

Semisolid Structure Formation and Semisolid Casting

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Semisolid metal forming has now been accepted as a viable technology for production of components with complex shape and high integrity. The advantages of semisolid metal forming can only be achieved when the feedstock material has a non-dendritic semisolid structure. A controlled nucleation method has been developed to produce such structures for semisolid forming. By controlling grain nucleation and growth, fine-grained and non-dendritic microstructures that are suitable for semisolid casting can be generated. The method was applied to hypoeutectic and hypereutectic Al-Si casting alloys, Al wrought alloys and a Mg alloy. Parameters such as pouring temperature, cooling rate and grain refiner addition were controlled to achieve copious nucleation, nuclei survival and dendritic growth suppression during solidification. The influences of the controlling parameters on the formation of semisolid structure were different for each of these alloy groups. The as-cast structures were then partially remelted and isothermally held. Semisolid structures were developed and followed by semisolid casting into a stepped die.

KEY WORDS: Semisolid processing; Semisolid structure formation; Semisolid casting; Controlled nucleation

1. Introduction

Semisolid metal forming (SSMF) is an emerging technology for near net-shape production of high quality engineering components, in which metal alloy is processed at a temperature between its solidus and liquidus temperatures^[1,2]. The process combines a number of advantages of both casting and forging. Compared to casting from the liquid state, SSMF provides a more stable filling front, places lower thermal loads on the metal dies, and has inherently less shrinkage to be fed. On the other hand, compared to solid forging, it permits the filling of more complicated shapes and thinner sections. The advantages of SSMF can only be achieved when the feedstock material has a non-dendritic grain structure. When the material is reheated to its semisolid state, it can achieve a semisolid structure that has globular solid particles suspended in a liquid matrix^[3]. The globular structure provides unique flow behaviour when it is sheared during the forming process, with minimal surface turbulence and splashing, and this smooth die filling assists in producing good microstructural integrity for the final products^[4].

SSMF has two important parts: semisolid structure formation and semisolid casting. To develop semisolid structure, external convection or shear is often applied during solidification of the melt, breaking up the dendritic structure. Several methods have been reported to successfully produce the semisolid structure in Al and Mg alloys using forced convection. These include mechanical stirring^[5], electromagnetic stirring^[6], ultrasonic stirring^[7] and, most recently, melt mixing^[8]. The external convection methods usually involve extra equipment and com-

plicated operation. This results in a significant cost increase for the feedstock, which is one of the main reasons that temper a wider application of SSMF. It has become more and more important to develop a simple, practical and less expensive method to produce the globular semisolid structure.

An alternative to the external forced convection is solidification control. During solidification, microstructure formation is affected by parameters such as nucleation density, grain growth, solute redistribution, ripening and interdendritic fluid flow. A controlled nucleation method has been developed to use the control of certain solidification conditions, such as pouring temperature, cooling rate and grain refinement, to maximise grain nucleation and suppress dendritic growth, thereby producing a starting microstructure that is fine-grained and less dendritic. Such microstructures can evolve to a globular semisolid structure after partial remelting and isothermal holding, which is usually employed prior to the semisolid casting^[4,9]. The method has incorporated some features of liquidus casting^[10], grain refinement^[11] and new rheocasting^[12]. In this study, the controlled nucleation method was applied to Al foundry alloys (hypoeutectic and hypereutectic Al-Si alloys), Al wrought alloys and Mg alloys. The relative effectiveness of each nucleation control mechanism in the different alloy systems, on the formation of semisolid structure was examined.

Semisolid casting can be performed in traditional die-casting machine or squeeze casting machine. Instead of the fully liquid metal, a semisolid slurry/billet with semisolid structure is used. In this work, the as-cast materials were partially remelted and isothermally held in the semisolid condition to enable microstructure evolution. Semisolid casting was performed in a squeeze casting machine. The effect of

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the feedstock microstructure on semisolid castability was determined.

2. Experimental

The alloys used in this study were hypoeutectic Al-Si alloy A356 (Al-7%Si-0.35%Mg), hypereutectic Al-Si alloy A390 (Al-17%Si-4.5%Cu-0.7%Mg), Al wrought alloy 6063 (Al-0.4%Si-0.6%Mg) and Mg alloy AZ91 (Mg-9.0%Al-0.65%Zn). The A356, 6063 and AZ91 were commercial ingots. Alloy A390 was produced in-house using 99.7% Al. For Al alloys, the ingots were melted in an induction furnace and degassed. Al-5Ti-B master alloy was used as grain refiner for alloy A356 and alloy 6063. AlP-ready master alloy ALCUP was used in A390 alloy for primary silicon phase refinement. For Mg alloy, AZ91 was charged in a dedicated electric resistance furnace under the protection of SF₆ cover gas. The melts were cast into cylindrical steel moulds, with a cylindrical cavity of 50 mm in diameter and 70 mm deep for a lower cooling rate and a cylindrical cavity of 20 mm in diameter and 200 mm deep for a higher cooling rate. Various pouring temperatures were used to give a range of superheats. The mould preheat temperature was 200°C.

Small cylindrical samples (ϕ 20 mm×10 mm) were cut from the castings. For Al alloys, the samples, after preheating, were placed in a molten salt-bath for partial remelting and isothermal holding. The heating rate was about 15°C/s. For Mg alloys, the samples were placed in an electric resistance furnace with the protection of SF₆. A large steel block was used as a heat reservoir. The isothermal holding was carried out at a temperature in the semisolid region determined from the phase diagram of each alloy. The samples were then quenched in iced water for microstructural examination.

Semisolid casting was carried out using an Ube 250 ton squeeze casting machine. The billet was placed on a ceramic insulator sample base and reheated in an induction furnace and held at 580°C for 5 min prior to semisolid casting. A stepped die was used in the semisolid casting experiments^[13].

3. Results

3.1 Hypoeutectic Al-Si alloy A356

Figure 1 shows the influence of different pouring conditions on initial as-cast microstructure and on semisolid evolved microstructure. The grain/particle size and morphology varied significantly with the pouring conditions. Figure 1(a) shows the as-cast microstructure of the sample from high temperature pouring (725°C). It had a coarse-grained dendritic structure with grain size 900 μ m and dendritic morphology. After 15 min of isothermal holding, the solid phase evolved to a rounded morphology, forming an interconnected solid network. A large amount of the eutectic (about 25%) is intragranular (Fig.1(b)). As the pouring temperature decreased, the grain size of the as-cast microstructure was significantly reduced. The grain size of the 675°C-poured mate-

rial was 350 μ m and the grains were equiaxed. However the microstructure was still dendritic, as shown in Fig.1(c). In its semisolid microstructure, the interconnected appearance had disappeared, α -phase evolved to rounded and separated particles, separated from each other by eutectic. Low temperature pouring (650°C) resulted in a fine-grained structure. The dendritic grain morphology was less obvious and, instead, some of the primary grains had a rosette-like morphology. The grain size was approximately 200 μ m. A high proportion of the eutectic phase was intergranular (Fig.1(e)). After isothermal holding, the microstructure developed into a clearly globular structure. Globules of α -phase with a mean size of 100 μ m are uniformly distributed in the eutectic matrix. Nearly all of the eutectic is intergranular (93%).

When the low temperature pouring was combined with grain refinement, the as-cast microstructure became more rosette-like, with an average grain size of about 160 μ m (Fig.1(g)). During microstructural evolution, it also evolved to a globular structure and attained a globular state faster than the non-grain refined material, presumably because its as-cast structure was less dendritic.

Semisolid casting was carried out using billets produced with different pouring conditions. The billets were reheated to 580°C and isothermally held for a certain time in an induction furnace before casting. Figure 2 shows the semisolid castings and their X-radiographs made by materials with pouring temperatures of 725, 675, 650 and 650°C combined with grain refinement and after 5 min isothermal holding.

The billet made by high temperature pouring did not fill the die cavity, with only the first two steps being filled, and the third step being partly filled (Fig.2(a)). As a result, a large portion of material remained in the biscuit. Even the nominally filled steps were of poor quality, with a big fold defect on the back of the casting. The radiograph revealed the presence of severe porosity throughout the casting, including cracks and large cavities. Oxide inclusions were also observed by optical microscopy.

The filling ability for the 675°C-poured material was improved, however there is still half of the last step unfilled (Fig.2(b)). A large sunken defect was observed in the first step. X-radiography revealed the presence of a large isolated pore and a large amount of microporosity. The low temperature poured material (650°C) showed a further improvement in filling ability. It almost completely filled the die except for one top corner. There are rough flow marks and local surface roughness on both sides of the casting. Radiography revealed that the internal defects are significantly reduced compared to the 725°C- and 675°C-poured materials, however porosity is still severe in the first two steps. The grain-refined material resulted in a completely filled casting with all of the die details clearly visible (Fig.2(d)). No defects were observed on the casting surface and the flow marks had become very faint. X-rays revealed a very sound casting free of internal porosity.

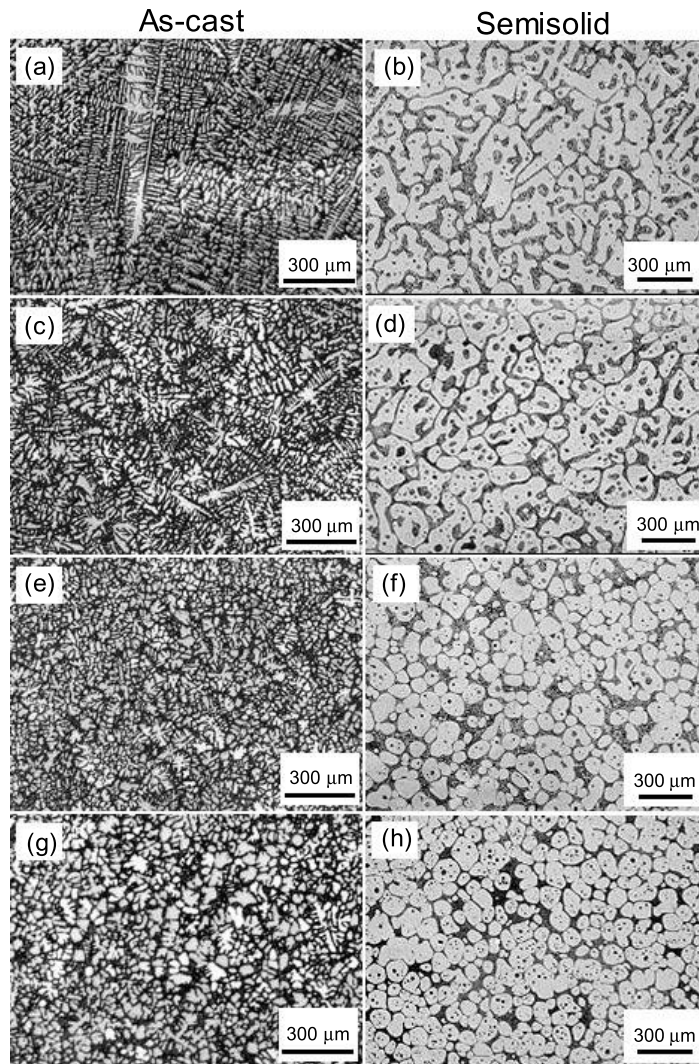


Fig.1 Microstructures of the castings produced by different pouring conditions (a) and (b) poured at 725°C, (c) and (d) poured at 675°C, (e) and (f) poured at 650°C, (g) and (h) 650°C pouring combined with grain refinement. (a), (c), (e) and (g) are as-cast microstructures, (b), (d), (f) and (h) are semisolid microstructures after partial remelting and isothermal holding at 580°C for 15 min

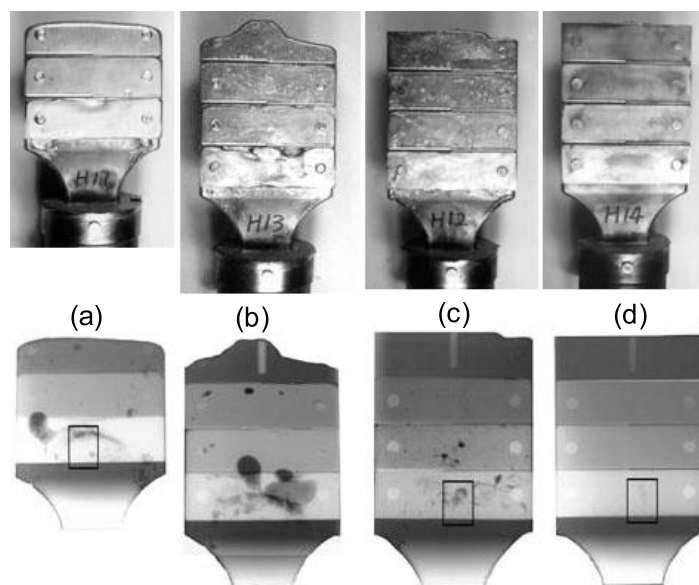


Fig.2 Semisolid castings and their X-radiographs; produced from billet poured at (a) 725°C, (b) 675°C, (c) 650°C and (d) 650°C, combined with grain refinement. The billets are isothermally held at 580°C for 5 min before casting

The semisolid castings were sectioned at the position where X-radiography revealed the most severe internal defects, which is indicated in Fig.2. For the 725°C-poured billet, extreme segregation occurred with almost total separation of the eutectic and primary phase, where the primary phase was packed in the gate area and almost pure eutectic was found in the stepped die. This is because the high-temperature-poured billet formed a structure with a solid network or skeleton in the semisolid slurry. When the slurry was used for semisolid injection, the solid skeleton could not pass through the narrow gate and thus became packed in that region. Only the liquid, which was distributed within the solid network, was then squeezed out and into the die cavity by the applied pressure. Poor castability of the non-globular semisolid slurry resulted in incomplete filling, internal cavities, porosity and cracks.

For the low-temperature-poured material, severe segregation was observed but the fraction of primary phase in the stepped die area was increased to about 0.15. Primary phase developed into rounded particles, separated from each other by the eutectic liquid. Compared with the solid network, the interlocking between the solid particles would have been less severe and the movement of such particles less hindered. This allowed more material, including some of the solid, to be pushed through the gate into the die. The mobility of the solid grains resulted in an improvement of the material filling ability. The semisolid castability and the casting quality could be further improved when the isothermal holding time increased, as the semisolid slurry developed into a more globular structure. This will be discussed in the following section.

When low temperature pouring was combined with grain refinement, there is no severe segregation in the whole casting. The α -phase particles are uniformly distributed in the eutectic matrix with a solid fraction of 0.5, which is close to that of the material placed in the shot sleeve before casting. In the microstructure of the casting, the primary phase appeared as globular particles approximately 100 μm in size, dispersed uniformly in a eutectic matrix. With this pouring condition, the rosette-like as-cast grains evolved to globular particles after isothermal holding. During semisolid processing, interlocking of the fine, globular solid particles is minimized, the solid particles could move, rotate, pass each other, and flow with the liquid phase during the filling process. The material behaved as a viscous liquid and this resulted in good casting quality with complete filling and less segregation.

3.2 Hypereutectic Al-Si alloy A390

As-cast microstructures and semisolid remelted microstructures of A390 are shown in Fig.3. The materials were cast in a permanent steel mould at a superheat of 115°C. The mould is specially designed to obtain a large heat extraction rate during solidification to prevent the “explosive” growth of Si phase. The as-cast microstructure consisted of a faceted pri-

mary Si phase, a dendritic α -Al phase and Al-Si eutectic. Semisolid isothermal holding was carried at 570°C for 10 min. The semisolid microstructure suggested the materials are about 40% solid at this holding temperature. Both primary Si and primary Al co-existed as solid and they are uniformly distributed in the liquid. The primary Si structure changed little from the as-cast microstructure. The particles are of a similar size to the as-cast materials, and the morphology remained polyhedral but more rounded. The primary Al dendrites, on the other hand, underwent significant structural evolution. The dendritic morphology transformed to a globular one. Most of the Al-Si eutectic became liquid line spacing.

Microstructures of A390 cast at a lower cooling rate and without the AlP-refinement revealed relatively large Si crystals with a size of about 100 μm (Fig.3(a)). Cooling rate had a minimal refinement effect on the primary Si phase (Fig.3(c)). Pouring temperature had no significant influence on the size and distribution of the Si crystals and the primary Al phase. In its semisolid structure, the Si particles remained about 100 μm in size, and primary Al particles are fully globular. If the material was refined with AlP, the size of primary Si crystals is significantly reduced to about 40~50 μm (Fig.3(e)). Semisolid isothermal holding did not change the size and morphology of the primary Si size. The Si particles retained their original size, and the primary Al phase became very globular with a size of about 60 μm . If the AlP refinement is combined with a higher cooling rate then a very fine Si structure is produced with a particle size of less than 20 μm , being uniformly dispersed in the matrix (Fig.3(g)). In the semisolid structure, a very globular structure is produced with Si particles at about 20 μm and Al particles at about 60 μm (Fig.3(h)). The size of the Al particles is not affected significantly by cooling rate.

3.3 Aluminium wrought alloy 6063

Figure 4 shows the microstructures of Al wrought alloy 6063. The samples were cast at a superheat of 60°C. Figure 4(a) shows the microstructure of the non-grain-refined 6063 without wall crystals. The material had a very coarse columnar-grained structure with a transverse grain size of several millimetres. Its semisolid microstructure also shows a very coarse structure. Figure 4(b) shows the microstructure with wall crystals as an additional nucleation mechanism. The microstructure is significantly different from that without wall crystals. The grain structure is equiaxed rather than columnar, with a grain size of about 450 μm . After semisolid isothermal holding, the grains acquired a polygonal shape. Figure 4(c) shows the microstructure with grain refiner. Compared with the non-grain-refined material (Fig.4(a)), the grain structure is dramatically changed. The grain morphology is very globular with a grain size about 60 μm . Figure 4(d) shows its semisolid microstructures after isothermal holding at 645°C for 15 min. The grain morphology remained globular. The semisolid microstructure suggested that some

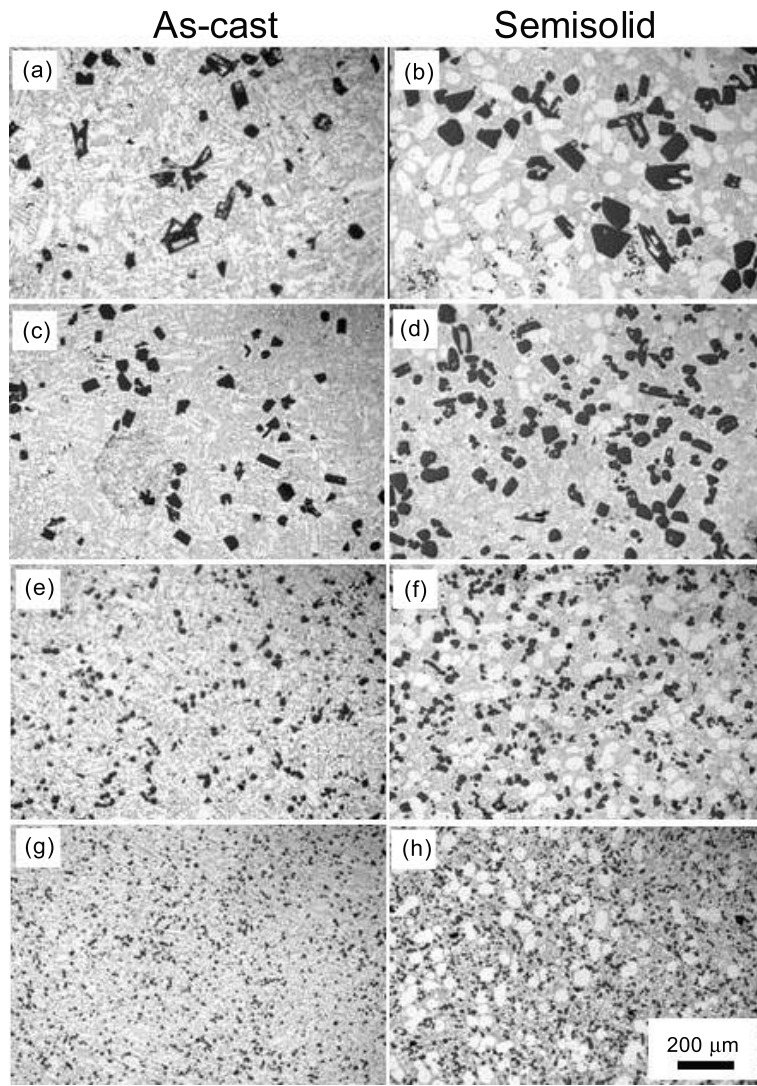


Fig.3 Microstructures of A390 alloy (a), (c), (e) and (g) are as-cast microstructures; (b), (d), (f) and (h) are semisolid microstructures after partial remelting and isothermal holding at 570°C for 10 min. The casting conditions are: (a) and (b) without AIP-refinement and low cooling rate; (c) and (d) without AIP-refinement and high cooling rate; (e) and (f) with AIP-refinement and low cooling rate; (g) and (h) 650°C with AIP-refinement and high cooling rate

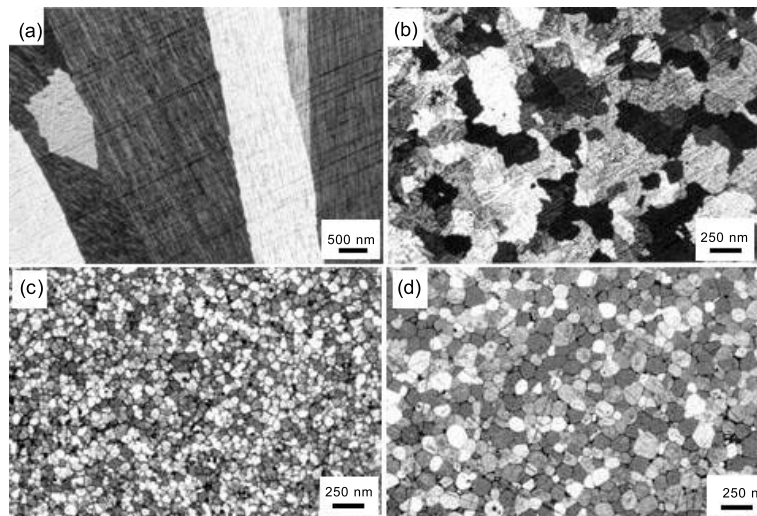


Fig.4 Microstructures of alloy 6063 cast at a superheat of 60°C (a) non-grain-refined without wall crystals, (b) non-grain-refined with wall crystals, (c) grain-refined without wall crystals, and (d) semisolid microstructure of (c) after isothermal holding at 645°C for 15 min

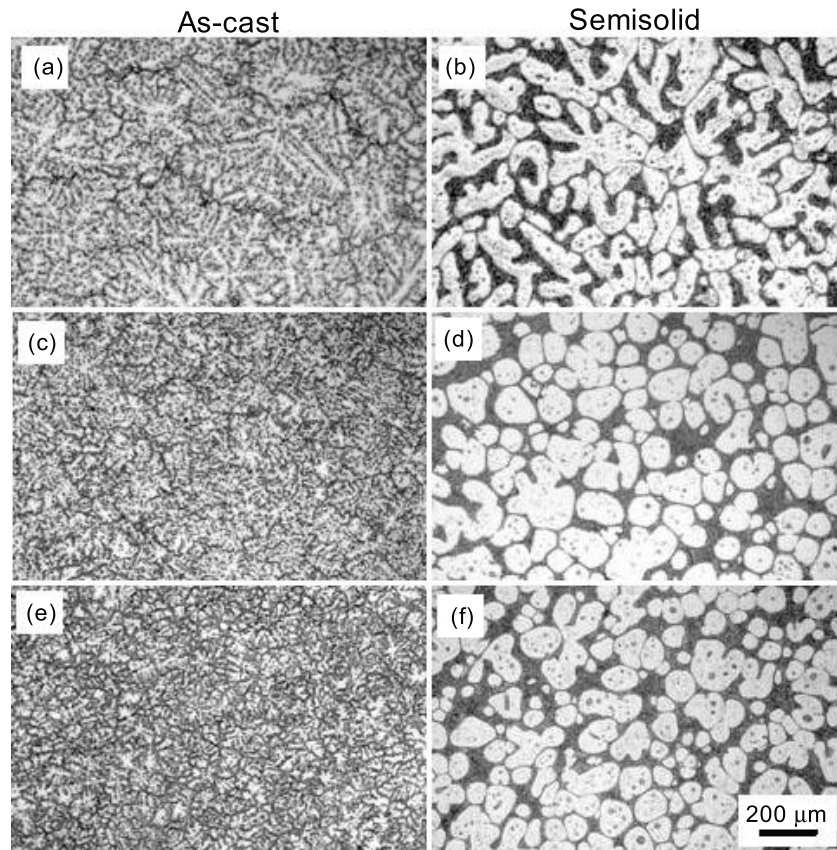


Fig.5 Microstructures of alloy AZ91 (a), (c) and (e) are as-cast microstructures; (b), (d) and (f) are semisolid microstructures after isothermal holding at 575°C for 10 min. The casting conditions are: (a) and (b) high superheat, low cooling rate; (c) and (d) low superheat, low cooling rate; and (e) and (f) low superheat, high cooling rate

coarsening of Al grains took place. It should be noted that in the Al wrought alloy the volume of eutectic is very small. To obtain sufficient liquid fraction for semisolid processing, some primary Al had to be remelted. It is difficult to control the fraction solid in the semisolid material just using control of the holding temperature.

3.4 Magnesium alloy AZ91

Figure 5 shows the as-cast microstructures and semisolid evolved microstructures of Mg alloy AZ91. Semisolid isothermal holding is carried out at 575°C for 10 min. Figure 5(a) shows the microstructure of the sample cast at higher superheat temperature (125°C). It consisted of primary α -Mg phase and divorced eutectic β -phase ($\text{Mg}_{17}\text{Al}_{12}$). The primary Mg phase is of dendritic morphology with a grain size of 360 μm . After partial remelting the primary phase became very irregular, and grains appeared separate from each other (Fig.5(b)). The structure is not suitable for semisolid processing. Figure 5(c) shows the microstructure poured at a lower superheat (10°C). The primary Mg phase is still of a dendritic morphology but the dendrites became much less distinct and the grain size is reduced to about 70 μm . After the semisolid remelting, the microstructure became very globular with a particle size of about 100 μm (Fig.5(d)). It seemed to be an ideal structure for semisolid processing. Figure 5(e) shows the mi-

crostructure from the same superheat but at a higher cooling rate. The higher cooling rate resulted in a more rosette-like structure but the grain size remained similar. The semisolid structure is also similar to the low cooling rate sample, as shown in Fig.5(f).

4. Discussion

In the controlled nucleation method, the key points are copious nucleation, nuclei survival and suppression of dendritic growth. The main sources of grain nuclei during solidification are the grain refiner, which supplies heterogeneous particles, and detached wall crystals. Constitutional undercooling controls the activation of nuclei and is strongly affected by the solute content in the alloy, which will also influence the consequent grain growth mode. However an increased alloy content, which promotes constitutional undercooling, also promotes a more dendritic morphology. Grain refiner addition can be very effective for some alloys, but less successful in others, such as Al contented Mg alloys. Wall crystals provide another nucleation source that can be exploited. Wall crystals are the crystals that are nucleated during pouring, at or near the relatively cold mould wall. They are then carried to the bulk of the melt by fluid flows within the melt and serve as very effective nuclei. Furthermore, dendrite arm root may melt off, providing multiple nuclei from a single original source. If a larger number of wall crystals are generated, transported and

survived in the melt, then a finer grain structure with less dendritic morphology will be produced. Parameters such as pouring temperature, cooling rate and other mould thermal conditions can be controlled to promote these nucleation mechanisms and achieve the maximum nucleation density. It is possible to control the solidification condition for grain nucleation and growth to produce a starting microstructure that is fine-grained and less dendritic. Followed by a partial remelting and isothermal holding, which is usually employed prior to the semisolid casting, such microstructures can evolve to a globular structure. The effects of these controlling parameters on the formation of semisolid structure and the contribution of each nucleation mechanism are different for the different alloy systems. These experiments have shown that there are substantial differences between alloy systems in the relative contributions from the different mechanisms.

In the hypoeutectic alloy A356, wall crystals make a significant contribution to the grain structure formation, as shown in Fig.1. The low pouring temperature is beneficial to the wall crystal mechanism as the survival rate of the wall crystals would be much higher at a low melt temperature. Grain refinement is very effective in high temperature pouring, however without the wall crystal mechanism, it is still not sufficient to generate a suitable semisolid structure (Fig.1(d)). The combination of wall crystals and grain refinement results in a fine-grained rosette-like structure, which evolves to a globular semisolid structure with a particle size of 100 μm after remelting (Fig.1(h)).

In the hypereutectic alloy A390, the coexistence of primary Si and primary Al is important for the formation of semisolid structure because the amount of primary Si by itself is insufficient for semisolid forming. The microstructure of the primary Si structure is effectively controlled by AlP refinement and the refinement due to solidification rate. Wall crystals does not assist in improving the microstructure of A390. A uniform semisolid structure is produced with Si particles at a size of 20 μm and Al particles at a size of 60 μm (Fig.2(h)).

In the Al wrought alloy 6063, grain refinement is very effective in generating the semisolid structure. It changes a very coarse columnar structure to extremely fine-grained structure with a grain size of only 50 μm . The wall crystal mechanism is also effective, producing, in the absence of grain refiner, a globular structure with a particle size of 300 μm .

In Mg alloy AZ91, the wall crystal mechanism promoted by low temperature pouring is effective. Cooling rate is less effective, at least in the low superheat material, at producing a finer structure, however it leads to a more globular grain morphology. A very globular structure with a particle size of about 100 μm is produced (Figs 5(d) and (f)).

5. Conclusions

(1) The controlled nucleation method uses the con-

trol of nucleation and grain growth to produce fine-grained and less dendritic structures. Such structures can evolve to a globular semisolid structure after semisolid remelting, ready for semisolid forming.

(2) Microstructures suitable for semisolid processing can be produced for each of the alloys studied, but the dominant nucleation mechanism varies between the alloys.

(3) The main mechanisms responsible for formation and activation of grain nuclei are wall crystal formation, grain refinement and constitutional undercooling. Solidification parameters such as pouring temperature, cooling rate and grain refiner addition can be controlled to maximise the nucleation density.

(4) In the hypoeutectic alloy A356, wall crystals and grain refiner have similar influences on the grain structure. In hypereutectic A390, AlP refinement and thermal refinement are effective, while wall crystals had negligible influence. In Al alloy 6063, Ti-B grain refinement is extremely effective, rather more so than the contribution from wall crystals. In Mg AZ91, the wall crystal formation mechanism is the most effective mechanism.

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