Research on microstructures of sub-rapidly solidified AZ61 Magnesium Alloy

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ABSTRACT

AZ61 magnesium alloy foils of 0.5–3.0 mm thick were successfully produced by using sub-rapid solidification technique. Microstructures of conventionally solidified (CS) and sub-rapidly solidified (sub-RS) alloys were examined by optical microscope (OM) and scanning electron microscope (SEM). The results showed that the cellular grain of 1.8–13.5 μm can be obtained during sub-rapid solidification process. Phase compositions and microdistribution of the alloying elements in the foils were analyzed by X-ray diffraction (XRD) and electron probe microanalyzer (EPMA), respectively. The eutectic transformation \(\text{L} \rightarrow \alpha\text{-Mg} + \beta\text{-} \text{Mg}_{17}\text{Al}_{12}\) and microsegregation in conventionally solidified AZ 61 alloy were remarkably suppressed in sub-rapid solidification process. As a consequence, the alloying elements Al, Zn, Mn showed much higher solid solubility and the sub-rapid solidification microstructures dominantly consisted of supersaturated \(\alpha\text{-Mg}\) solid solution. Meanwhile, the \(\beta\text{-} \text{Mg}_{17}\text{Al}_{12}\) phases located in the \(\alpha\text{-Mg}\) grain boundaries are largely decreased due to high solidification cooling rate.

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1. Introduction

Magnesium alloys have great potential for “3C” products, handheld tools and automotive applications primarily owing to low density, easiness of recycling and electromagnetic shielding characteristics [1–4]. However, its low mechanical properties, poor corrosion resistance and poor formability at room temperature severely restrict its widespread applications [5,6]. Due to their hexagonal lattice structure, magnesium alloys show a comparatively limited cold workability [7]. At room temperature, their deformation mainly depends on the slip that only takes place on the basal plane (0001) and in the \(<1120>\) direction and the twinning occur on the pyramidal plane (1012) [7]. It is well known that many properties of a crystalline material are influenced by its microstructure, and a finer grain size may contribute significantly to improve the room-temperature ductility performance, mechanical property and corrosion resistance of magnesium alloys: castings having fine grains usually show a more uniform distribution of solute and a better dispersion of secondary phases within their structure [8,9].

In general, grain size and microstructure of cast metals are directly influenced by the cooling rate. Different degrees of structural refinement and transformation can be obtained by the technology of sub-rapid solidification processing, which presently provide cooling rates up to \(10^3\) K/second. The microstructural changes involve phases with large and non-equilibrium solid solubilities, metastable crystalline phases, and microcrystalline structures [10]. The available literature on sub-rapidly solidified magnesium alloys is very sparse and few significant works have been undertaken so far.

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The present work is aimed at utilizing self-designed setup to prepare AZ61 magnesium alloy foils of 0.5–3.0 mm in thickness. The microstructural characteristics of the sub-RS Mg-alloy foils were investigated. For comparison, the cast specimen of AZ61 magnesium alloy was also fabricated by conventional solidification method. And the effect of cooling rate on grain size, morphology, alloying elements microdistribution and precipitated phase were presented.

2. Experimental Procedure

2.1. Preparation of Cast Specimens

Magnesium alloy for study (Mg-6%Al-1%Zn-0.2%Mn) was prepared in steel crucible in an electric resistance furnace. Magnesium, aluminium and zinc metal ingots of 99.90% purity were used and manganese was added as Al-10%Mn intermediate alloy. Magnesium was melted and aluminium and zinc were then added to the molten magnesium. A part of the molten alloy was poured into permanent steel mold which had been preheated to 300 °C and was cooled to room temperature in air, thus AZ61 conventionally solidified (CS) sample was obtained. All the above procedures were conducted in a flowing protective gas (50 vol.% dry air+50 vol.% CO2+0.3 vol.% SF6) to prevent burning and oxidation.

Fig. 1 shows the schematic of the self-designed sub-rapid solidification setup which is composed of fixed bracket, copper mold, crucible, thermocouple and vacuum container, etc. The copper mold contains a slide block which can be adjusted to change the thickness of the die cavity. When control valve turn on, the molten metal was injected into the cold cavity of the mold with a high injection rate under vacuum pressure, thus Mg-alloy foils with different thickness varied from 0.5 mm to 3.0 mm were obtained.

2.2. Measurements of Specimens

Specimens of the cast Mg-alloy were cut, mounted, polished and etched with 3% Nital according to standard metallographic preparation techniques [8]. Subsequently, microstructures of etched specimens were characterized by optical microscope (OM) and scanning electron microscope (SEM, JSM-5600LV). The grain size was evaluated by using the intercept method described in ASTM E-112 [11]. The grain boundaries were marked manually and evaluated statistically to obtain the mean grain size, and more than 50 intercepts were counted for each measurement. In order to obtain detailed information on the phase distribution in the specimens, phase identification was performed by X-ray diffraction (XRD-6000), and microdistribution of the alloy elements was analyzed by electron probe microanalyzer (EPMA-1600).

Fig. 1–Schematic of the experimental setup: 1.fixed bracket 2.copper mould 3.needle valve to control flow 4.thermocouple 5.quartz tube 6.molten metal 7.crucible 8.Argon 9.vacuum container.

Fig. 2–The sub-rapidly solidified AZ61 alloy foil.

Fig. 3–Optical micrographs of (a) CS AZ61 casting and (b) sub-RS AZ61 alloy foil of 1.0 mm in thickness at cross-section from a surface to the center.
3. Results and Discussion

Fig. 2 shows an AZ61 magnesium alloy foil obtained by sub-solidification technique in the present study. This foil is about 35 mm in length, 15 mm in width and 1.0 mm in thickness.

Microstructures of cross-section of the conventional specimen and the sub-rapidly solidified foil of 1.0 mm in thickness are presented in Fig. 3(a) and (b). The casting process determines shape and distribution of the different phases. The typical optical micrograph of conventional AZ61 alloy casting consists of a matrix $\alpha$-Mg with $\beta$ phase (the brittle intermetallic $\text{Mg}_17\text{Al}_{12}$) along the $\alpha$-Mg grain boundaries [12,13] in the form of continuous network, as shown in Fig. 3(a). In addition, grain size is quite large and its distribution is non-uniform due to the slower cooling rate [7]. Fig. 3(b) shows the optical micrograph of the AZ61 foil at cross-section from a surface to the center consisting of (I) equiaxed chill crystal zone, (II) columnar crystal zone and (III) equiaxed crystal zone. Comparing Fig. 3(a) with Fig. 3(b), it can be inferred that the microstructure of the sub-solidified AZ61 foil with a high rate of solidification is much more finely and homogeneously distributed than that of the conventional cast alloy. Moreover, the volume fraction, shape and distribution characteristics of intermetallic $\beta$-$\text{Mg}_17\text{Al}_{12}$ located in the $\alpha$-Mg grain boundaries as well as grain size are largely affected by solidification rate, and the microstructure dominantly consists of supersaturated $\alpha$-Mg solid solution. In order to discern the grain size of the cast foils in different thickness, scanning electron microscopy was performed to further observation. SEM micrographs

Fig. 4 – SEM micrographs of equiaxed crystal zone of sub-RS foils of (a)0.5 mm, (b)1.0 mm, (c)1.5 mm, (d)2.0 mm, (e)2.5 mm, (f)3.0 mm in thickness.
(Fig. 4) of equiaxed crystal zone of sub-RS foils show very fine grain size (1–20 μm) compared to 200–300 μm obtainable through conventionally air-cooled specimen. The grains of the sub-RS foils in all the deferent thickness are globular or cellular and uniformly distributed, as shown in Fig. 4(a)–(f), and grain size is evaluated by using the intercept method. The effect of thickness on mean grain size of the foils, observed in the present investigation, is given in Fig. 5, which shows that as the thickness is decreased from 3.0 mm to 0.5 mm, the grain size is significantly decreased from 13.5 μm to 1.8 μm. The results described above show that different degrees of structural refinement can be achieved by using the sub-rapid solidification device (Fig. 1) in this study. This can be attributed to the difference in solidification cooling rate. Fast cooling leads to fine grains whereas slow cooling produces coarse grains [13].

The X-ray diffraction patterns of sub-RS foils and CS casting AZ61 alloy are illustrated in Fig. 6. It can be clearly seen that the peaks of sub-RS foils are broader than that of the CS casting. The effect can be attributed to the grain refinement [14]. The results indicate that the phases in the CS AZ61 alloy casting, as in Fig. 6 (a), consist of α-Mg and β-Mg17Al12, which are also clearly visible in its optical micrograph (Fig. 3(a)).

However, compared with the phases in the CS casting, the eutectic transformations L → α-Mg + β-Mg17Al12 in conventionally solidified AZ61 alloy were suppressed to a great extent due to high cooling rates. Therefore, the diffraction peaks of the β-Mg17Al12 phase in the XRD patterns of sub-RS foils, displayed in Fig. 6 (b)–(d), are not obvious. Thus the main phase in sub-RS foils consists of α-Mg solid solution (see Fig. 3 (b)). Moreover, it can be seen that a little migration of diffraction peaks in the XRD patterns of sub-RS foils. The effect can be attributed to the α-Mg phase solid solution, which may lead to the change of lattice constant. Although the lattice constant is different between the phase of CC and RS alloys, they belong to the same phase [14].

EPMA micrographs of CS casting and sub-RS foils, presented in Fig. 7, show the microdistribution of the alloying elements Mg, Al, Zn and Mn in the microstructure. It is well known that sub-rapid solidification can obviously enlarge the solid solubility limit of solute elements and even can form single phase solid solution structure. In sub-rapid solidification, the solute atoms near solid/liquid interface cannot diffuse sufficiently, and the solidification is non-equilibrium solidification condition. When the movement velocity of solid/liquid interface is very fast and the solidification time is very short, there is no time for the solute atoms near interface to enrich and diffuse sufficiently to the far distance of the melt, thus they are captured by the high-speed moving solid/liquid interface.

It can be clearly seen that both CS casting and sub-RS foils have the similar element microdistribution trend: the Mg element distributes most inside of the grain, only a very small amount exits in grain boundaries; On the contrary, the Al, Zn and Mn elements mainly distribute on grain boundaries and few exist in grain. However, the whole microdistribution of the elements in the sub-RS alloy is much more homogeneous than that in CS casting. Additionally, the finer of grain size and the higher of solidification cooling rate, the whole microdistribution of the elements in the foils becomes more homogeneous.

4. Conclusions

AZ61 Mg-alloy foils varied from 0.5 mm to 3.0 mm in thickness were obtained by using sub-rapidly solidification technique. The microstructures of the sub-RS foils are much more finely and homogeneously distributed than that of the conventional casting. Moreover, the shape and distribution characteristics of intermetallic β-Mg17Al12 located in the α-Mg grain boundaries are largely affected by solidification rate. The phases in the CS AZ61 alloy casting consist of α-Mg and β-Mg17Al12 whereas the microstructures in the sub-RS foils dominantly consist of supersaturated α-Mg solid solution. As the thickness of foils is decreased, the grain size is significantly decreased. At the

**Fig. 5** – Mean grain size values of sub-RS AZ61 foils as a function of thickness.

**Fig. 6** – XRD patterns of (a) CS AZ61 casting and sub-RS foils of (b) 0.5 mm, (c) 1.5 mm, (d) 2.5 mm in thickness.
meantime, the finer of grain size, the whole microdistribution of the elements in the alloy becomes more homogeneous.

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Fig. 7 – EPMA micrographs of (a) CS casting and sub-RS foils of (b) 0.5 mm, (c) 1.0 mm, (d) 1.5 mm, (e) 2.5 mm, (f) 3.0 mm in thickness showing microdistribution of the elements.