Effects of novel self-inoculation method on microstructure of AM60 alloy

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Abstract: A novel cast processing method, self-inoculation method (SIM), was proposed. The process involves the addition of self-inoculant to melt, then pouring the melt to a mould through a multi-stream mixing cooling channel. In this paper, the process parameters were investigated. Results indicate that the melt treatment temperature, the amount of self-inoculant added, and the slope angle of the cooling channel are the key factors for SIM process. The optimized parameters are that the melt treatment temperature is between 680 and 700°C; the addition of self-inoculant is between 5wt.% and 7wt.%; and the slope angle of the cooling channel is between 30° and 45°. Further analysis reveals that SIM changes the solidification microstructure of slurry by controlling the nucleation and growth of the primary phase in the melt.

Key words: AM60 alloy; self-inoculation method; multi-stream mixing cooling channel; nucleation and growth

Microstructure of material is the core content of material science and engineering, and the key bridge between processing and performance of material. There is a common view that fine-grain structure of metal material enables an improvement of mechanical properties. Therefore, effective control of metal material’s microstructure during the forming process is a target of research. During the past several decades, people have invented some chemical methods to refine the grains by the addition of chemical grain refiner, such as adding selenium to lead alloys, or adding Al-Ti-B/C to aluminum alloys. These chemical methods achieved the effect of refined grain, but also had some disadvantages, such as strict limit of alloy types and chemical pollution of metal melt. With further development of research work, people have tended to obtain fine grain by controlling heat diffusion and dynamic conditions during the casting process, which include vibration or stirring of melt; such as electromagnetic vibration, electromagnetic stirring or mechanical stirring. Among them, the idea that let the slightly superheated alloy melt flows through a cooling slope to enter a mould is the most interesting because of its simplicity and effectiveness in obtaining non-dendritic microstructure [1-3]. However, the application is restricted sometimes, such as an inconvenient control for getting slightly superheated alloy in practice. This paper introduces a novel casting method which includes some characteristics of the “cooling slope method” and makes an improvement from it, named the “self-inoculation method (SIM)”, which involves the addition of self-inoculant to melt, then pouring the melt to mould through a multi-stream mixing cooling channel. The experiments show the formation of a non-dendritic microstructure using the SIM method even if the melt was poured with a greater degree of superheating.

1 Experimental procedures

The main features of the SIM process are illustrated schematically in Fig. 1. It consists of a crucible, a multi-stream mixing cooling channel and a mould. Figure 2 shows the multi-stream mixing cooling channel. The cooling channel was specially designed with an optimal slope control and a water cooling system, which absorbs the melt superheat and mixes the two streams of melt into one. The self-inoculant, 5 mm x 5 mm x 5 mm in size, was prepared with the same composition as the melt to avoid chemical pollution of the melt. The processes of SIM can be divided into three stages. First, melt the alloy, then reduce it to the desired temperature. Second, add the self-inoculant to the melt and stir the melt quickly. Finally, pour the melt into the mould through the multi-stream mixing cooling channel.

The alloy used in this work is commercial AM60 alloy with a liquidus temperature of 615°C and a solidus temperature of 540°C. The chemical composition of AM60 alloy is shown in Table 1.
All experiments were carried out using an SG2-75-10 electrical resistance furnace with a steel crucible that 150 mm in diameter and 250 mm in height. A K-type thermocouple was inserted into the middle of the crucible to measure the temperature of the melt. The AM60 alloy was heated to melt and held at 730°C for 10 min, degassed with C2Cl6 and held at 710°C for 10 min. Then the temperature of the melt was adjusted to the required temperature (melt treatment temperature), the self-inoculant was added to the melt, and the melt was quickly stirred with a steel bar preheated to 200°C; finally, the melt was poured into the mould through the multi-stream mixing cooling channel.

The processing parameters were investigated; namely the melt treatment temperature, the addition amount of self-inoculant and the slope angle of the cooling channel. At first, different melt treatment temperatures were tested with a constant addition of self-inoculant and a constant slope angle. In this way an optimized melt treatment temperature was obtained. Then, an optimized addition of self-inoculant was obtained using the optimized melt treatment temperature and a constant slope angle. Finally, the optimized slope angle was determined, using the optimized values for the melt treatment temperature and the addition amount of self-inoculant.

The billets fabricated were cut into samples for heat treatment and metallographic examination. The metallographic samples were polished and etched using a solution containing 4% HNO₃. Another group of samples was treated by solid solution treatment. The evolution of the microstructure during the solid solution process includes two main steps. During the first step, the eutectic phase on grain boundary was totally decomposed into primary phase and the size of primary particles was distinct. During the second step, the primary phase was obviously coarsening with the increasing holding time. So in order to reflect the grain size of initial microstructure, the solid solution parameters were controlled at 420°C for 12 h to avoid the obvious coarsening of the primary phase. The samples after solid solution treated were polished and etched using picric acid. The microstructure was examined by an MEF-3 optical microscope, and the equivalent diameter of grains was obtained according to the average values of several measurements.

2 Results and discussion

2.1 Microstructure of the alloy by conventional solidification

Figure 3(a) shows the as-cast microstructure of an AM60 billet prepared by conventional casting with a pouring
temperature 700°C. The white phase is primary $\alpha$-Mg and the dark continuous matrix is the eutectic Mg$_{17}$Al$_{12}$ phase. The predominantly dendritic morphology of conventional solidification is clearly visible. The grain size is up to 191 μm.

2.2 Effect of melt treatment temperature on microstructure of AM60 alloy

Figure 4 shows the microstructure of AM60 alloy at different melt treatment temperatures. The billets were cast using a constant addition of 5% and slope angle of 30°. Figure 4(a) shows that the primary $\alpha$-Mg with a dendritic morphology is formed at the high treatment temperature of 720°C. Figures 4(b, c), however, show that the primary $\alpha$-Mg changes gradually to an intermediate rosette-like morphology and then some granular structures when the treatment temperature decreases to 700°C and 680°C, respectively. When the treatment temperature further decreases to 660°C, the dendritic structure is visible again (Fig. 4(d)). The primary $\alpha$-Mg morphology varied with decreasing treatment temperature from dendritic to a globular morphology, then to tiny dendritic. Therefore, the microstructural evaluation implies that the optimized melt treatment temperature is between 680 and 700°C.

![Fig. 4: As-cast microstructures of AM60 billet by SIM at different melt treatment temperature: (a) 720°C, (b) 700°C, (c) 680°C, (d) 660°C](image)

Figure 5 shows the relationship between the melt treatment temperature and the grain size, and the corresponding solid solution microstructure of AM60 alloy prepared by SIM. The average grain size decreases from 68 μm to 52 μm with decreasing melt treatment temperature from 720°C to 680°C. But, when the temperature decreases to 660°C, the size increases to 58 μm.

The melt treatment temperature influences both the undercooling of the melt and the dissolving of the self-inoculants. It is well known that a big undercooling promotes copious heterogeneous nucleation throughout the melt to form fine equiaxed morphology\[^{[4]}\]; so the high melt treatment temperature reduces the heterogeneous nucleation. The self-inoculant play a role as internal chill which are used in enhancing heterogeneous nucleation of the melt. If the treatment temperature is high, the self-inoculant is dissolved fully. So the heterogeneous nucleation is weakened, leading to the development of a dendritic microstructure. However, if the pouring temperature is very low, a solid shell forms readily on the cooling channel because more
heat is extracted through the cooling channel, which reduces the number of effective nuclei for solidification relative to the growth rate as the dendritic morphology forms finally (660 °C).

2.3 Effect of addition amount of self-inoculant on microstructure of AM60 alloy

Figure 6 shows the effect of addition amounts of self-inoculant on the microstructures of AM60 alloy. The billets were prepared with a constant temperature of 680 °C and a slope angle of 30°. The amount added governs the undercooling and heterogeneous nucleation in the melt. If a lesser amount was added, the self-inoculants dissolved quickly and a small temperature drop was achieved, the efficiency of self-inoculants weakened (Fig. 6(a)). In contrast, if an excessive amount was added, the alloy temperature reduced greatly and a solid shell forms easily on the cooling channel. As a result, the dendrites were produced (Fig. 6(d)). The optimized addition amount of self-inoculant should be between 5% and 7%.

![Fig. 6: As-cast microstructures of AM60 billet by SIM with different addition amount of self-inoculant: (a) 3%, (b) 5%, (c) 7%, (d) 9%](image)

Figure 7 shows the relationship between the addition amount of self-inoculant and the grain size and the corresponding solid solution microstructure of AM60 alloy by SIM. With the addition amount increases to 7%, the grain size decreases to 41 μm. However, with the excessive addition of 9%, the size increases to 62 μm.

2.4 Effect of slope angle of the cooling channel on microstructure of AM60 alloy

The microstructures of AM60 billets obtained with slope angles of 15°, 30°, 45°, and 60° are shown in Fig. 8 (with constant temperature of 680 °C and addition amount of 5%). Figure 8(a) shows that dendritic structure is clearly visible at 15°. Figures 8(b) and 8(c) show that the non-dendritic microstructure was obtained with slope angles of 30° and 45°. However, the results show that for slope angles higher than 45°, the solid particles show a non-uniform and coarse size (Fig. 8(d)). The solid solution microstructure and equivalent grain diameter (Fig. 9) show that the slope angle of 45° generated fine size of 51.4 μm.

![Fig. 7: Relationship between the addition amount of self-inoculants and the grain size and the corresponding solid solution microstructure of AM60 alloy by SIM](image)
The slope angle of the cooling channel affects the flowing time and shear stress of the melt on the cooling channel. If the slope angle is too large, the melt flows over the cooling channel within a very short time; and the temperature drop is small due to a weak cooling effect of the cooling channel. Correspondingly, the small degree of under-cooling weakens the heterogeneous nucleation of the solidification process. Besides, the large slope angle reduces the shear stress which can spall the quench crystals formed on the channel surface. In contrast, the small angle leads to a slow speed of melt flow and the solid shell forms on the cooling channel readily.

2.5 Solidification behavior in the SIM process

Flemings, et al[5] revealed that under a constant cooling rate, the grain multiplication leads to granular crystals of castings. It seems that the formation of a large amount of initial nucleus grains during the solidification process is the key part of microstructure refining. During the SIM process, the essential metallurgical feature can be summarized as the forced nucleation and survival of nuclei, resulting in the increase of grains density and a small grain size as the growth of grains is limited.

(1) Effect of self-inoculant

The suspension cast process was invented by researchers of the former Soviet Union in the 1960s; and involves adding small solid metal particles into the liquid metal during pouring[6]. The critical under-cooling is decreased greatly due to the addition of un-melted particles, which act as foreign nuclei. As a result, the ingot with fine grains is obtained. In the SIM process, the step of adding self-inoculant to the melt has similar features to suspension casting. On the one hand, the self-inoculant plays the role of decreasing the melt temperature. When the self-inoculant is added to the melt, it absorbs heat from the melt, leading to local under-cooling of the melt around the particles of self-inoculant. According to the classic solidification theory, the interface energy of heterogeneous nucleation is less than the homogeneous nucleation. The critical grain size is small and nucleation will happen with a small under-cooling. So the atom clusters of short-range order in the melt infiltrating around the particles will be the nucleation substrate and lead
to heterogeneous nucleation in the under-cooling. On the other hand, with the optimized melt treatment temperature, the self-inoculant in the melt will be partly dissolved, which leads to a large amount of atom clusters. As the self-inoculant was prepared from the same composition as the liquid, the structure of these atom clusters is close to that of the melt, so the solute atoms attach those atom clusters easily and the atom clusters grow to grain with a small under-cooling. Therefore, compared to the traditional cooling slope method that there is no nucleation in the melt, the alloy poured into the cooling channel in the SIM process was partly nucleated, which effectively increased the grain density during the finally solidification process.

(2) Effect of multi-stream mixing cooling channel
The melt poured into the cooling channel is divided instantly into two parts. The channel takes away heat from the liquid on the channel surface. According to the wall mechanisms [7], when liquid flows over the cooling slope nucleation takes place on or near the slope wall. Under the vigorous shear action offered by the slope wall, which exerts a great force on the nuclei growing along the slope wall, the nuclei can be easily separated from the wall and dispersed in the melt. With the continuous pouring, new nuclei can form on the surface of the wall and go through the same cycle so that the melt contains a larger number of smaller nuclei, provided that solid shell was avoided. At the same time, two flows of melt are merging in the middle of the channel; the mixing action promotes the heat to be given off from the melt and provides a uniform temperature field and concentration field, which limits the trend of dendritic growth. Therefore, the possibility of survival of nuclei is high throughout the volume of the melt; and nucleation is expected to occur progressively throughout the entire volume of the liquid. As a result, they can grow into individual grains, resulting in significant grain refinement.

2.6 Advantages of SIM process
As a novel casting method for refining grains of the alloys, the advantages of SIM over other existing refining methods are high flexibility in terms of processing conditions and the requirements for materials. First, it is convenient in practice; the melt could be poured at a high over-heating to expand the operational range of temperature. Second, the self-inoculant, prepared from the same composition as the melt, avoids chemical pollution of the melt. Third, the method is widely applicable. Our experiments showed that the SIM method could be used for Mg-alloys, Al-alloys, Zn-alloys and Cu-alloys. Meanwhile, the SIM is a simple and low cost process. Complex equipment is not required, and the scrap metal is reusable. All of these offer a lower integrated part cost. Finally, the novel process can be applied to existing die casting machines or forging machines without significant changes.

3 Conclusions
The SIM process has been shown to effectively refine grains of the AM60 alloy. With the optimized process parameters, the primary $\alpha$-Mg phase has a uniform distribution in the eutectic matrix, presenting in fine size and non-dendritic morphology. The process involves self-inoculants being added to melt, then the melt being poured to mould through a multi-stream mixing cooling channel. The self-inoculant decreases the melt temperature and promote heterogeneous nucleation. The rapid cooling and forced mixing provided by the multi-stream mixing cooling channel enhance wall nucleation and limit the trend to dendritic growth. Thus refined grains of alloy can be obtained over short times. During the process, the melt treatment temperature, the amount of addition of self-inoculant, and the slope angle of the cooling channel were the key factors. The optimized temperature is between 680 and 700°C; the addition of self-inoculant is between 5wt.% and 7wt.%; and the slope angle of the cooling channel is between 30° and 45°.

References