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Grain Refiner Fade in Aluminium Castings

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Abstract

The successful grain refinement of aluminium castings depends on several parameters. In particular, the time interval between the addition of the grain refiner and casting can significantly influence the level of grain refinement achieved. Extended holding times are often encountered in the foundry and, as a consequence, the grain refining action diminishes, or fades, with time. It has been shown that deviations from the optimum grain refiner level can result in the formation of defects in castings and the grain refiner level must therefore be maintained at an optimum level. There is a difference in density between the grain refining particles (~4.5g/cm^3) and the aluminium melt (~2.3g/cm^3) and it has been proposed that settling is the mechanism predominantly responsible for fade. However, Stokes law for particle settling does not accurately explain the observed rapid loss of grain refinement. Agglomeration of the grain refining particles is thus also believed to play an important role in grain refiner fade. Agglomeration of nucleant particles may be initiated as early as in the manufacture of the master alloy, and is likely accelerated by turbulence and the formation and entrainment of oxide films during casting. A comprehensive laboratory investigation has been undertaken to investigate the mechanism of fade by determining the rate at which it occurs, characterising the particles associated with the agglomerates and the contribution of various process parameters to the overall fade.

1. Introduction

Grain refinement of aluminium castings is common practice in the foundry. It is undertaken to achieve small equiaxed aluminium grains and the associated benefits of successful grain refinement have been well documented [1]. Although grain refinement is an integral part of the casting process, there are many accepted methods to achieve a fine equiaxed microstructure and grain refinement practices can vary greatly from foundry to foundry. One problem encountered in some foundries is that the melt is often held for long periods of time without agitation and this can lead to fade. Fade is the loss of grain refining potency with time and it is thought that settling of the grain refining particles is the main contributing factor to the mechanism of fade. Using Stokes settling theory, typical grain refining particles (1-5μm) should take more than 1000 minutes to settle a distance of one meter. However, it has been shown previously that Stokes settling theory does not account for the rate of fade that is observed in practice and therefore several other variables may contribute to the mechanism of fade [2]. Recent experiments have shown that fade occurs much faster than theory suggests and that a large proportion of the grain refining particles settle within 30 minutes after addition.

2. Experimental Procedures

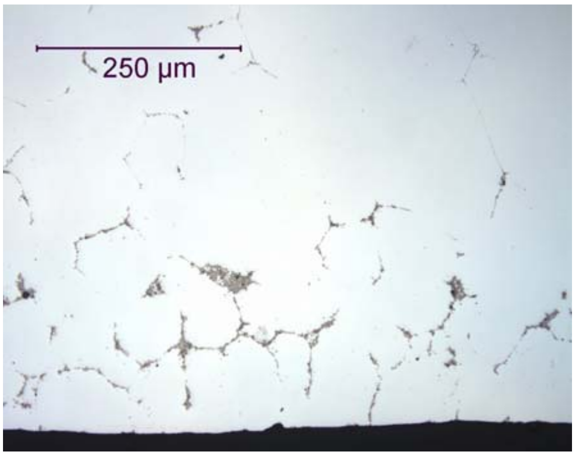
A series of experiments was designed to investigate the various mechanisms that contribute to fade. This paper will discuss one of the experiments which was undertaken in order to determine the rate of fade in a stagnant melt. The melt was prepared in an induction furnace by melting five kilograms of commercial purity aluminium in a clay graphite crucible. After melting the melt was transferred to a resistance furnace for holding. Once placed in the resistance furnace the melt temperature was allowed to stabilise at 720°C±5°C. Al-5Ti-1B grain refiner master alloy was added to give a concentration of 0.05wt%Ti and stirred for one minute. Graphite cups of 50mm diameter, 60mm height and 5mm wall thickness were filled from the grain refined melt and placed in the resistance furnace with the clay graphite crucible containing the remainder of the melt. Each cup remained in the furnace for different settling times. The first cup was removed after 5 minutes and was allowed to solidify in air on a fibrefrax mat and was covered with a fibrefrax lid. This process was repeated with the remainder of the cups being removed periodically after 10, 20, 30 and 60minutes of settling.

The samples were then analysed to determine the rate at which fade occurs. The castings were sectioned longitudinally with one half being used for microscopy and the second half for chemical analysis. The samples for microscopy were cut in to smaller pieces allowing them to be mounted in epoxy resin. The mounted samples were then polished and photographed using both optical and scanning electron microscopy. The microscopy also involved a surface area analysis to assist with determining the rate at which fade occurs. The scanning electron microscope was used to characterise the particles using a microprobe. The chemical analysis was also used to assist with the particle characterisation. Sections at 5mm intervals were taken from the bottom to the top and analysed using inductively coupled plasma atomic spectroscopy (ICP-AES).

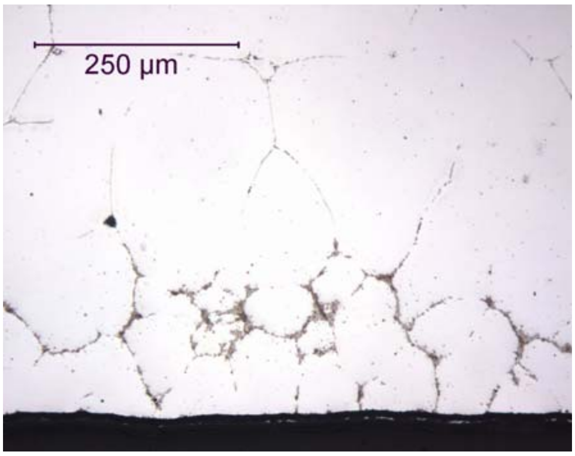
3. Results and Discussion

Upon observing the bottom layer it can be noted that the volume of the settled layer increases with time (Figure 1). Through measuring the boron content in the castings and in increments of 5mm from the bottom of the casting to the top it was found that the boron and titanium content were higher in the bottom 5mm of the sample than in the bulk of the casting for both the 5 minute and 60 minute samples. The boron and titanium level in the bottom of the 60 minute sample were higher than in the 5 minute sample which is to be expected as the longer settling time would permit more of the TiB_2 grain refining particles to settle to the bottom of the casting. The particles at the bottom of the melt were also analysed with a microprobe, and showed a high titanium count. These two analyses have been reported in previous work and are highlighted here to demonstrate that the settled particles are most likely to be grain refining particles [2].

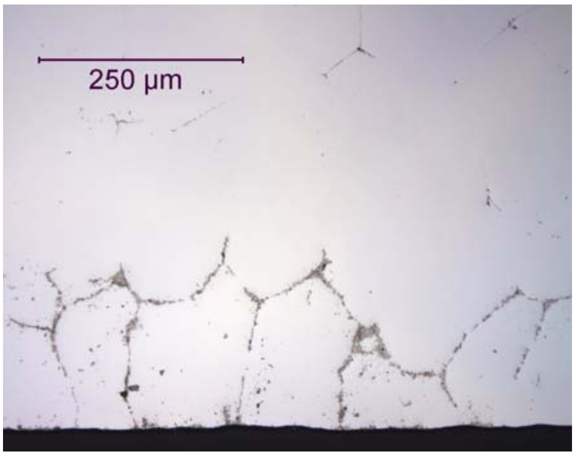
Micrographs of the bottom layer for the various settling times are shown in Figure 1. After only 5 minutes of settling, particles can be seen at the bottom of the casting. These particles appear to be concentrated in the regions between the grains and in particular at triple points. After 20 minutes of settling the grains at the bottom of the casting are already surrounded by a layer of the settled particles and the area of particles surrounding the grains increases again in the 30 minute sample. However, there appears to be no discernible difference between the 30 minute sample and the 60 minute sample, suggesting that a large proportion of the particles have already settled after 30 minutes.



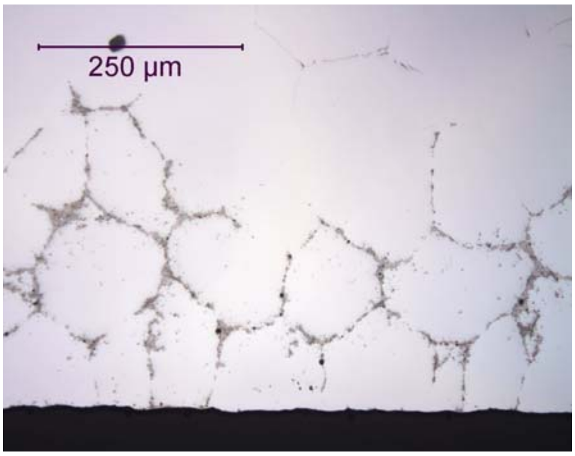
(a) 5 minutes



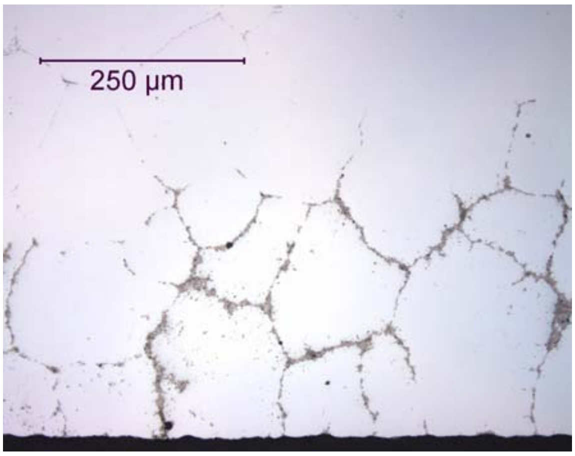
(b) 10 minutes



(d) 20 minutes



(c) 30 minutes



(e) 60 minutes

Figure 1: The settled layer for various settling times. A bottom layer is observed after only 5 minutes of settling (a) and the area of the settled layer appears to increase with time. However, by 30 minutes (c) the layer appears to have reached a maximum and this is confirmed when the micrograph from the 60 minute sample (e) is observed as the bottom layer does not appear to have increases in area.

The development of the layer at the bottom of the castings with time shown in Figure 1 suggests that particles settle to the bottom of the castings quite quickly. An interesting point is that the grains surrounded by the settled grain refiner in the 5 minute sample appear to be quite small. As more grain refiner settles the grain size in the settled layer increases with time and this becomes obvious when the 5 minute sample is compared to the 30 and 60 minute samples. This difference in grain size could be the result of agglomeration and consequent loss of nucleating potency. After addition of the master alloy and stirring for 1 minute the master alloy should be completely dissolved and many potent heterogeneous nucleating particles are distributed throughout the melt. After 5 minutes of settling, although small pockets of grain refiner can be seen between the grains at the bottom of the casting, it is possible that a large proportion of the settled particles are still potent enough to act as heterogeneous nucleation sites. Since the small amount of settling after 5 minutes has not adversely affected the potency of the nucleating particles, there are more potent particles at the bottom of the casting than in the bulk and as a result the grain size is smaller. As settling progresses more particles will descend to the bottom of the casting and collide with the already settled particles. It is likely that after colliding the particles form larger agglomerates and as a result their potency is reduced. Although there are more grain refining particles at the bottom of the melt after 30-60 minutes, they are unable to take part in the nucleation process due to their increased size and reduced potency and this is why the grain size at the bottom of the casting increases with time.

From the micrographs in Figure 1 two series of measurements were taken. Firstly the surface area fraction of the settled grain refining particles at the bottom of each sample was measured. Figure 2 shows the results of these measurements and it can be seen that the surface area of the settled particles appears to increase linearly with time for the first 30 minutes after which the area fraction remains constant. From Figure 2 it is immediately obvious that the majority of the grain refining particles have settled after only 30 minutes confirming the observation from the micrographs. As in the micrographs there is no significant change in the amount of grain refining particles at the bottom of the casting after 30 minutes.

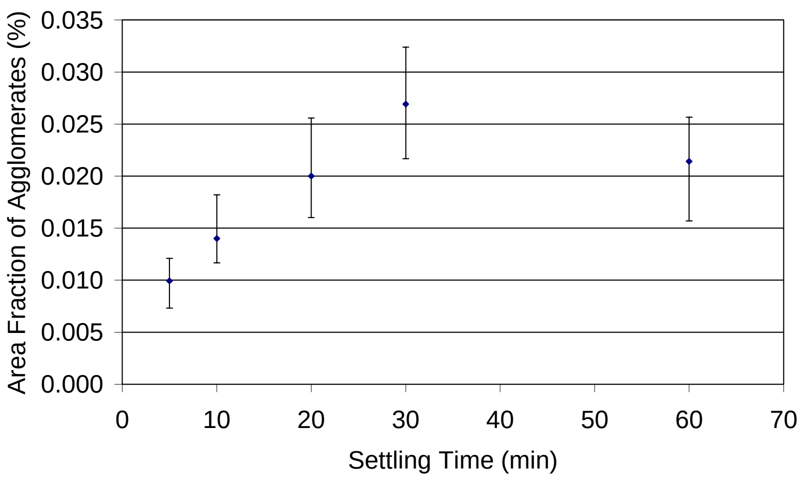


Figure 2: Area fraction of settled particles for the different settling times. Note the area fraction of the settled particles appears to increase linearly for the first 30 minutes of settling and after 30 minutes there is no increase in the amount of settled grain refining particles.

The second series of measurements conducted on the samples was to measure the height of the settled layer and determine its relationship with settling time. The results from these measurements can be observed in Figure 3 and confirm that the majority of settling has occurred after 30 minutes.

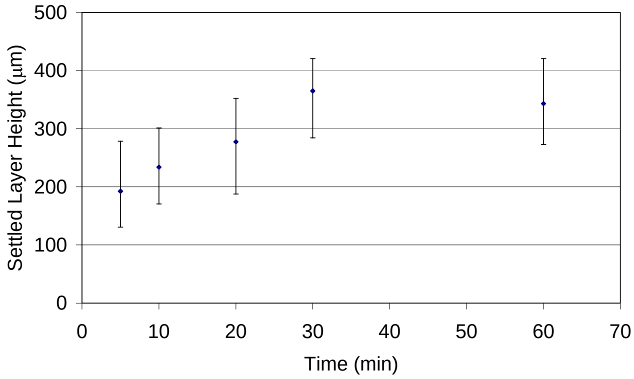


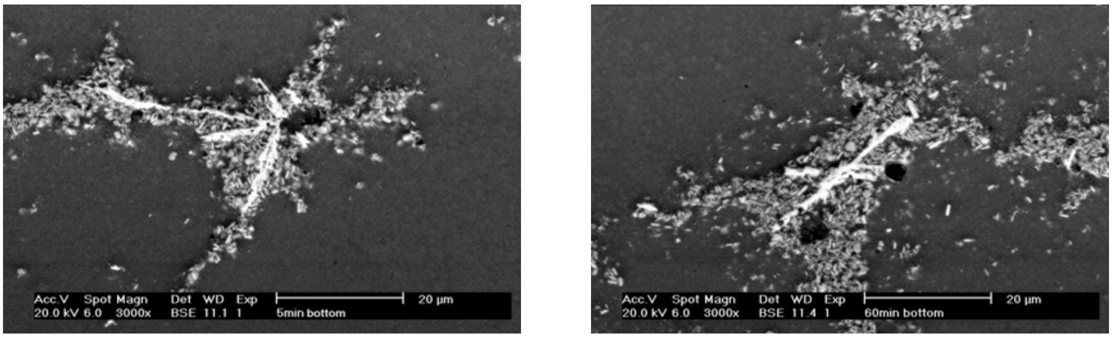
Figure 3: Height of the settled layer for the various settling times. Like the area fraction measurements the height of the settled layer appears to increase linearly for the first 30 minutes and beyond 30 minutes remains constant.

From Figures 1, 2 and 3 it can be inferred that the majority of particles have settled after 30 minutes and there is minimal settling after this time. In contrast to the rate of settling described by Stokes theory the settling observed in the above experiments is much faster. It is unlikely that the individual grain refining particles would settle in 30 minutes, but it is more likely that the particles could agglomerate and form larger particles that would settle much faster than the individual particles. Several possible mechanisms that could contribute to agglomeration have been previously outlined [2]. Of the possible mechanisms that can contribute to agglomeration, particle collision is likely to play a significant role in the settling of grain refining particles in the experiments described in this paper. Stokes theory suggests that larger particles will settle faster than smaller particles. With this being the case, when two particles collide they will settle faster than an individual particle. The larger particle can then potentially catch the smaller, slower settling particles and collide forming a larger agglomerate. This snow ball effect of particles becoming bigger and bigger as they catch slower settling particles could be the reason the rate of fade is so much faster than described by Stokes theory.

Typical agglomerates from both the 5 minute sample and 60 minute sample are shown in Microprobe work has shown these agglomerates to contain titanium and iron. The lighter needle like phase is most likely to be an iron intermetallic and the exterior of the agglomerates is made up of many individual particles and is likely to be grain refining particles that have agglomerated during settling. However, it can not be concluded from this work that the particles have agglomerated during settling and it is quite possible that they have been pushed together by the solid liquid interface in the final stages of solidification. It is the aim of future work to develop a greater understanding of the mechanisms that contribute to agglomeration and their impact on fade.

4. Conclusions

This work has shown that fade occurs much faster than initially thought. Stokes settling theory does not account for the observations of the above experimental work as a large proportion of fade occurs with in 30 minutes. After addition and dissolution of the grain refining master alloy the way in which the grain refining particles interact with each other will have a significant impact on the rate of settling. Although this experimental work can not conclusively show that agglomeration is the main mechanism contributing to the observed rapid settling of grain refining particles, agglomerates were observed in the castings and further experimental work is aimed at determining their role in the mechanism of fade.



(a) (b)

Figure 4: Agglomerates observed at the bottom of the casting after 5 minutes (a) and 60 minutes (b).

References

[1] D.G. McCartney, Int. Mater. Rev., 34, 247-260, 1989.
[2] P.L. Schaffer, A.K. Dahle, and J.W. Zindel, Light Metals 2004, Minerals, Metals and Materials Society, 2004.