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Fabrication and Microstructure of Layers Containing Intermetallic Phases on Magnesium

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Abstract

Al- and Al/Zn-enriched layers containing intermetallic phases were deposited on the Mg substrate by heating the Mg specimens in contact with the powdered materials in a vacuum furnace. The Al-enriched surface layers were produced using Al powder, whereas the Al/Zn-enriched layers were obtained from an 80 wt.% Al + 20 wt.% Zn powder mixture. The microstructure and composition of the layers were analyzed by optical microscopy, scanning electron microscopy and X-ray diffraction. The results showed that the Al-enriched layer comprised an $Mg_{17}Al_{12}$ intermetallic phase and a solid solution of Al in Mg. The layer obtained from the Al+Zn powder mixture was composed of Mg-Al-Zn intermetalic phases and a solid solution of Al and Zn in Mg. Adding 20% of Zn into the Al powder resulted in the formation of a considerably thicker layer. Moreover, the hardness of the surface layers was much higher than that of the Mg substrate.

Keywords: Heat treatment, Magnesium, Al-enriched surface layers, Intermetallic phases, Hardness

1. Introduction

The application of magnesium and its alloys to produce automobile, computer and aerospace components, mobile phones, sporting goods, handheld tools and household equipment has increased steadily in recent years. Magnesium has a number of advantageous properties including high strength to weight ratio, high dimensional stability, high thermal conductivity, good machinability and it is also easy to recycle. However, for many applications, magnesium and its alloys have insufficient corrosion and wear resistance due to their high reactivity and low hardness (HV 60-80) [1]. There are various methods of modification that may be used to improve the surface properties of magnesium and its alloys which have their own advantages and disadvantages. Gray and Luan [2] provide a critical review of the technologies, applied to improve the corrosion and wear resistance of magnesium-based substrates. Of all the available surface treatments, the fabrication of Al-enriched surface layers containing intermetallic phases seems to be a promising surface treatment method as it contributes to an increase in wear and corrosion resistance. These layers also do not significantly increase the density of the material. Al-enriched layers can be deposited on magnesium and its alloys using different methods, i.e. laser processing [3-4], physical vapor deposition (PVD) [5], diffusion treatment in molten salts [6-8], diffusion aluminizing treatment [9-12], ion implantation [13], magnetron sputtering deposition combined with vacuum annealing [14], post-cold spray heat treatmebt [15], electrodeposition [16].

The objective of the research project was to examine the microstructure and hardness of Al- and Al/Zn-enriched surface layers containing intermetallic phases fabricated by heating Mg specimens in contact with Al or Al+Zn powder mixture in vacuum furnace.

2. Experimental procedure

Specimens with dimensions of 40 mm x 20 mm x 10 mm were sectioned from a pure magnesium ingot (99.9 % Mg) . The surfaces of the specimens were ground using 600 grit SiC paper, cleaned with ethanol and then dried in air. The Al powder used to produce the Al-enriched layers had particles with a diameter of 2-15 μm . The particles of the Zn powder were within the range of 50 μm to 120 μm. To produce Al-enriched layer Al powder was mixed with glicerol to obtain dense paste, which ensures better contact with the substrate. The specimens painted with the paste were embedded in a dry Al powder in a steel container. Then, the powder was pressed down, the container was closed and placed in a vacuum furnace. The specimens were heated up from room temperature to 450 ^{o}C for 30 min, kept at that temperature for 40 min, and cooled down with the furnace to 150 ^{o}C for 2 h, and finally taken out to the air. The Al/Zn-enriched layers were produced from 80 wt.% Al + 20 wt.% Zn powder mixture using the same preparation and heat treatment procedures. After the heat treatment the specimens were prepared for metallographic observations using a STRUERS automatic polishing machine. Polishing was performed using colloidal silica. The structure of the layers was observed with a Nikon ECLIPSE MA 200 optical microscope and a JEOL JSM-5400 scanning electron microscope. The specimens for microscopic observation were not etched. A chemical composition analysis was carried out using an X-ray energy dispersive spectrometer (EDS) attached to the SEM. The measurements of microhardness were conducted using a MATSUZAWA MMT Vickers hardness tester at a load of 100 g.

3. Results and discussion

3.1. Al-enriched layers on Mg

The microstructure of a layer obtained by heat treatment of Mg specimen in contact with Al powder is shown in Fig.1. The layer 80÷150 μm in thickness is compact and covers the whole Mg surface. No cracks or pores have been found in the layer or at the layer-substrate interface. A layer comprises two phases. Figure 2 shows the layer microstructure and profiles of Mg and Al concentration along the index line. EDS quantitative analysis was performed at points marked in the Fig.2. The results of the chemical composition were summarized in Table 1. The Mg and Al contents of the dark phase (area 1 in Fig.2) indicates a solid solution of Al in Mg according to the Mg-Al diagram. For the bright phase (area 2 in Fig.2) the Mg:Al ratio is not much different from $Mg_{17}Al_{12}$ intermetallic compound. These results show that the two-phase structure of the layer is the eutectic mixture containing $Mg_{17}Al_{12}$ intermetallic phase and the solid solution of Al in Mg. The zone of the solid solution of Al in Mg (area 3 in Fig.2) with a thickness of 20÷30 μm was formed as a transition region from Mg substrate to the two-phase structure.

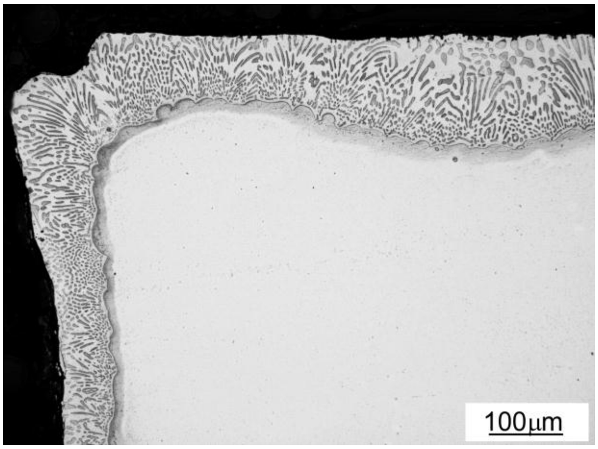

Fig. 1. A corner of the Mg specimen with Al-enriched layer

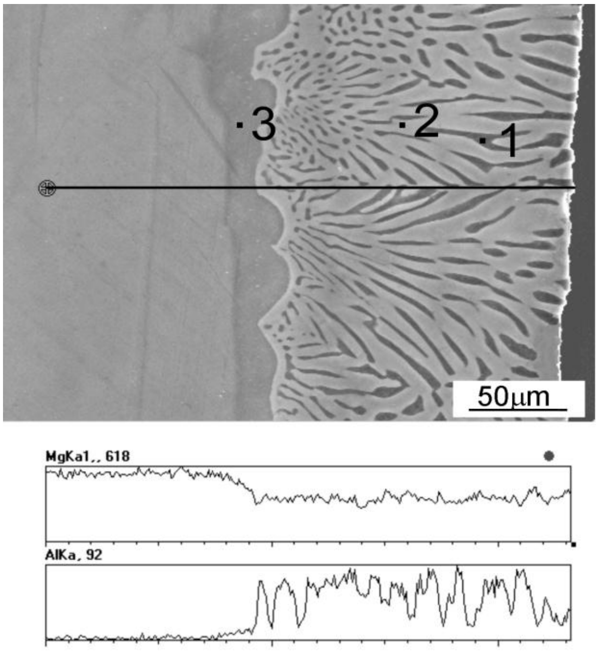

Fig. 2. SEM image of Mg specimen after heat treatment in contact with Al powder and the element distribution along the index line

Table 1.
EDX results of the Al-enriched layer corresponding to the points marked in Fig. 2

Point	Mg (at.%)	Al (at.%)
1	86.54	13.46
2	63.63	36.37
3	91.85	8.15

Figure 3 presents indentations in the Al-enriched layer after a Vickers hardness test. The microhardness of two-phase layer was 183-201 HV. The microhardness of the Mg substrate was also measured and it ranged 35-37 HV. The results show that the microhardness of the Al-enriched layer is about five times higher than that of the Mg substrate.

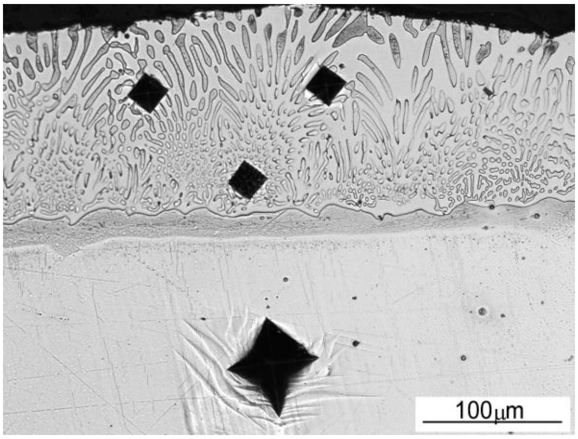

Fig. 3. Microhardness measurement indentations in the Al-enriched layer and the Mg substrate

Based on the above experimental results, we can conclude that the process of formation of the Al-enriched layer proceeds as follows. During the heat treatment process diffusion occurs at the interface between the Mg substrate and the Al particles and leads to the formation of the $Mg_{17}Al_{12}$ phase. In this experiment, the annealing temperature (450 ^oC) was higher than the melting point of $Mg_{17}Al_{12}$ phase. This phase begins to melt and the resulting liquid layer covers the entire surface of the Mg specimen. After cooling the Al-enriched layer forms on the substrate surface.

3.2. Al/Zn-enriched layers on Mg

Figure 4 shows a cross-section of the layer on Mg after heat treatment in contact with the 80 wt.% Al + 20 wt.% Zn powder mixture and distribution of Mg, Al and Zn along the index line. The microstructure of the layer is dense and defect-free. The thickness of the layer is 150÷200 µm. Table 2 shows the EDS analysis results corresponding to point depicted in Fig.4. It can be seen that the microstructure of the layer consists of large grains of the grey phase (area 1 in Fig.4), large grains of the dark phase (area 2 in Fig.4) and areas of fine grained eutectic which is composed of a light phase (area 3 in Fig.4) and a dark phase. EDS analysis reveals that the chemical composition of the grey phase is close to the $Mg_{17}Al_{12}$ phase, in which some of Al atoms are replaced by Zn atoms. This phase is defined in the literature as $Mg_{17}(Al,Zn)_{12}$ [17]. The chemical composition of the dark phase indicates a solid solution Al and Zn in Mg. The Zn content of the light phase is much higher than that of the grey and dark ones and the composition of this phase based on the Mg-Al-Zn phase diagram is similar to the ternary φ phase. According to the results reported by Donnadieu et al. [18], the φ phase has the nominal formula $Mg_5Al_2Zn_2$ or $Mg_6(Al,Zn)_5$ with the composition variation of 53-55 at.% Mg, 18-29 at.% Al and 17-28 at.% Zn. A zone of solid solution of Al and Zn in Mg with a thickness of 20-30 µm was formed between the Al/Zn-enriched layer and the substrate

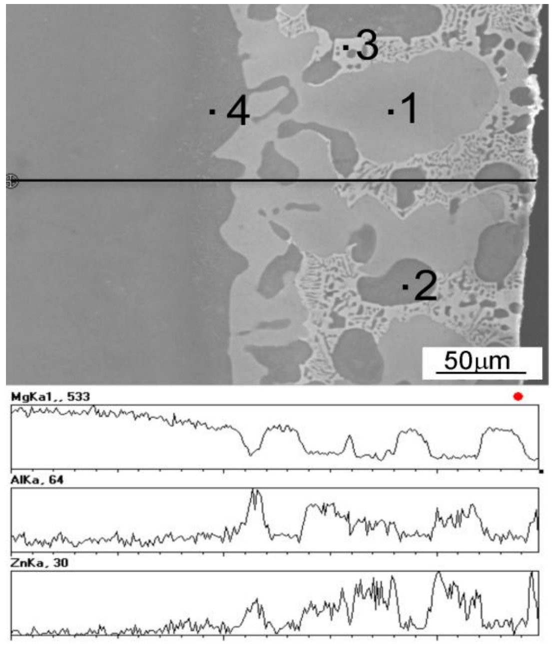

Fig. 4. SEM image of the Mg specimen after heat treatment in contact with the Al+20% Zn powder mixture and the element distribution along the index line

Table 2.
EDS results of the Al/Zn-enriched layer corresponding to the points marked in Fig. 4

Point	Mg (at.%)	Al (at.%)	Zn (at.%)
1	61.77	29.5	8.73
2	91.14	6.51	2.35
3	54.6	22.7	22.7
4	91.37	5.98	2.65

The microhardness of the Al/Zn-enriched layer changes from 80 HV for the large grains of the solid solution Al and Zn in Mg to 178 HV for the fine eutectic structure and 210 HV for the large grains of the $Mg_{17}(Al,Zn)_{12}$ phase. Cracks were observed near the edges of the indentations in $Mg_{17}(Al,Zn)_{12}$.

To summarize, the Al/Zn-enriched layers containging Mg-Al-Zn intermetallic phases can be produced by heating the Mg specimens in contact with an 80 wt.% Al + 20 wt.% Zn powder mixture. A comparative analysis of the Al- and Al/Zn-enriched layers shows that adding 20% of Zn to the powder mixture leads to the formation during heat treatment process (450^oC) intermetallic phases which have a melting point lower than $Mg_{17}Al_{12}$ phase. As a results, under the same heat treatment parameters the thicker Al/Zn-enriched layer forms on the Mg substrate. The structure of this layer is non-homogenous.

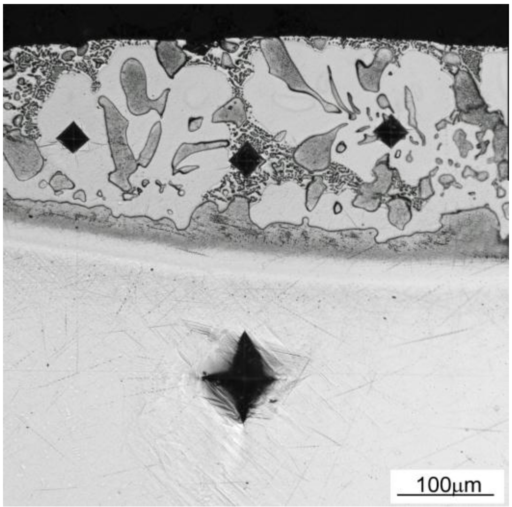

Fig. 5. Microhardness measurement indentations in Al/Zn-enriched layer and Mg substrate

4. Conclusions

1. An Al-enriched layer with a thickness of 80÷150 μm can be formed on Mg surface by heating Mg specimen in contact with Al powder at 450^{o}C for 40 min. The layer is composed of $Mg_{17}Al_{12}$ intermetallic phases and a solid solution of Al in Mg.
2. By adding Zn to the powder mixture it is possible to obtain Al/Zn-enriched layer on the Mg surface. This layer has a thickness 150-200 μm and contains Mg-Al-Zn intermetallic phases and a solid solution Al and Zn in Mg.
3. The microhardness of the Al- and Al/Zn-enriched layers is much higher than that of the Mg substrate. The high hardness of the surface conditions an improvement in wear resistance.

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