffusion coating reported

by Ma et al. [7] and Liu et al. [12,13], respectively. Up to date, most of the work has used conventional powder pack cementation process and has to be carried out at high temperatures (near or even above the Mg–Al eutectic reaction temperature of 437 °C). However, such high temperatures may lead to surface melting and cracking, which will limit its applications for practical cases. Therefore, the major challenge for diffusion-alloyed coating of Mg alloys is to lower the treatment temperature in order to avoid the negative effects during the coating process [14].

Previous work from our group [15] has prepared intermetallic coatings on Mg alloys at a lower temperature of 420 °C through adding $ZnCl_2$ as activator; however, the formed intermetallic coating is not continuous and does not exhibit passive behaviour in aggressive NaCl solutions. Recently, Zhang et al. [16] have successfully lowered the diffusion coating temperature of AZ91D alloy to 380 °C by surface nanocrystallization of the substrate by surface mechanical attrition treatment (SMAT) first. Unfortunately, current SMAT techniques are not suitable for components with complex shapes.

Thus, the present work aims to develop a novel and simple process to prepare continuous Mg–Al intermetallic coatings with passive behaviour on AZ91D substrate at lower temperatures, without the need to pretreat the substrate specially. In this work, diffusion coating process of the AZ91D Mg alloy in a new system, i.e., molten salts containing aluminum ions, was studied. The hardness and the corrosion resistance of the coating were investigated by nanoindentation test and electrochemical measurements, respectively.

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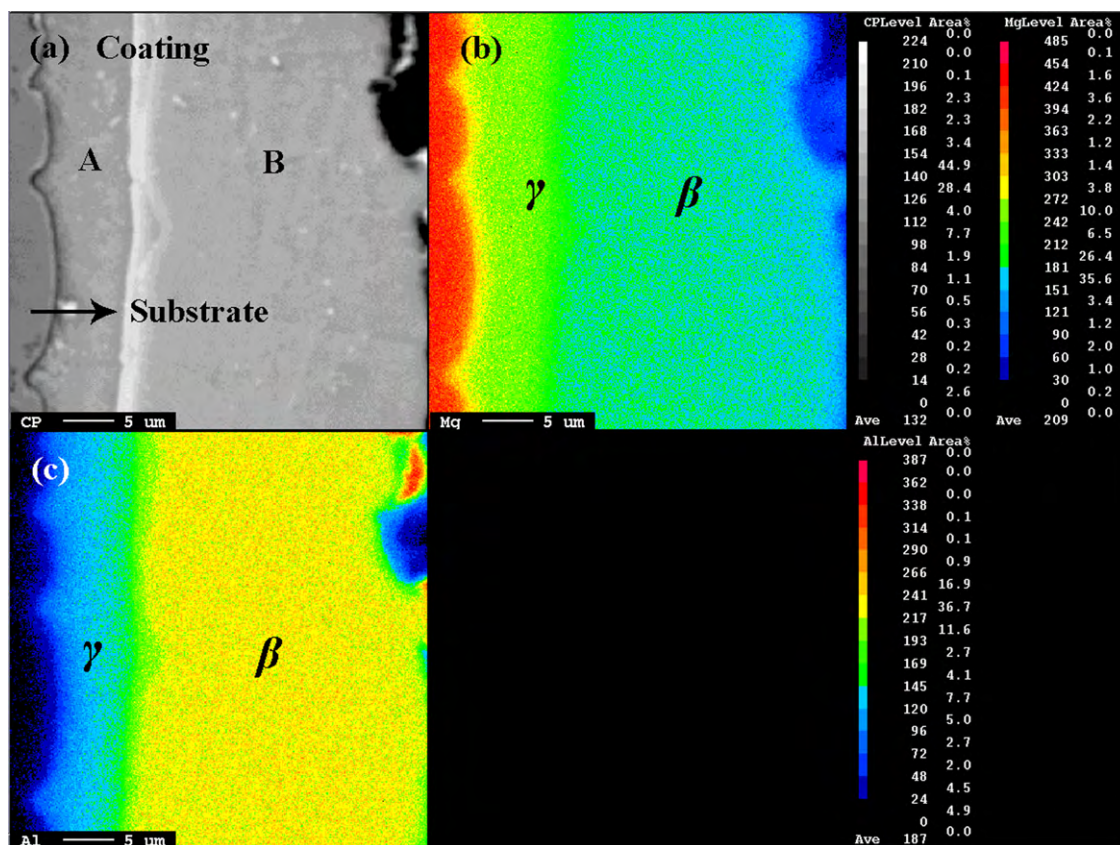


Fig. 1. Cross-sectional SEM micrographs of the (a) intermetallic coating on the AZ91D Mg alloy, EPMA-determined distribution of (b) Mg and (c) Al.

2. Experimental

An as-received AZ91D Mg alloy ingot was cut into 20 mm diameter \times 10 mm long specimens and polished up to 1000 grit SiC paper. The salt mixture containing 50% NaCl and 50% AlCl₃ (molar ratio) was selected for this study. The cleaned AZ91D specimens were put in the mixed salts in a ceramic container. An electric resistance furnace that can automatically control the temperature was employed to achieve the diffusion coating process, which was carried out in the temperature from 300 °C to 400 °C. During the whole process, protective pure argon gas (99.999%) was used to ensure the non-oxidation of the specimens.

The morphology and composition of the coated specimens were analyzed by a FEI SIRION-200 scanning electron microscopy (SEM), an X-ray energy dispersive spectrum (EDS) and a JSM-6700F electron probe microanalyzer (EPMA). Nanoindentation measurements were performed on the intermetallic layers and the substrate using a Hysitron Triboindenter. The maximum indentation load was 1000 μ N. There was a stabilized time of 5 s between the loading and unloading stages.

The corrosion property of the specimens was investigated using a PARSTAT 2273 potentiostat. Potentiodynamic polarization measurements were conducted in a deaerated 3.5 wt.% NaCl solution at 25 °C. A classic three-electrode cell was used, with the test specimen as the working electrode, a platinum plate as the counter electrode, and a saturated calomel electrode (SCE) as the reference electrode. A polarization scan was started from a cathodic potential at a sweep rate of 0.33 mV/s and stopped when the anodic current reached 5 mA/cm².

3. Results and discussion

Fig. 1 shows a typical SEM image of the cross-section of an AZ91D specimen which is treated in molten salts at 400 °C for 6 h. Fig. 1(b) and (c) shows the corresponding distributions of Mg and Al, respectively obtained by EPMA mapping, and the right scale bars indicate the relative intensities of Mg and Al. It is seen that an obvious coating is formed on the surface of the substrate at a temperature that is much lower than the conventional powder pack cementation temperature. Three distinct zones can be observed, i.e., the inner layer of the coating (region A), outer layer of the coating (region B) and

the substrate. Elemental mapping shows that Mg is enriched in the inner layer than the outer layer (Fig. 1(b)), while Al concentration in the outer layer is much higher than that in the inner layer (Fig. 1(c)). Furthermore, Fig. 1(b) and (c) shows no significant variations in the elemental concentrations at the same distance from the surface, indicating that Al and Mg are homogeneously distributed in the same depth of the layer. It is worth mentioning that most previously reported diffusion coatings are featured with a higher proportion of intermetallic phase than exists in the Mg matrix; few of them can prepare continuous intermetallic coatings. The difference will be further discussed later.

Fig. 2(a)–(c) shows typical SEM images of the cross-section of AZ91D specimens, which are treated in molten salts in the temperature range from 300 °C up to 400 °C. In addition, EDS elements line scanning along the yellow line in the cross-section of the treated AZ91D specimens are also given in Fig. 2. It is seen that the microstructure of the diffusion coating varies with the treatment temperature. An obvious coating is formed on the surface of the substrate at the temperature of 300 °C (Fig. 2(a)). As shown in Fig. 2(b), after treatment at 350 °C, the total thickness of the coating increases and two subzones can be observed in the coating, i.e., an inner dark grey layer (marked as “A”) and an outer light grey-coloured layer (marked as “B”). This suggests that the coating is composed of different phases, which will be clearly illustrated later by quantitative EDS analysis. When the treatment temperature increases to 400 °C, the thickness of the coating keeps increasing and it is interesting to note that the outer layer of the coating grows faster than the inner layer of the coating (Fig. 2(c)). Besides, EDS elements line scanning spectrum show that the concentration of Al decreases while Mg content increases with the increasing distance from the treated surface of the specimens (Fig. 2(a)–(c)). And the concentration of Mg and Al keeps constant in the substrate. It is

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