Effects of Pb on microstructure, mechanical properties and corrosion resistance of as-cast Mg97Zn1Y2 alloys

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Abstract: In this present work, Pb was applied in the Mg97Zn1Y2 alloy to improve its microstructure and properties, using conventional casting methods. The microstructure and properties of the Mg97Zn1Y2 alloy and Mg97Zn1Y2-xPb(x=0.6wt.%, 1.2wt.% and 1.8wt.%) alloys were observed by optical microscopy, scanning electron microscopy method and analyzed by X-ray diffraction, hardness and strength measurement and electrochemical testing. After adding Pb to the Mg97Zn1Y2 alloy, a new particle phase Mg₂Pb was identified along the grain boundaries, and dendrites were refined. In addition, the hardness of Mg97Zn1Y2-xPb(x=0.6wt.%, 1.2wt.% and 1.8wt.%) alloys was higher than that of the Mg97Zn1Y2 alloy; with the increase of Pb content, the hardness of the alloy increased first and then decreased, followed by a final slight increase, and reached a maximum of 89.1 HV when the Pb content was 0.6wt.%. The strength of the alloy increased first and then decreased as the Pb content increased. Moreover, adding a small amount of Pb to the alloy can effectively inhibit corrosion, and Mg97Zn1Y2-0.6wt.%Pb exhibits the best corrosion resistance ability.

Key words: Mg-Zn-Y-Pb alloy; microstructure; LPSO phase; mechanical properties; corrosion resistance

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Engineered magnesium alloys with low density, good electromagnetic shielding, and excellent damping suspension properties are widely used in the sports equipment, aerospace, and electronic equipment industries^[1-2]. Over the years, Mg-Y-Zn alloys have fascinated scientist and technicians due to a long-period stacking ordered (LPSO) structure^[3-4]. Studies show that the strengthening effect of LPSO phase is dominant over that from the grain refinement and texture^[5-6]. The formation of the LPSO phase requires a quantitative relationship between the Y and Zn atoms^[7-9]. The hexagonal close-packed structure of Mg can produce stacking faults, and the solid solution atoms (Zn and Y) filled into the staggered layer can result in the lattice distortion. At the same time, the lattice distortion provides an additional stress field that accelerates atomic diffusion and promotes nucleation and growth of LPSO structures^[7-9].

The high strength of Mg97Zn1Y2 alloy is derived from the refinement of grains and the enhancement of fine LPSO phase^[10], and the strengthening effect of LPSO phase with different morphologies is different^[11-13]. Therefore, other added elements which influence the formation of LPSO, can affect the properties of the alloy.

Pb has a high solid solubility in Mg, which can inhibit the diffusion of Mg atoms in solid solution and improve the morphology and distribution of the secondary phase^[14]. Zhang^[15] found that when Pb was added in the AZ91 alloy, the substitutional solid solution was formed, which causes the lattice distortion of Mg and impedes the migration motion of atoms such as Mg and Al, so that the growth of β -Mg₁₇Al₁₂ phase was hindered and the microstructure of AZ91 alloy was refined. Furthermore, the mechanical properties of the alloys have been improved by adding a small amount of Pb into the alloy^[16]. Some work^[17-20] has been reported on the effect of minor Pb addition to AZ91 alloy, but the effect of Pb addition on the mechanical and corrosion behaviour of Mg-Y-Zn alloy has not been reported. In this work, the effects of Pb on the microstructure, mechanical properties, and corrosion resistance of as-cast Mg97Zn1Y2 alloy were investigated.

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1 Experimental procedures

Pure Mg, pure Zn, and Mg-Y and Mg-Pb master alloys were used in these experiments. The preparation process of Mg97Zn1Y2 alloy and Mg97Zn1Y2-xPb ($x=0.6\text{wt.}\%$, $1.2\text{wt.}\%$ and $1.8\text{wt.}\%$) alloys comprises the following steps: an empty clean crucible was placed in the furnace and heated at $720\text{ }^{\circ}\text{C}$ for 5 minutes. Afterwards, the pure Mg, pure Zn, and Mg-Y master alloys were added into the crucible and stirred for approximately 10 minutes after the complete melting, and then the ternary alloy was poured into a steel mold. When the temperature raised to $720\text{ }^{\circ}\text{C}$, the Mg-Pb master alloy was added into Mg-Zn-Y melt and the mixture was stirred for approximately 20 minutes until it completely melted. The size of the resulting alloy casting was $140 \times 30 \times 30\text{ mm}^3$, and all samples were cut from the castings.

The microstructure was observed with an optical microscope (OM, COOLPIX-4500) and a scanning electron microscope (SEM, JSM6701F). Phase analysis was conducted by X-ray diffraction (XRD, Siemens D5000), using monochromatic $\text{Cu-}\kappa\kappa$ radiation and a 0.05° step length. The scanning range was set from 20° to 80° .

Hardness was measured using a microhardness tester (HXD-1000TMB/LCD) at a load of 0.5 kgf and a loading time of 12 seconds. In the testing process, five indentations' data were created to provide an average of hardness values. Tensile tests were performed at ambient temperature using a microcomputer-controlled electronic universal testing machine (M-4030) with an electronic extensometer (YYU-25/50) at a crosshead speed of $1\text{ mm}\cdot\text{min}^{-1}$ with a gauge

length of 50 mm and a cross sectional area of $15 \times 3\text{ mm}^2$. Electrochemical polarization in 3.5% NaCl solution was conducted in a three-electrode cell on an electrochemical workstation (CHI660A) at a scanning rate of $5\text{ mV}\cdot\text{s}^{-1}$, and the size of the corrosion samples is $10 \times 10\text{ mm}^2$. The dynamic polarization curves were measured three times after open-circuit immersion for 1 hour in 3.5wt.% NaCl aqueous solution. The average corrosion potentials and currents density of the alloys were obtained after the Tafel-type fitting with the three polarization curves.

2 Results and discussion

2.1 Microstructure

Figure 1(a) shows the microstructure of the as-cast Mg97Zn1Y2 alloy, mainly composed of the α -Mg matrix and the secondary phase at grain boundaries. The dendrites are developed and distributed continuously. In terms of the Mg97Zn1Y2-xPb ($x=0.6\text{wt.}\%$, $1.2\text{wt.}\%$ and $1.8\text{wt.}\%$), the dendrite size decreased and changed from developed ones into the undeveloped broken and island-like dendrites, as shown in Fig. 1(b), (c), and (d). Compared with the as-cast Mg97Zn1Y2 alloy, after adding Pb into the Mg97Zn1Y2 alloy, a new particle phase appeared at the grain boundary and its volume fraction increased with increasing Pb content. The solid solution can be easily formed due to the high solid solubility of Pb in Mg. Pb was added to the Mg alloy, which helped to refine the microstructure of the alloy and changed the morphology of the secondary phase, thereby improving

Fig. 1: Microstructure of Mg97Zn1Y2 alloy (a) and Mg97Zn1Y2-xPb alloys [$x=0.6\text{wt.}\%$ (b); $1.2\text{wt.}\%$ (c); $1.8\text{wt.}\%$ (d)]

properties of the alloy^[21]. During the solidification process of the alloy, Pb concentration in the liquid phase was higher than that in the solid phase due to the difference concentration around the solid-liquid interface. The diffusion of the Mg atoms was hindered by the nuclei formation of LPSO phase, which hindered the growth of the secondary phase, and the network microstructure was changed to discontinuous^[14]. At the same time, the growth of the LPSO phase was also hindered.

The XRD patterns used to determine the phase composition of the Mg97Zn1Y2 alloy and Mg97Zn1Y2-xPb ($x=0.6\text{wt.}\%$, $1.2\text{wt.}\%$ and $1.8\text{wt.}\%$) alloys are shown in Fig. 2. The Mg97Zn1Y2 alloy consists of α -Mg matrix phase and Mg_{12}ZnY phase (the LPSO phase). After adding Pb into the Mg97Zn1Y2 alloy, the Mg97Zn1Y2-xPb ($x=0.6\text{wt.}\%$, $1.2\text{wt.}\%$ and $1.8\text{wt.}\%$) alloys predominantly consist of three phases: Mg_{12}ZnY phase, the α -Mg matrix phase, and a new particle phase of Mg_2Pb with weak diffraction peaks.

Figure 3 shows the SEM images of the Mg97Zn1Y2 alloy and Mg97Zn1Y2-xPb ($x=0.6\text{wt.}\%$, $1.2\text{wt.}\%$ and $1.8\text{wt.}\%$) alloys, and the island-like phases (new particle phase mentioned above) A, B, and C can be observed. According to the XRD spectrum (Fig. 2) and the SEM images (Fig. 3), these island-like phases are identified as the Mg_2Pb phase. Through the EDS analysis at point 001, 002, 003 in Fig. 3 and Table 1, the secondary phase at grain boundaries is Mg_{12}ZnY phase. Furthermore, the network-like compounds apparently changed from the continuous to the discontinuous and became randomly distributed around the matrix after the addition of a

Fig. 2: XRD patterns of Mg97Zn1Y2 alloy and Mg97Zn1Y2-xPb alloys ($x=0.6\text{wt.}\%$, $1.2\text{wt.}\%$ and $1.8\text{wt.}\%$)

small amount of Pb. Kashyap et al.^[22] found that the addition of Pb reduces the strain energy at the grain boundary of the Mg alloy, thus resulting in a decrease in the number of the secondary phases.

2.2 Mechanical properties

2.2.1 Hardness analysis

Figure 4 shows the hardness value of the Mg97Zn1Y2 alloy and Mg97Zn1Y2-xPb alloys ($x=0.6\text{wt.}\%$, $1.2\text{wt.}\%$ and $1.8\text{wt.}\%$). Compared with the as-cast Mg97Zn1Y2 alloy, the hardness value of the Mg97Zn1Y2-xPb alloys increased. When the Pb content is $0.6\text{wt.}\%$, the hardness value reaches the maximum of 89.1 HV. When the Pb content is further increased, the hardness value starts to decline. According to

Fig. 3: SEM images of Mg97Zn1Y2 alloy (a) and Mg97Zn1Y2-xPb alloys [$x=0.6\text{wt.}\%$ (b); $1.2\text{wt.}\%$ (c); $1.8\text{wt.}\%$ (d)]

Table 1: EDS elemental analysis for Mg97Zn1Y2 alloy and Mg97Zn1Y2-xPb alloys (x=0.6wt.% and 1.8wt.%)

Spot	Mg (at.%)	Zn (at.%)	Y (at.%)	Pb (at.%)
001	84.84	7.66	7.50	0
002	88.60	5.41	5.99	0
003	33.55	2.42	9.24	54.79

the alloy phase diagram^[23], Pb has the high solid solubility in Mg. During the casting process, partial Pb elements are dissolved in the matrix and partial Pb elements to form Mg₂Pb compound. And the matrix lattice distortion occurred, which plays a role in solid solution strengthening. Hence, the hardness of the alloy increased when Pb was initially added to the alloy. As Pb content continued to increase, the hardness of the alloy became relatively less due to the generation of a large amount of Mg₂Pb phase. Pb is a soft metal, so the hardness of the alloy reduced since more Pb atoms were incorporated into the Mg matrix^[24]. However, with the further increase of the Pb addition to 1.8wt.%, the hardness of the alloy increased slightly. This may be due to the dispersed secondary phase particles being well distributed throughout the substrate, which is useful in increasing the hardness of alloys.

Fig. 4: Hardness value of Mg97Zn1Y2 alloy and Mg97Zn1Y2-xPb alloys (x=0.6wt.%, 1.2wt.% and 1.8wt.%)

2.2.2 Tensile properties

Figure 5 shows the tensile properties of the Mg97Zn1Y2 alloy and Mg97Zn1Y2-xPb alloys (x=0.6wt.%, 1.2wt.% and 1.8wt.%). The tensile properties of the Mg97Zn1Y2 alloy increased slightly with the addition of Pb. When the Pb content was 1.2wt.%, the elongation of the alloy reached 8.4%, an increase of 12.3% compared to the Mg97Zn1Y2 alloy, but when the Pb content was 1.8wt.%, the strength and elongation of the alloy evidently declined.

The main effect of Pb on the Mg97Zn1Y2 alloy is to change the morphology and distribution of the secondary phase (Fig. 3). Since Pb mainly functions as a solution strengthening element^[25], the dendrites are refined and Mg₂Pb phases formed at the grain boundaries after adding an appropriate

Fig. 5: Tensile stress-strain curves of Mg97Zn1Y2 alloy and Mg97Zn1Y2-xPb alloys (x=0.6wt.%, 1.2wt.% and 1.8wt.%)

amount of Pb. The addition of Pb inhibits the grain boundary slipping effectively and prevents the extension of cracks, thereby increasing the strength of the alloy^[26]. Therefore, the grain boundary strengthening is the main reason for the improvement in the mechanical properties of the alloy with Pb content increasing from 0.6wt.% to 1.2wt.%. Therefore, the mechanical properties of the alloy containing 1.2wt.%Pb were better, especially for the elongation property.

The Mg₂Pb phase is distributed around the α -Mg matrix, which easily inducing cracks during the deformation process, resulting in fracture and poor elongation. Therefore, dendrite refinement not only improves the strength of the material, but also improves its plasticity. When dislocations occurred and passed through the hard phases, more dislocation rings formed which had the effect of strengthening and toughening on the alloy. When the addition of Pb was 1.8wt.%, the Mg₂Pb phase increased and extended into the inside of the grains, and the matrix was separated and the mechanical properties (tensile strength and elongation) of the alloy obviously decreased.

The α -Mg is a closed-packed hexagonal structure in which micro-cracks formed easily on the base surface, and cracks of different heights formed cleavage steps through the secondary cleavage or tearing. Figure 6 shows the tensile fracture morphology of the Mg97Zn1Y2 alloy and the Mg97Zn1Y2-xPb (x=0.6wt.%, 1.2wt.% and 1.8wt.%) alloys, while Fig. 6(a) shows the Mg97Zn1Y2 fracture morphology characterized by a typical intergranular brittle fracture and a coarse microstructure. From Fig. 6(b) and (c), we can see that a small quantity of shallow dimples appeared on the fracture surface of Mg97Zn1Y2-xPb (x= 0.6wt.% and 1.2wt.%) alloys, which is conducive with the elongation. With a further increase in Pb, the alloy underwent brittle fracturing, as shown in Fig. 6(d). As the load was applied, it acted directly on the brittle phase, making the alloy break, thereby cracks formed and expanded along the grain boundary, causing the alloy to fracture. Because the alloy has low bearing capacity and poor ductility, it fractured rapidly during the loading process; excessive Pb and Mg formed a compound phase at the grain boundary, so that its microstructure became continuous and coarse. The cracks were easily initiated at the interface between

Fig. 6: Tensile fracture morphology of Mg97Zn1Y2 alloy (a) and Mg97Zn1Y2-xPb alloys [x=0.6wt.% (b), 1.2wt.% (c) and 1.8wt.% (d)]

the secondary phase and the matrix, leading to the fracture having the characteristics of intergranular fracture. Through the fracture analysis, a regular pattern of toughness-plasticity showed in the fracture morphology, which was consistent with the analysis of the mechanical properties.

2.3 Corrosion resistance

The corrosion resistance of the alloy is closely related to the corrosion potential (E_{corr}) and current density (i_{corr}). On immersion in 3.5wt.% NaCl solution, the self-corrosion potentials of the designed alloys were -1.514, -1.427, -1.476 and -1.498 V, corresponding to Pb content from low to high, as shown in Fig. 7. Meanwhile, the corrosion current densities of the alloys were obtained by Tafel-type fitting. With an increase in Pb content, the self-corrosion potential of the alloy increased, then shifted in the negative direction. Table 2 summarizes self-corrosion potentials (E_{corr}) and current densities (i_{corr}) of the alloys. It indicates whether the addition of Pb improves the corrosion resistance of the alloy depending on the amount of Pb added. The secondary phase plays two roles in the corrosion process. When the amount of the secondary phase is large and the distance between the atoms is close to the distance between the matrix atoms, the secondary phase can play an effective role in inhibiting corrosion. When corrosion occurs in the secondary phase, its onset is delayed due to the nature of the secondary phase to a certain extent. In addition, a lot of micro batteries will be formed due to the potential of the secondary phase being more positive than that of the matrix, thereby accelerating the corrosion of the magnesium alloy.

The Mg97Zn1Y2-0.6wt.% Pb alloy exhibits the best corrosion resistance, and Mg97Zn1Y2-1.8wt.% Pb alloy exhibits poor corrosion resistance compared to the Mg97Zn1Y2 alloy, as shown in Table 2. The reason is that with the increase in Pb content, the amount of Mg_2Pb increased. The metal compound phase acted as the cathode and the α -Mg matrix as the anode, a lot of micro batteries could be formed, and the corrosion rate of the matrix was accelerated. At the same time, the continuous network microstructure became discontinuous with an increase in Pb content, leading to an increase in corrosion rate. Therefore, the corrosion resistance of the alloy was improved by adding a small amount of Pb (0.6wt.%).

Fig. 7: Tafel polarization curves of Mg97Zn1Y2 alloy and Mg97Zn1Y2-xPb alloys (x=0.6wt.%, 1.2wt.% and 1.8wt.%)

Table 2: Corrosion properties of Mg97Zn1Y2 alloy and Mg97Zn1Y2-xPb alloys (x=0.6wt.%, 1.2wt.% and 1.8wt.%)

Alloys	E_{corr} (vs.SCE) (V)	i_{corr} (A·cm ⁻²)
Mg97Zn1Y2	-1.514	3.766×10^{-3}
Mg97Zn1Y2-0.6wt.%Pb	-1.427	2.892×10^{-3}
Mg97Zn1Y2-1.2wt.%Pb	-1.476	3.284×10^{-3}
Mg97Zn1Y2-1.8wt.%Pb	-1.498	4.261×10^{-3}

3 Conclusions

In this work, the influence of Pb on the microstructure and the mechanical properties and corrosion resistance of the Mg97Zn1Y2 alloy was clarified and the following conclusions were drawn:

(1) Pb has an effect on the morphology and distribution of dendrites. A new phase, identified as Mg₂Pb, appears along the grain boundary, and dendrites refinement are attributed to the addition of Pb.

(2) Pb can improve tensile strength and hardness of Mg97Zn1Y2 alloy. When the Pb content is 0.6wt.%, the hardness of the alloy reaches a maximum of 89.1 HV. The improvement of the strength and hardness of as-cast Mg97Zn1Y2-xPb alloy is achieved by the effects of solid solution strengthening and grain boundary strengthening.

(3) Pb has an effect on the fracture mode of the alloy. It is evident that the fracture mode changes from a small amount of small-sized dimple fracture to fracture along the grain boundary as the Pb content increasing.

(4) The corrosion resistance of the Mg97Zn1Y2 alloy is improved by adding a small amount of Pb, and the Mg97Zn1Y2-0.6wt.% Pb exhibits the best corrosion resistance.

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