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# Commercial AM60 alloy for semisolid processing: Effects of continuous rheoconversion process on microstructure

XING Bo (邢 博)<sup>1</sup>, LI Yuan-dong (李元东)<sup>1,2</sup>, MA Ying (马 颖)<sup>1,2</sup>, HAO Yuan (郝 远)<sup>1</sup>, D.APELIAN<sup>3</sup>

 State Key Laboratory of Gansu Advanced Nonferrous Metal Materials, Lanzhou University of Technology, Lanzhou 730050, China;
Key Laboratory of Nonferrous Metal Alloys and Processing, Ministry of Education, Lanzhou University of Technology, Lanzhou 730050, China;
Advanced Casting Research Center, Metal Processing Institute, WPI, Worcester, MA 01609, USA

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**Abstract:** In order to verify the effect of CRP (continuous rheoconversion process) in the preparation of magnesium alloy semisolid billets, AM60 alloy billets were fabricated with CRP, and effects of pouring temperature, slope angle, and length of the reactor on the microstructure of AM60 alloy were investigated. The results show that the grain size is reduced with the decrease of pouring temperature. A small block/rosette grain is obtained when the pouring temperature is less than 680 °C. Therefore, the available temperature process interval/window has to be required. To change slope angle of reactor (from 30° to 45°) is helpful for formation of the small and block/rosette grains. Meanwhile, the reactor with a length of 500 mm is enough for copious nucleation of primary phase. CRP changes the solidification microstructure of billets by controlling the nucleation and growth of the primary phase in melt. **Key words:** CRP; semi-solid processing; AM60 alloy; nucleation; growth

# **1** Introduction

With the development of auto industry that turns to be more environmentally friendly, much attention is paid to the use of magnesium alloy in the auto production to comply with the trend of "low mass" as such alloy has a series of advantages such as low density, high specific strength and rigidity[1–3]. Among the forming processes of magnesium components, high press die casting (HPDC) plays a very important role. It was reported that the die-castings used in auto industry were up to 80%–90% among all of the magnesium die-castings[4]. Nonetheless, the use of magnesium die-castings also involves many obstacles, for example the alloy cannot be used in high temperature fields and cannot be strengthened by heat treatment. Therefore, in order to improve the quality of magnesium die-castings to expand its application scope, an advanced forming process should be employed.

Semisolid metal process is a potential advanced

process discovered by FLEMINGS et al since the early 1970s[5-6]. The process, which has various advantages compared with conventional liquid casting and solid extrusion based on the thixotropic behavior of non-dendritic microstructure metals, provides the possibility to produce near-net-shaped magnesium components with high quality[7].

Nowadays, one of the ways, the rheo-forming is the preferred one for development of new SSM process, wherein the slurry is formed during its solidification from molten melt to semisolid. Many methods have been employed to realize the fabrication of non-dendritic slurry, also known as "slurry-on-demand" or SoD. Among them, the new slurry-making technology CRP (continuous rheoconversion process) invented at WPI (Worcester Polytechnic Institute). exhibits great potential for commercial prospect for its simplicity and effectiveness to obtain non-dendritic microstructure[8–10]. In this process, two melt streams are mixed in a special reactor. The reactor provides heat extraction and forced convection of

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melt, resulting in copious nucleation and redistribution of nuclei throughout the melt. This enables highly globular slurry on demand for casting processes.

Previously, in order to provide a starting point for industry to adopt the CRP, researchers in WPI have created different reactors based on the mechanistic understanding of CRP to simplify the operation process. These reactors include tortuous reactor, split-channel plate and sloped steel tube[11]. The split-channel plate is a block of low carbon steel with two parallel channels which converge partway down the plate. Fundamental idea of the design is to provide copious nucleation within two separate flow streams which are then mixed with one another. In this work, the split-channel plate was used to investigate the preparation of magnesium alloy billets for semisolid forming. The effect of heat extraction and forced convection rate on microstructure of as-cast AM60 billet was investigated by controlling the pouring temperature, slope angel and length of split-channel plate. The aim of the work is to verify the effect of CRP in the preparation of magnesium alloy billets and to provide practical experience in the realization of the semisolid forming of magnesium components.

### 2 Experimental

Alloy used in this work is commercial AM60 alloy. The alloy optimization via thermodynamic simulation for semisolid processing was presented in the first part of the work. The available temperature of AM60 alloy is 170 °C.

The CRP apparatus used in this work is shown in Fig.1. The split-channel plate is a block of low carbon steel with two parallel channels which converge partway down the plate. The cooling pipe was designed at the bottom of plate. The reactors were mounted on a cart so that they could be wheeled around the floor. The cart can angle the reactor over a wide range. A steel pouring cup was attached to the top of reactor.

The alloy was placed in a metal holding crucible and melted in a resistance furnace. K-type thermocouples were used to measure the temperature of melt. When the alloy was heated to melt and held at 730 °C for 10 min, degassed with  $C_2Cl_6$  and hold at 710 °C for 10 min, then the temperature was adjusted to a required level, and finally, the melt was poured into pouring cup and flows onto the split channel plate. After traversing the plate, the melt cascades into receiving metal mold. In this work, the various pouring temperatures (700, 680, 660, 640 °C), slope angles of split-channel plate (15°, 30°, 45°, 60°), and lengths of reactor (500 mm and 850 mm) were employed. The detailed experimental arrangements are summarized in Table 1.



Fig.1 Split-plate reactor

Table 1 Processing conditions of CRP process			
Pouring	Slope	Length	Mold
temperature/°C	angle/(°)	/mm	temperature/°C
700,680,	30	500	25
660,640			
680	15,30,	500	25
	45,60		
680	30	500,850	25
	Processing condition Pouring temperature/°C 700,680, 660,640 680 680	Processing conditions of CRP point       Pouring     Slope       temperature/°C     angle/(°)       700,680,     30       660,640     680       680     15,30,       45,60     30	Processing conditions of CRP process       Pouring     Slope     Length       temperature/°C     angle/(°)     /mm       700,680,     30     500       660,640     680     15,30,     500       45,60     680     30     500,850

The fabricated billets were cut into samples with a diameter of 10 mm and a height of 10 mm. Samples were polished and etched with a solution containing 4% HNO<sub>3</sub> (mass fraction). The solid solution treatment was operated at 420 °C for 12 h. Samples after solid solution treatment were polished and etched with picric acid. The microstructure was examined by using MEF-3 optical microscope, and equivalent circle diameter was detected by software Image Pro plus 6.0 with solid solution treatment microstructure.

## **3 Results and discussion**

# 3.1 Microstructure resulted from different pouring conditions

Fig.2 shows the as-cast microstructure of AM60 billet prepared by conventional casting with metal mold at 680 °C. The white phase is primary  $\alpha$ -Mg and the dark continuous matrix is eutectic Mg<sub>17</sub>Al<sub>12</sub> phase. The predominantly dendritic morphology of conventional solidification is clearly visible.

Fig.3 exhibits the representative micrographs from experiments operated by CRP with different pouring temperatures. The solidification microstructures were primarily consisted of primary  $\alpha$ -Mg grains and eutectics at intergranular boundaries. It can be seen from the morphology that each of microstructures is refined to the

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traditional typical dendritic billets. In Fig.3(a), as the billet is produced in a high superheat (85 °C), the tiny dendrite is visible. Fig.3(b) exhibits a similar microstructure features, but includes more refined polygon particles as the superheat is relatively reduced In Fig.3(c), the most uniform as-solidified structure is observed at the pouring temperature of 660 °C, with the morphology with totally non-dendritic and more spherical particles. Furthermore, when pouring temperature decreases to 640 °C, the non-dendritic microstructure is sustained. Thus, the primary  $\alpha$ -Mg morphology varies with the decrease of pouring temperature from tiny dendritic to a globular morphology.



Fig.2 Typical dendritic microstructure of AM60 alloy during conventional solidification

It is concluded from this group of experiments that non-dendritic microstructures can be obtained in a relatively high superheat by using CRP process with the split-channel plate. This indicates that the reactor is able to extract very large amounts of heat in a short time. Therefore, it is not necessary to have a precursor liquid very close to the liquidus temperature in order to obtain thixotropic structures with this process. Certainly, a lower superheat is helpful for formation of more refined grains, but the experiment process shows that the excessively low superheat would lead to melt freeze onto reactor, and this phenomenon would decrease the yield of pours. Therefore, the available processing temperature interval/window for the preparation of AM60 billets with CRP has to be controlled above 640 °C.

As the split-channel plate reactor is used in CRP, slope angle is another important processing parameter having effects on the heat extraction and rate during forced convection of melt. Fig.4 exhibits the representative micrographs from experiments operated with different slope angles. It can be seen from the morphology that slope angle has remarkable effect on microstructure of AM60 billets. When the angle is small, the primary phases present in typical dendrite with small or short secondary arms, as shown in Fig.4(a). However, Figs.4(b) and (c) show that the primary  $\alpha$ -Mg evolutes gradually into an intermediate rosette-like morphology and slope angles of granular structures increase to 30° and even 45°. As the slope angle increases to 60°, the dendrite is visible again (Fig.4(d)). The primary  $\alpha$ -Mg morphology has varied with increasing slope angle from dendritic to a globular morphology, then to dendritic. Therefore, the microstructural evolution implies that the optimum slope angle is from  $30^{\circ}$  to  $45^{\circ}$ .

Effects of slope angle on the resultant microstructure may be due to the contact time between melt and reactor. If the angle is too small, melt would flow slowly and the contact time would be relatively long, and the reactor is



Fig.3 As-cast microstructures of AM60 billets with CRP at different pouring temperatures: (a) 700 °C; (b) 680 °C; (c) 660 °C; (d) 640 °C



**Fig.4** As-cast microstructures of AM60 billet with CRP at different slope angles of reactor: (a)  $15^{\circ}$ ; (b)  $30^{\circ}$ ; (c)  $45^{\circ}$ ; (d)  $60^{\circ}$ 

able to extract a large amount of heat, thus the solid shell is formed and the nucleation rate of combined melts is decreased. Conversely, if the angle is excessively large, melt would flow over the reactor in a very short time, the temperature drop would be small, which is disadvantageous for the survival of nuclei, therefore the thermal conditions of reactor lead to a lower level of nucleation. As a result, the grains grow freely and finally dendrite forms.

In order to investigate the effect of length of reactor on copious nucleation of primary phase during the early stage of solidification, different lengths are employed. Fig.5(a) shows the microstructures of AM60 billet produced with the length of 500 mm. It is clear from the micrograph that particle morphology irregularity reaches non-dendritic or just tiny dendritic. But with the length extending to 850 mm, the dendritic net-frame is visible (Fig.5(b)). Theoretically, a longer reactor would be helpful for heat extraction, but actually in the experiment, is can be found that the thick solid shell forms easily at the exit of reactor if the excessive length is employed. Thus, the reactor with length of 500 mm is enough for the copious nucleation of the primary phase.

From the research on microstructures of AM60 billets, it can be seen that in order to obtain a fine and equiaxial semisolid microstructure, the heat extraction and forced convection during CRP should be strictly controlled by the controlling of pouring temperature, slope angle and length of reactor. As a result, the optimum pouring temperature are determined between

640 °C and 680 °C, slope angle is from 30° to 45°, and length of reactor should be controlled to be 500 mm.



Fig.5 As-cast microstructures of AM60 billets by using CRP with different lengths of reactor: (a) 500 mm; (b) 850 mm

### 3.2 Size of particles

Fig.6 shows the microstructure of AM60 billet after solid solution treatment. During solid solution process,

the eutectic phase on grain boundary is totally solved into primary phase and the size of primary particles is distinct. It can be seen from Fig.6 that the size of particles of CRP billet is visibly smaller than that of conventional casting with metal mold.

Fig.7 shows the size distribution of particles. For the metal mold cast billet, most of the particles are greater than 150  $\mu$ m, and some are even up to 240  $\mu$ m. For the CRP billet, most of the particles are in the range of 40–80  $\mu$ m, and some beyond 100  $\mu$ m. So, it is evident from the size analysis that the grains are refined with CRP. But at the same time, there is a hint that the size distribution of CRP billet is uneven, which may be due to the fact that the convection rate of liquid is low and can be avoided by narrowing the width of reactor exit.



**Fig.6** Solid solution microstructure of AM60 billet from: (a) Metal mold cast; (b) CRP

#### 3.3 Mechanism of microstructure formation

During conventional solidification process, the wall of mold is the main interface that extracts superheat of melt. The heat lost on surface of casting leads to positive temperature gradient in casting. Therefore, the wall nuclei flow to centre of mold with high superheat and finally remelt, and a small part is survived only, so the grains grow to typical dendritic morphology under cooled melt, as shown in Fig.2.

If the solidification process is artificially controlled to enhance the copious nucleation, and at the same time the temperature field and concentration field is controlled to be uniform to ensure that the nucleated nuclei is survived, the growth of grains would be effectively suppressed[12–15]. This is the operative mechanism of CRP process. When melt flows over the reactor, the heat extraction and forced convection, offered by reactor, will lead to copious nucleation and survival of nuclei via homogeneous temperature. These nuclei are dispersed uniformly throughout the bulk liquid. The result is total suppression of dendritic growth of primary phase.



**Fig.7** Size distribution of particles observed on AM60 billet from: (a) Metal mold cast; (b) CRP

The pouring temperature changes final microstructures by affecting the undercooling of melt. According to solidification principle, a large undercooling leads to more nuclei nucleated as much heterogeneous substrate that meets the requirements of nucleation. Meanwhile, the low melt temperature is favorable for the survival of nuclei, thus the number of effective nuclei is increased if pouring temperature is low[16]. In CRP, if the high pouring temperature is employed, the reactor would fail to extract superheat of melt, and the positive role of reactor in enhancing nucleation would be suppressed, so the solidification process is similar to that without reactor. Conversely, if pouring temperature is close to that of liquidus, superheat is extracted easily as melt flows over reactor, and the copious nucleation would be enhanced. But as mentioned above, it is impossible to totally enhance nucleation rate by reducing pouring temperature, the

excessive low temperature leads to solidification of wall nuclei on surface of reactor and the pours shell forms, which will reduce the number of effective nuclei during subsequent solidification.

The slope angle alters the process of nucleation by means of the effect of heat extraction on cooling rate. Heat extraction can be calculated by the following equation[17]:

$$Q = \alpha t A (T - T_0) \tag{1}$$

where Q is the quantity of heat extraction,  $\alpha$  is heat transfer coefficient, t is time of heat extraction, A is area of heat extraction, T is temperature of melt, and  $T_0$  is temperature of reactor wall.

When melt flows over the reactor, the change of thermal and physical parameters of melt can be ignored, The following formula can be established:

$$\alpha \propto Re_{\rm m}^{0.8} Pr^{1/2}$$

where *Re* is Reynolds number, *Pr* is Prandtl number. Reynolds number increases with flow velocity. It can be seen that  $\alpha$  increases with flow velocity, and at the same time, the heat extraction also increases. On the other hand, if flow velocity increases, the stay time of melt on reactor *t* will decrease, and the heat extraction of melt is also decreased. Thus, there is a appropriate cooperation between  $\alpha$  and *t* that makes the heat extraction of melt arrive to the maximum and the largest undercooling will be obtained. So, in order to promote the effect of reactor on enhancing of copious nucleation, the slop angle should be adjusted in a appropriate range to control flow velocity of melt in the reactor.

### **4** Conclusions

1) The microstructures of AM60 alloy billets with non-dendritic morphology can effectively be fabricated by using CRP as the split-channel plate. The resultant microstructures consist of fine and polygon  $\alpha$ -Mg grains mainly due to the copious nucleation generated by high heat extraction and forced convection capability of the reactor.

2) The process parameters, such as pouring temperature, slope angle, and length of reactor, have influence on the final microstructure. In order to obtain a totally non-dendritic microstructure, the optimized pouring temperature are determined between 640 °C and 680 °C, the slope angle is from 30° to 45°, and the length of the reactor should be controlled to be 500 mm.

3) The pouring temperature affects the undercooling of melt, and finally affects the heterogeneous nucleation rate. The slope angle and length of reactor alter the process of nucleation by the effect of heat extraction on cooling rate of melt. In order to promote effective role of reactor, slop angle and length should be adjusted in a appropriate range to control flow velocity of melt and avoid melt freeze onto reactor.

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