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Evolution of globular microstructures during processing of aluminium slurries

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Abstract: Slurry processing experiments were performed with AlSi7Mg0.6 to identify the globularization mechanisms. The melt sample water quenched slightly above the liquidus point is predominantly dendritic while that cooled into the semi-solid temperature range internally via stirring the melt with a rotating cylindrical block of the alloy itself becomes fully globular. The globules are much smaller when internal cooling and stirring are employed longer to achieve higher solid fractions before casting. Coarse dendrite fragments of various sizes are revealed, in the case of stirring after an initial fraction of solid is first formed without the benefit of additional internal cooling.

Key words: Al alloys; AlSi7Mg0.6 alloy; solidification; microstructure; internal cooling; stirring; solid fraction

1 Introduction

Rheocasting, an attractive semi-solid processing route, uses slurry-on-demand and has become popular among aluminium foundries which rely on the semi-solid processing route for the manufacture of cost-efficient high integrity structural parts [1−3]. This innovative technology requires slurries with a globular solid fraction. Mechanical stirring is the first process employed to produce slurries for rheocasting [4] and its favourable impact on semi-solid features has been investigated for over 30 years [5−10]. Several other processes to produce rheocasting feedstock with attractive features have been developed since then [11−16].

The early account of non-dendritic morphologies based on dendrite fragmentation under forced convection is indeed a plausible one [1, 3, 4, 17]. Other mechanisms of dendrite fragmentation have also been proposed [18]. The broken dendrite arms then undergo a coarsening process to provide the globular structures. However, recent investigations provide evidence that the non-dendritic structure is more likely to originate from natural nucleation of the primary phase and the subsequent growth in the stirred melt, rather than via dendrite fragmentation [19−24]. Slurry processing experiments were performed in the present work to identify the mechanisms responsible for globular structures in aluminium slurries for rheocasting. Internal cooling and stirring, the key elements of the recent slurry production methods [12,14−16], were employed to understand their impact on the evolution of slurry features.

2 Experimental

The hypoeutectic AlSi7Mg0.6 alloy was melted in a silicon carbide crucible and then brought to and held at 620 °C, approximately 10 °C above the liquidus point, for 60 min for temperature equilibration. The hot ceramic mould with the molten alloy was then withdrawn from the furnace to produce slurries. The first set of slurries were obtained by stirring the melt with a cylindrical block of the alloy itself to cool the melt into the semi-solid temperature range [15,16] (Figs. 1(a−c)). The slurry samples were finally quenched in water once they cooled to predetermined semi-solid temperatures. These temperatures were estimated from the solid fraction (F_s) vs temperature curves obtained from the heat flow vs temperature data recorded during solidification in a differential scanning calorimetry (DSC) cell (Fig. 2). The second set of slurry samples were produced by first cooling the melt just below the liquidus point on its own to initiate solidification and then stirring the slurries thus obtained with a graphite rod until they were finally quenched in water at exactly the same solid fractions as

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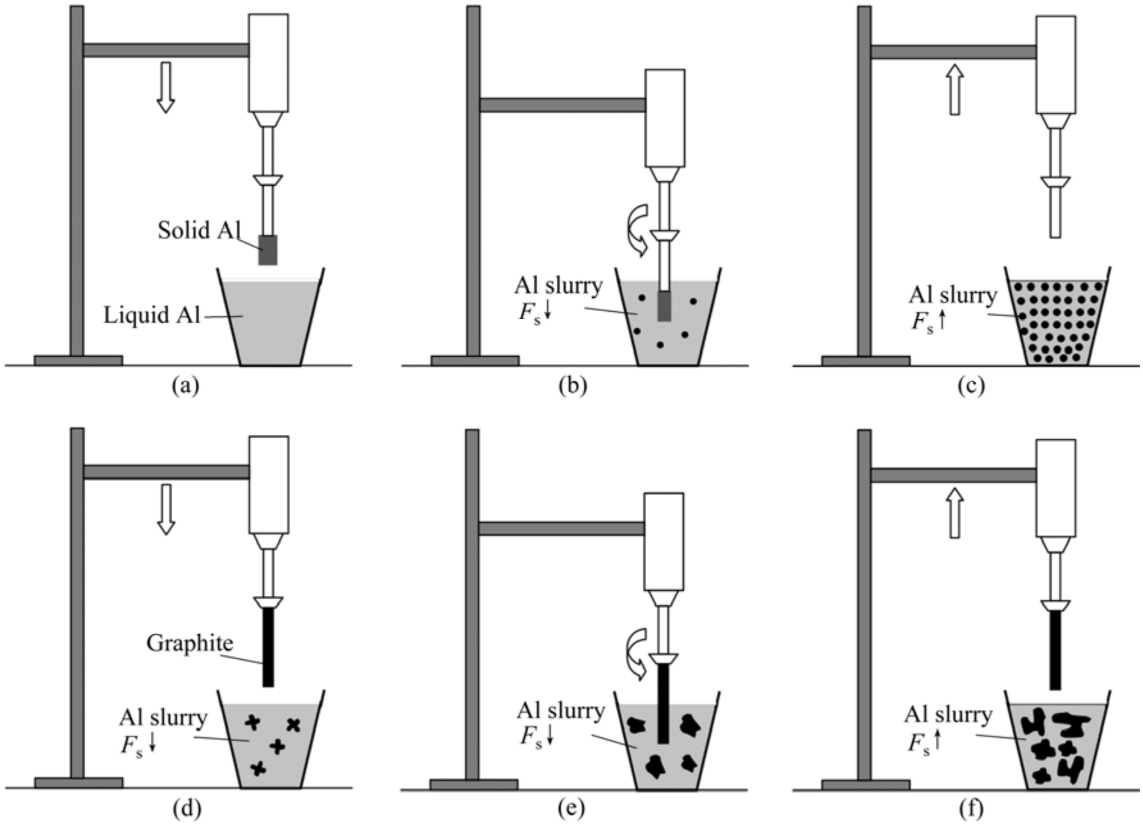

Fig. 1 Schematic illustration of slurry processing routes employed in the present work: (a), (b), (c) Slurry processing with internal cooling and stirring until slurry cools to semi-solid temperature range; (d), (e), (f) Slurry processing with stirring after melt is first cooled into semi-solid temperature range where they were finally quenched in water

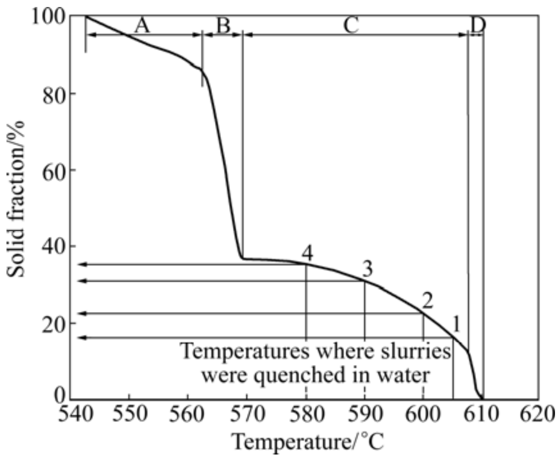

Fig. 2 Solid fraction vs temperature curve of A357 alloy obtained from heat flow vs temperature data recorded during cooling from molten state (Points 1 to 4 mark temperatures and solid fractions where the slurry samples were quenched in water): A—F_s is too high for semi-solid route; B—Sensitivity of F_s to T is too high, dF_s/dT>0.09 K^{-1}; C—Suitable F_s−T range, dF_s/dT~0.002 K^{-1}; D—F_s is too low to take advantage of semi-solid practice, dF_s/dT~0.09 K^{-1}

those in the first set of slurry samples (Figs. 1(d−f)). The water-quenched samples were prepared with standard metallographic procedures and were investigated with an optical microscope to find out about the evolution of the primary solid with time. A commercial image analysis software was employed to estimate the size and the shape factor of the α(A) globules.

3 Results and discussion

The melt sample quenched at 620 °C, just above the liquidus point, is predominantly dendritic. Typical features of conventionally cast hypoeutectic aluminium alloys with fine α(A) dendrites and an interdendritic network of the Al−Si eutectic phase are evident (Fig. 3). The melt samples cooled into the semi-solid temperature range internally via stirring the melt with a cylindrical block of the alloy itself (Fig. 4(a)), on the other hand, are predominantly globular with occasional fine dendritic clusters that have formed via rapid solidification of the liquid fraction upon quenching the slurry (Figs. 4(b) and (c)). Nearly equiaxed, compact α-Al grains start to form; dendritic features become less frequent and dendritic clusters almost vanish with decreasing slurry temperatures (Figs. 4(b), (c) and (d)). In the meantime, α(A) rosettes dominate, are refined and become increasingly more globular. Finally, the microstructure of the slurry so processed and quenched from 580 °C, with an estimated solid fraction of 35%, is entirely globular (Fig. 4(e)). The average size of the α(A) grains is

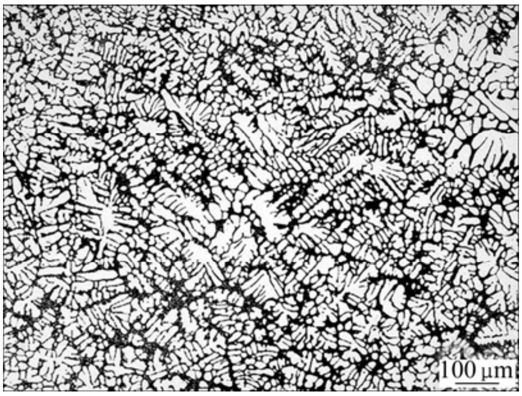

Fig. 3 Microstructure of water-quenched melt

approximately 30 μm, much smaller than the globule size obtained with this alloy in the thixo route [25].

The marked improvement in grain size when internal cooling and stirring are employed until casting without allowing the slurry to cool on its own in a quiescent state [15] is demonstrated in Fig. 5. α(A) grains, that coarsen when the slurries are allowed to cool on their own further in the semi-solid temperature range, are instead refined when cooled internally with concurrent stirring to higher solid fractions before they are quenched [15]. The globularization process is accelerated, namely, globular structures are obtained earlier at higher semi-solid temperatures and the α(A) globules are much smaller when internal cooling with stirring is extended to achieve higher solid fractions in the slurry before the water-quenching.

The slurry features are markedly different in the case of stirring after an initial fraction of solid is first formed, without the benefit of internal cooling (Fig. 6(a)). α(A) dendrites which have formed when the melt was first cooled into the semi-solid temperature range to kick off an initial amount of solidification are all fragmented (Fig. 6(b)). A considerable portion of the slurry so processed and quenched from 600 °C is made up of dendritic fragments of various sizes (Fig. 6(c)). The slurry sample cooled further to achieve higher solid fractions before water-quenching exhibits coarse α(A)

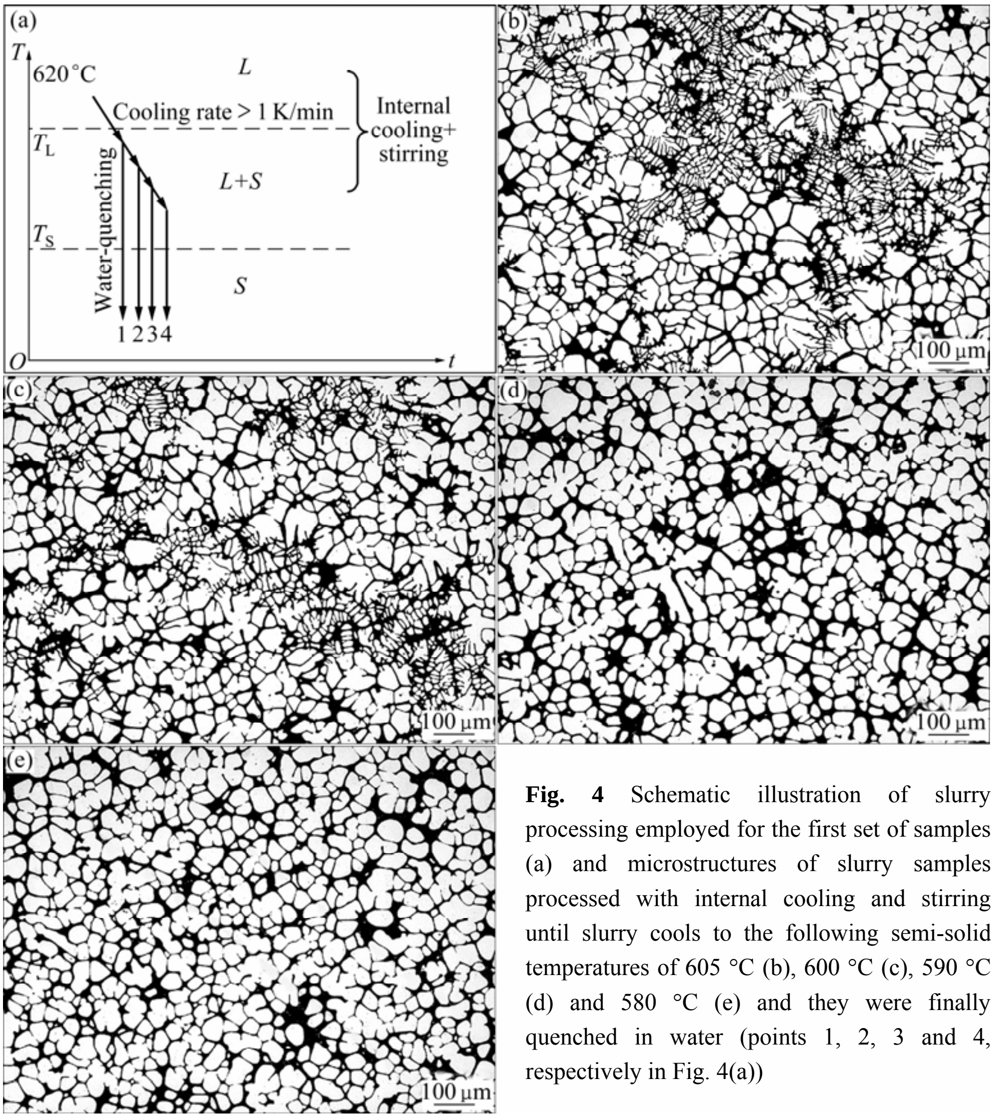

Fig. 4 Schematic illustration of slurry processing employed for the first set of samples (a) and microstructures of slurry samples processed with internal cooling and stirring until slurry cools to the following semi-solid temperatures of 605 °C (b), 600 °C (c), 590 °C (d) and 580 °C (e) and they were finally quenched in water (points 1, 2, 3 and 4, respectively in Fig. 4(a))

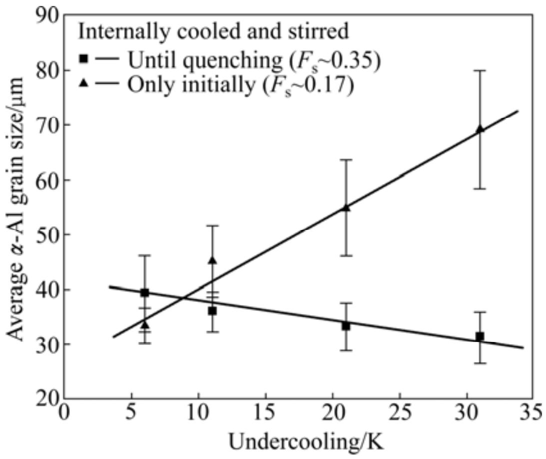

Fig. 5 Change in α(A) grain size of slurry processed with immersion cooling and concurrent stirring to a solid fraction of approximately 17% and then cooled further on its own to a solid fraction of 35%, and of slurry processed in same fashion but to a higher solid fraction of 35%

grains with odd morphologies (Fig. 6(d)). Both the size and the shape of the α(A) grains are markedly different with respect to those observed in the slurries processed with internal cooling and stirring until casting (Fig. 7). These features suggest the welding of the dendrite arms, a coarsening mechanism very likely to dominate under these circumstances owing to a favourable crystallographic match between the neighbouring dendrite arms. Coarsening via coalescence is responsible for the heterogeneity in morphological features.

The present results show that the globular features essential for rheocasting cannot be obtained via dendritic fragmentation. The globular structures obtained with stirring and internal cooling to achieve partial solidification in the first set of slurry samples, cannot be accounted for by dendritic fragmentation that was held responsible for this feature in previous investigations [1,4,17]. The globular α(A) grains in Fig. 4(e) reveal no evidence of entrapped liquid that would imply an earlier transient dendritic structure. Dendrite fragments are not observed either. Dendritic fragmentation is noted only when melt stirring is employed after an initial amount of solidification is first obtained, as observed in the second set of slurry samples (Fig. 6).

Alpha-Al grains that nucleate when the temperature of the melt drops below the liquidus point find themselves in a uniform temperature and solute distribution field owing to the melt convection provided by the stirring action. Stirring allows the slurry to cool uniformly, promoting bulk nucleation and uniform dispersion of nuclei across the melt due to melt convection while the cylindrical solid block serves as a heat sink and provides rapid cooling via thermal exchange. The insulated hot crucible discourages rapid heat loss to the surroundings and also helps to maintain uniform temperature profiles. As the melt chemistry

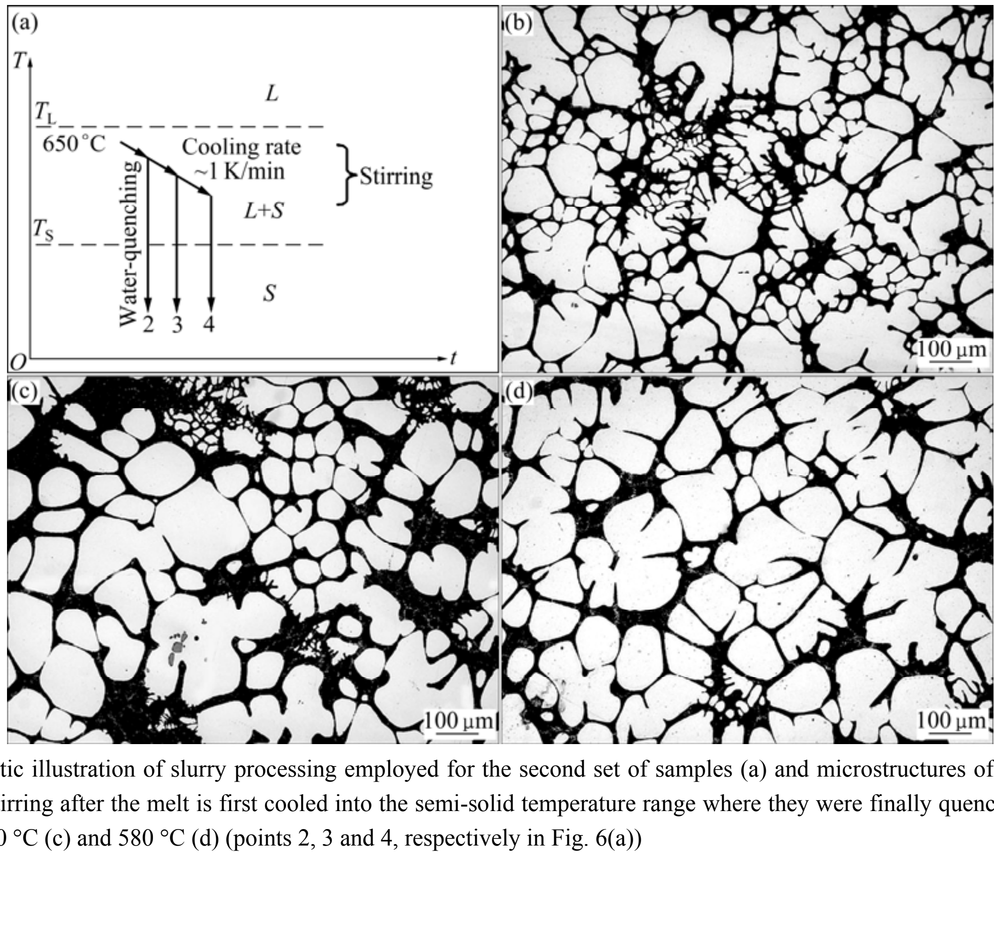

Fig. 6 Schematic illustration of slurry processing employed for the second set of samples (a) and microstructures of slurry samples produced by stirring after the melt is first cooled into the semi-solid temperature range where they were finally quenched in water of 600 °C (b), 590 °C (c) and 580 °C (d) (points 2, 3 and 4, respectively in Fig. 6(a))

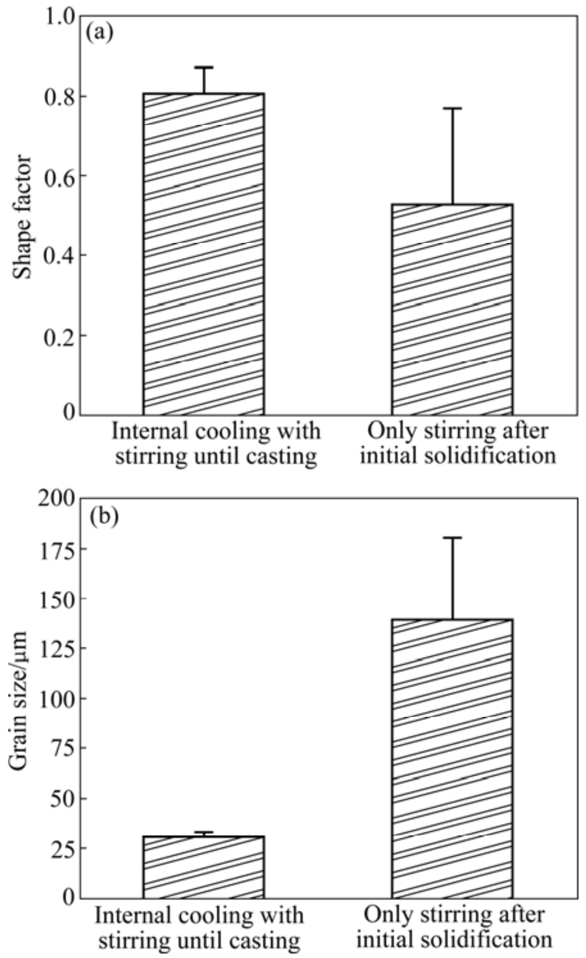

Fig. 7 Average shape factor (a) and average grain size (b) of α(A) grains in slurries processed with stirring after partial solidification and with internal cooling and stirring until casting

along the solid−liquid interface is relatively uniform, constitutional undercooling is highly unlikely; the interface stability is maintained; perturbations are avoided and the solid grows with a planar front producing compact structures. This suggests that isotropic planar growth is responsible for the globular grains, rather than dendrite fragmentation followed by spheroidization when stirring and internal cooling are employed together. Once nucleated, the *α(A)* grains remain fairly equiaxed throughout the semi-solid cooling. They become increasingly more globular with time in the semi-solid temperature range so as to minimize the surface energy.

Some investigators have reported an increase in the α(A) grain size with the increase in shearing time and have offered the Ostwald ripening mechanism for the coarsening of α(A) grains through dissolution of the smaller ones at the expense of coarse grains [26]. This is in quite contrast to the results presented in the present work. α(A) grains were observed to coarsen only when the stirring process was terminated. They were refined, however, when internal cooling with stirring is extended to achieve higher solid fractions before quenching (Fig. 5). The decrease in size, i.e. the increase in number of globular α(A) grains at lower slurry temperatures implies increasing nucleation activity via increasing nucleation rates when internal cooling with stirring is extended. Besides, the nucleation occurs over a wider temperature range when the slurry samples are quenched from lower temperatures. The impact of heterogeneous nucleation throughout the melt and of forced convection on the primary solidification features is evident and confirms the earlier reports [10, 13, 27].

The predominance of globular features in the entirety of the water-quenched slurry samples, in spite of a maximum solid fraction of 35% at the time of water-quench, seems to imply that the dendritic solidification is suppressed once a critical fraction of globular solid is first formed. Solidification of the remaining liquid apparently occurs, at least in part, through the planar growth of the α(A) globules, which have formed during slurry processing before quenching. A fully globular microstructure is then possible even when the melt is stirred only briefly and much of the primary solidification occurs in a quiescent state, providing that a critical population of heterogeneous nuclei is first made available throughout the entire volume of the melt. In fact, this is the hypothesis behind the semi-solid rheocasting process [12].

4 Conclusions

The melt sample water quenched slightly above the liquidus point is predominantly dendritic. A marked improvement in cast features is noted when the melt is processed so as to provide both melt convection and internal cooling. The melt samples cooled into the semi-solid temperature range internally via stirring the melt with a rotating cylindrical block of the alloy itself become fully globular. The globules are much smaller when internal cooling and stirring actions are employed longer to achieve higher solid fractions before casting. The slurry features are markedly different, with coarsened dendrite fragments of various sizes, in the case of stirring after an initial fraction of solid is first formed, without the benefit of additional internal cooling. Coarsening via coalescence is responsible for the heterogeneity in morphological features.

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铝合金半固态浆料中球形颗粒的演变

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摘　要：用 AlSi7Mg0.6 半固态浆料进行实验，研究球形颗粒的演变机理。在略高于液相线温度下水淬的样品中结晶相主要为枝晶，而采用旋转圆筒的方式来搅拌熔体使其冷却到半固态温度范围内时样品的结晶相完全球化。在铸造前进行较长时间的内冷却和搅拌，可以得到较高的固相分数。不同大小的枝晶碎片的存在表明，在搅拌过程中，初始的固相分数形成后不再受冷却的影响。

关键词：铝合金；AlSi7Mg0.6 合金；凝固；微观结构；内部冷却；搅拌；固相分数

(Edited by Hua YANG)