Thixotropic Structure Formation in Hypoeutectic Al-Si Alloys by Controlled Nucleation

H. Wang¹, Z.L. Ning¹, X.D. Yao¹, C.J. Davidson², D. St.John³

¹Division of Materials, School of Engineering, the University of Queensland, Brisbane, Australia
²CSIRO Manufacturing & Infrastructure Technology, PO Box 883, Kenmore, QLD 4069 Australia
³CRC for Cast Metals Manufacturing (CAST), The University of Queensland, Brisbane, Australia

Keywords: semisolid casting, thixotropic structure, Al-Si alloys

Abstract

Semisolid metal forming requires feedstock material with a fine-grained, non-dendritic structure to achieve thixotropic properties. In this study, a Controlled Nucleation method without stirring was used for the formation of thixotropic structures. Hypoeutectic Al-Si alloy was cast into a permanent mould using different pouring temperatures, with and without gauze. As the pouring temperature increased, the grain size increased and the tendency to form columnar grains also increased. Gauze experiments show that the mechanism of the “wall crystals” plays a very important role at low pouring temperatures. The survival rate of the wall crystals determines the final thixotropic structure including both grain size and morphology up to 675°C pouring temperature, while other nucleation mechanisms might operate at higher temperatures.

1. Introduction

Semisolid metal forming (SSMF) is an emerging new technology, in which metal alloy is processed to near net-shape at a temperature above its solidus but below liquidus temperature [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. These advantages can only be achieved when the SSMF feedstock material has a non-dendritic grain structure. When the material is reheated to its semisolid state, it can achieve a 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 [4]. Several methods have been developed to produce these feedstock materials with such thixotropic structure for SSMF, such as mechanical stirring processes [5-6], electromagnetic stirring [7], SIMA [8] and melt mixing [9].

The Controlled Nucleation method is an alternative to the stirred processes for producing a suitable starting microstructure for semisolid forming [10-12]. By controlling certain solidification conditions, it is possible to adjust grain nucleation and growth to produce a starting microstructure that is fine-grained and less dendritic. Such microstructure can
evolve to a globular structure after partial remelting and isothermal holding, which is usually employed prior to the semisolid casting. In this study, the effect of melt pouring temperature on the grain structure formation and evolution of the feedstock materials of hypoeutectic Al-Si alloy were investigated. The role of “wall crystals” in the formation of thixotropic structure will be discussed.

2. Experimental Methods

The alloy used in this study was commercial hypoeutectic Al-Si alloy A356 and its chemical composition is given in Table 1. The ingot was melted in an induction furnace. The melt was maintained at a temperature of 750°C and degassed for 10 minutes with argon gas through a graphite lance. No grain refining or modifying addition was made. To investigate the effects of pouring temperatures on grain formation, the melt was cast into a cylindrical steel mould with a cylindric cavity 50 mm in diameter and 70 mm in depth. A stainless steel gauze cylindrical cup 30 mm in diameter was suspended in the centre of the cylindrical mould cavity with approximately 10 mm gap at the bottom. The gauze was made from stainless steel wires of 0.28 mm diameter with a hole aperture 0.56 mm square, serving as an effective mechanical barrier to the melt movement between inside and outside of the gauze. The mould preheat temperature was 200°C.

Table 1: Chemical composition of cast alloy (wt %)

<table>
<thead>
<tr>
<th></th>
<th>Si</th>
<th>Mg</th>
<th>Ti</th>
<th>Sr</th>
<th>Fe</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>6.62</td>
<td>0.36</td>
<td>0.016</td>
<td>0.002</td>
<td>0.04</td>
<td>Balance</td>
</tr>
</tbody>
</table>

As-cast samples were sectioned and polished. For macrostructural observation, polished samples were deeply etched by a solution of 120 mL hydrochloric acid, 50 mL nitric acid, 10 mL hydrofluoric acid and 10 mL water. For grain size measurement, the samples were given a metallographic polish. They were then anodised in a solution of 760 mL water and 40 mL fluoroboric acid at 30 volts for 2 minutes. Grain size was measured using the linear intercept method outlined in section E112-88 of ASTM standards by means of an optical microscope in a polarised light mode.

3. Experimental Results

3.1 The effect of pouring temperature on as-cast grain size

Figure 1 shows the as-cast macrostructures of castings poured at 625, 650, 675, 700 and 725°C, respectively (liquidus temperature for the alloy is 618°C). It shows the effect of the pouring temperature on the grain structure. As the pouring temperature increases, there is a significant change in both the grain morphology and grain size. The samples with pouring temperatures of 625 and 650°C exhibit a fine grained and fully equiaxed structure. For the high pouring temperature samples, the equiaxed grains become coarser and a columnar zone forms near the mould wall. The length of the columnar zone increases with pouring temperature. The average grain sizes of the as-cast microstructures from different pouring temperatures were measured from the anodised samples and are plotted in Figure 2. It shows the strong dependence of grain size on the pouring temperature. The pouring temperature affects the grain size significantly when the pouring temperature is above 650°C. The grain size increases from about 200 µm at a pouring temperature of 650°C to 1000 µm at 725°C. However, at pouring temperatures below 650°C, the effect of the
pouring temperature on grain size is much less significant. The difference in grain size between the samples poured at 625 and 650°C is only about 20 µm.

![Macrostructures of A356 alloy poured at different temperatures](image)

Figure 1: Macrostructures of A356 alloy poured at: (a) 625°C; (b) 650°C; (c) 675°C; (d) 700°C and (e) 725°C. Each image is half of the casting. The wall is on the left side of each image and the centre-line is on the right.

![Grain size vs. pouring temperature](image)

Figure 2: Grain size as a function of pouring temperature.

3.2 Effect of Gauze on Grain Formation

Figure 3 shows the macrostructures from the gauze experiment. The melt was poured inside the gauze and the pouring temperatures were 625, 650, 675, 700 and 725°C, respectively. The grain structure outside the gauze varies from fine equiaxed at low pouring temperatures, to coarse equiaxed, then fully columnar grains for a pouring temperature of 725°C. Inside the gauze, the grain structure remains equiaxed at all pouring temperatures, but small grain size is only obtained at a pouring temperature of 625°C and then increases dramatically with increased pouring temperature. Figure 4 shows that the measured grain size increases with pouring temperature both inside and outside the gauze. For the columnar structure, the width of columnar grains was used as grain size.
4. Discussion

There are a number of nucleation mechanisms that could be responsible for grain formation during solidification [13]. Whether a particular mechanism becomes operative depends on the alloy system and solidification conditions. For experiments where material is prepared by controlled solidification, the mechanisms of “wall crystals” and the constitutional undercooling should be the most important. 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 [14]. To investigate the relative contribution of wall crystals to overall grain formation, gauze experiments are carried out. The gauze creates a physical barrier to the movement of crystals from the wall to the centre of the sample, while minimising heat flow. Chill crystals formed at or near the mould wall would be isolated in the region between the wall and the gauze. The gauze heats up to the temperature of the melt very quickly during pouring, owing to its low thermal mass. It also has a small surface area, hence, it is reasonable to assume that of creation of wall crystals on the gauze would be much less likely than on the mould, and the number of

Figure 3: Macrostructures of samples in gauze experiment at pouring temperatures of (a) 625°C; (b) 650°C; (c) 675°C; (d) 700°C and (e) 725°C. The wall is on the left side of each image and the centre-line is on the right.

Figure 4: The effect of pouring temperature on as-cast grain size in gauze experiments.
crystals created by gauze would be quite small. With the slight insulating effect of the gauze and the relatively high temperature inside the gauze, these crystals tend to be remelted and make a limited contribution to grain formation. Therefore, the mechanism of wall crystals for microstructure formation would be not operative inside the gauze. The gauze experiment can to a large extent separate the contribution to grain formation from wall crystals and crystals formed by constitutional undercooling [15].

As shown in Figure 3, the grains inside the gauze exhibited very coarse grains, while the grains outside the gauze were very fine, which indicated that wall crystals have a significant contribution to the microstructure formation under these conditions. The microstructure formation inside the gauze would be dependent on the heterogeneous nucleation on impurity particles, which have a much higher nucleation barrier than the wall crystals (indeed, the wall crystals are already nucleated crystals and the only barrier for the crystals becoming grains is the curvature undercooling that must be overcome for free growth). These heterogeneous nuclei give rise to a much lower density of effective nucleation, thus a much coarser grain structure is formed. Comparing the results between Figure 3 and Figure 1 at the 725°C pouring temperature, it is found that the columnar zone was much longer when the gauze was used than when it was not. A fully columnar zone between the gauze and the mould was obtained. For the sample with the gauze, this suggests either that the wall crystals could not become detached or that remelting of nuclei was more severe. Both possibilities could arise due to modification to flow patterns by the presence of the gauze. In the first case, the gauze might cause the flow against the wall to be less turbulent and vigorous, leading to fewer nuclei being formed. In the second, the gauze probably reduces or suppresses natural convection, which will reduce the number of detached nuclei that can be swept away from the wall.

It should be noted that the sample poured at a temperature of 625°C exhibited a very fine equiaxed grain structure inside the gauze. This means that there must be many crystals surviving in the liquid within the gauze. One possible reason is that the gauze itself forms a chill surface and that there is so little superheat that those nuclei formed upon pouring survive. However, the mechanisms remain unproven and require further study.

Once the wall crystals are formed, they will be carried to the bulk of the melt either by residual momentum flows immediately after filling or by thermosolutal convection. The survival rate of the wall crystals would be much higher at a low melt temperature. The possibility of crystal survival is dependent on the local thermal conditions and the degree of the constitutional undercooling in the liquid. Undercooling has contributions from both thermal and constitutional effects. If the local liquid temperature is higher than the liquidus temperature, the crystals will start to remelt at a melting rate that is determined by the rate at which the surrounding liquid can supply heat. The small crystals will disappear and the large ones will decrease their sizes with time. The rate of crystal remelting becomes much higher with an increase of pouring temperature because this reflects the degree of superheating left after pouring. Furthermore, small crystals are even unstable in an undercooled liquid if the degree of undercooling is less than the curvature undercooling. The kinetics of the crystal remelting should be further investigated. One hypothesis could be that the creation of the wall crystals is largely independent of the pouring temperature because nucleation of wall crystals is dominated by pouring method and mould wall conditions. If this were the case, then the final grain size will depend mainly on the survival of the wall crystals. This is because further nucleation from the impurity particles is largely limited, due to their relatively large nucleation barrier (as the crystals grow, the latent heat will be released to increase the local liquid temperature, which will decrease the local
undercooling so that the nucleation barrier will be hard to overcome). If Figures 1 and 2 are considered, then it is consistent with this hypothesis that the samples poured at 625 and 650°C did not return above liquidus after filling, and so retained all nuclei formed during pouring, leading to the smallest grain size. Higher pouring temperatures result in a gradual increase of grain size, implying a gradual increase of superheat and hence remelting. It would be expected that eventually a temperature would be reached where complete remelting occurred and growth is fully columnar. It is highly unlikely that nuclei could survive 20°C superheat and so the hypothesis cannot explain the moderate sized equiaxed grains at the highest pouring temperature. The columnar grains seen at 700 and 725°C pouring temperatures indicate that all wall crystals have been remelted. If they have been remelted in the coldest areas near the wall then survival in the centre is impossible. The equiaxed grains at the higher temperatures must derive from another nucleation mechanism.

5. Conclusions

The pouring temperature acts as an effective means in the Controlled Nucleation method to create thixotropic structure for semisolid feedstock. It has significant effects on both grain size and grain morphology. Fine equiaxed grains are obtained by using low temperature pouring. As the pouring temperature increases, the grain size increases, more rapidly above 650°C. Meanwhile, a columnar crystal zone is formed at high pouring temperatures. It is found by gauze experiments that wall crystals are the dominant source of nuclei at low pouring temperatures. The survival rate of these wall crystals will mainly determine the final grain size up to 675°C pouring temperature, while other nucleation mechanisms must operate at higher temperatures.

Acknowledgement

This work is supported by the Australian Research Council.

References