Effect of melt superheating treatment on the cast microstructure of Mg-1.5Si-1Zn alloy

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Abstract The effect of melt superheating treatment on the microstructure of Mg–1.5Si–1Zn alloy is investigated in this article. The microstructure of samples poured into sand and metal molds shows that the primary Mg₂Si is considerably refined in size when the melt is superheated from 750 to 900 °C. However, its polyhedral morphology does not change. In addition, also the size of eutectic Mg₂Si phase obviously decreases, but the morphology still exhibits Chinese script type. Meanwhile, it is concluded that the reduction of the heredity plays an important role in the grain refinement of Mg–1.5Si–1Zn alloy.

Introduction

Recently, the strong demand for weight reduction of transportation vehicles has generated great research interest for light alloys from automobiles to aircraft industries. The Mg–Si system alloy deserves more and more attention owing to the excellent combination of various properties [1, 2], which mainly depends on the size and morphology of Mg₂Si. Whereas, the primary Mg₂Si phase in Mg–Si system alloy exhibits very coarse in normal and thus gives rise to poor properties of alloys [3, 4]. It is well known that the grain refining is responsible for improving mechanical properties. Besides modification, however, the superheating

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Key Laboratory of Automobile Materials of Ministry of Education and Department of Materials Science and Engineering, Jilin University, Nanling Campus, No. 5988 Renmin Street, Changchun 130025, P.R. China e-mail: jiangqc@mail.jlu.edu.cn treatment is also a simple and effective method for grain refinement to improve the microstructure and mechanical properties of the as-cast alloys [5–9].

During the past years, the phenomenon of grain refinement by superheating is attractive for extensive investigation from various perspectives. However, the exact mechanism has not been fully understood so far.

It is reported that the distribution of alloying elements will be homogeneous due to the thermal diffusion with the further increase of melt superheating temperature [7]. This will clearly affect the subsequent solidification process. Yin et al. [7] have found that the slight grain refinement after the melt superheating can be attributed to the increase in the undercooling of the melt of M963 superalloy. However, Li et al. [5] studied the effect of melt superheating on the microstructure of Al-16Si alloy and concluded that the higher cooling rate led to more effective nucleation of primary Si and resulted in the formation of the finer microstructure. In addition, Popel et al. [10] investigated the effect of repeated melting of the mother ingot on the thermal stability of a Zr₆₀Al₁₅Ni₂₅ glassy alloy and proposed that the liquid structure, consisting of moving atom clusters, is not homogenous, and one cluster may have a different atomic configuration from another. The atomic configuration of clusters in a liquid inherits from that of the mother ingot. Yin et al. [7] thought that the mechanisms of grain refinement without any or with low temperature superheating treatment of the melt can be attributed to the presence of undissolved primary carbide particles in the melt. Recently, Chen et al. [6] investigated the thermal behaviors of Al-7Si-0.55Mg melt and concluded that the superheating treatment modified the Si phase owing to reducing the possibility of heterogeneous nucleation of Si phase and changing the growth process of Si phase. Additionally, Qin et al. [11] studied the effect of melt superheating on the microstructure of Al–Si–Mg–Cu alloys and stated that the anisotropic growth of Mg_2Si particles was transformed to isotropic growth owing to the change of the original structure and heredity of the melt. According to the above analysis, the influence of melt superheating on the microstructure may be explained by the undercooling and heredity.

However, the investigation on the microstructure in Mg–Si–Zn system alloys refined by superheating has seldom been carried out in the previous work. Accordingly, the purpose of the present study focuses on the influence of melt superheating temperature on the microstructure of Mg–1.5Si–1Zn alloy. It is expected that the preliminary results can be significant in promoting the development of Mg–Si–Zn system alloys.

Experimental procedure

Industrially pure Mg ingot (99.85 wt.% purity) and Si (99.95 wt.% purity) were used as starting materials to prepare Mg-1.5Si alloy. Detailed preparation of Mg-Si alloy was described in our previous study [2]. About 250 g of Mg-1.5Si alloy was remelted in a graphite crucible in an electric resistance furnace under the protection of a mixed gas atmosphere of SF₆ (1%, v/v) and CO₂ (Bal.), and superheated to 750, 800, 850, and 900 °C, respectively. After the melt was hold for 5 min, the pure Zn (99.98 wt.% purity) preheated at about 150 °C was added to the melts to get the designed composition of Mg-1.5Si-1Zn alloys. The chemical composition of the experimental alloys, as measured with an ARL4460 Metals Analyzer, is shown in Table 1. The melts were manually stirred for about 2 min using a stainless steel impeller, held for 20 min, and then poured into a steel mold preheated at 150 °C to produce tabular samples of $12 \times 40 \times 95 \text{ mm}^3$.

Metallographic samples were prepared in accordance with standard procedures used for metallographic preparation of metal samples and deeply etched with alcohol solution containing 2.2%HNO₃ + 2.5%HCl (in vol.) for about 10 min. The microstructure characteristics of the specimens were investigated by using scanning electron microscopy (SEM) (JSM-5310, Japan), optical microscopy

Table 1 The chemical composition of alloys (wt.%)

Superheating temperature (°C)	Designed composition			Analyzed composition		
	Mg	Si	Zn	Mg	Si	Zn
750	Bal.	1.5	1.0	Bal.	1.24	1.15
800					1.30	1.04
850					1.66	1.35
900					1.78	1.21

(OM) (LEICADM.IRM), and X-ray diffraction (XRD) (D/Max2500PC Rigaku, Japan). Solidification temperature of samples was studied by differential thermal analysis (DTA) (Model Rigaku-8150, Japan). About 50 mg samples were heated at the speed of 20 °C/min, and then cooled to room temperature at the speed of 45 °C/min under the protection of high pure argon gas. Grain size was estimated by the line intercept method under low magnification of the microstructures.

Results and discussion

Figures 1 and 2 show the XRD pattern and SEM microstructures of the as-cast Mg–1.5Si–1Zn alloys at different melt superheating temperatures, respectively. The results reveal that the alloys only consist of Mg and Mg₂Si phases as expected, and no other phase presents, with the increase in melt superheating temperature. The microstructures of Mg–1.5Si–1Zn alloy can be described as consisting of polyhedral primary Mg₂Si particles and Chinese script type eutectic Mg₂Si phase in the Mg matrix.

It is very clear that the size of Mg_2Si changed with the increase in melt superheating temperature. Compared with



Fig. 1 XRD pattern of as-cast Mg-1.5Si-1Zn alloys in different superheating temperatures of (a) 750, (b) 800, (c) 850, and (d) 900 °C

Fig. 2 Scanning electron micrographs of as-cast Mg– 1.5Si–1Zn alloys in different melt superheating temperatures of (**a**) 750, (**b**) 800, (**c**) 850, and (**d**) 900 °C



the microstructure of Mg-1.5Si-1Zn alloys in different melt superheating temperatures, the morphology of primary Mg₂Si exhibits polyhedral and has almost no change, as shown in Fig. 2. When the melt is only held at 750 °C, the primary Mg₂Si shows coarse polyhedral morphology (Fig. 2a) and their average size is larger than 18 µm. While the melt superheating temperature reaches 800 °C, the primary Mg₂Si phase exhibits grain refinement (Fig. 2b), the average size of which is approximately 14 µm. With temperature increasing up to 850 °C, the average size of primary Mg₂Si further decreases to 11 µm (Fig. 2c), and their number slightly increases. When the melt is superheated to 900 °C, the size of primary Mg₂Si phase significantly decreases and the particles are well distributed throughout the Mg matrix with an average size of about 9 µm (Fig. 2d). Meanwhile, their amount obviously increases because of the reduction of size. The effect of the melt superheating temperature on the average size of primary Mg₂Si is shown in Fig. 3.

Furthermore, it can also be found that the size of eutectic Mg_2Si sharply reduces with the increase in melt superheating temperature, as shown in Fig. 2a–d. The detailed size and morphology of eutectic Mg_2Si are compared in Fig. 4. Under the condition of low melt superheating temperature (750 °C), the eutectic Mg_2Si shows highly developed Chinese script type morphology (Fig. 4a). With the melt superheating temperature (900 °C) increasing further, the eutectic Mg_2Si becomes finer in size; however, it still exhibits conventional Chinese script type in morphology (Fig. 4b).



Fig. 3 The change in primary Mg_2Si size with the increase in melt superheating temperature

To investigate the influence of the melt superheating temperature on microstructure of Mg–1.5Si–1Zn alloys at low cooling rate, the melts heated to 750 and 900 °C, respectively, were poured into a sand mold, and the OM microstructures are shown in Fig. 5a and b. It indicates that the coarse primary Mg₂Si particle is sharply refined and its amount slightly increases with the increase in melt superheating temperature. However, the polyhedral morphology still has no change. In addition, the eutectic Mg₂Si keeps Chinese script type morphology invariable, whereas, the





Fig. 5 Optical micrographs of Mg–1.5Si–1Zn alloys poured into sand mold in different melt superheating temperatures of (**a**) 750 and (**b**) 900 °C

size obviously reduces. It is observed that the microstructure of samples poured into sand and metal molds shows the same grain refinement rule, which is in accordance with the conclusion of Li et al. [5], namely, the Mg₂Si size is gradually refined and its morphology has almost no change with the increase in melt superheating temperature.

To understand the mechanism of grain refinement of Mg–1.5Si–1Zn alloy, the solidification behavior of Mg–1.5Si–1Zn alloy was investigated by DTA, and the heating and cooling curves obtained in the DTA experiments are shown in Fig. 6a–d.

From Fig. 6a and b, it can be concluded that the onset temperature of solidification in DTA increases by about 1.6 °C with the increase in heating temperature from 750 to 800 °C. Therefore, it is confirmed that the reduction of heredity has an important effect on the grain refinement in the case of less undercooling difference. At the same time, from Fig. 6c and d, the curves exhibit that the onset temperature of solidification increases by 5.4 °C with the heating temperature increasing from 800 to 900 °C, confirming that the undercooling has an influence on the grain refinement by avoiding the effect of the heredity. Therefore, it is clearly seen that the undercooling decreases gradually with the increase in melt superheat temperature, which is in accordance with the mention of Cao et al. [8]. It

is noted that superheating causes a reduction in the amount of undercooling observed before solidification begins [8]. However, it is well known that the increment of undercooling might cause a grain refinement. Therefore, the influence of undercooling on the microstructure refinement of Mg-1.5Si-1Zn alloy could be ruled out. It can be easily concluded that the reduction of heredity plays an important role in the grain refinement of Mg-1.5Si-1Zn alloy. Under the low melt superheating temperature (750 °C), there are large moving atom clusters in the melt. The clusters have a characteristic similar to that of the mother ingot because of the heredity. During the solidification, the large moving atom clusters are responsible for nucleating, and therefore, the grain size becomes larger. With the melt superheating temperature (900 °C) increasing further, the large moving atom clusters are gradually dissolved and become small in size. Finally, these small atom clusters can grow into refining particles, as shown in Figs. 2, 4, and 5.

Conclusions

(1) With the increase in melt superheating temperature from 750 to 900 °C, the coarse primary Mg_2Si particle is significantly refined, with an average size

Fig. 6 DTA curves attained by heating the superheated (750 °C) Mg–1.5Si–1Zn alloys to (**a**) 750 °C and (**b**) 800 °C, as well as by heating the superheated (900 °C) alloys to (**c**) 800 °C and (**d**) 900 °C, respectively, (the onset temperature of solidification is denoted by an arrow)



decreasing from about 18 to 9 μ m. However, the polyhedral morphology is still maintained. In addition, the developed Chinese script type eutectic Mg₂Si is also sharply refined without any variation in morphology.

- (2) The microstructure of samples poured into sand and metal molds shows the same grain refinement rule. The Mg₂Si is sharply refined in size, while it keeps almost no change in morphology with the increase in superheating temperature.
- (3) The reduction of heredity plays an important role in the grain refinement of Mg-1.5Si-1Zn alloy.

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