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Influence of fading and stirring on the performance of the AlTi5B1 and AlTi3C0.15 grain refiners in an Al-Fe alloy

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Abstract

An Al-Fe alloy was in one case grain refined with the AlTi5B1 grain refiner and in the other with the AlTi5B1 and AlTi3C0.15 grain refiners. Both melts were stirred and the samples were cast two, fifteen and twenty-five minutes after the addition of the grain refiners. The grain size increase with increasing holding time was almost negligible in the case of the addition of both grain refiners. A much larger increase was found in the case of the addition of just the AlTi5B1 grain refiner, indicating the fading of the effectiveness the AlTi5B1 grain refiner and the effectiveness of the AlTi3C0.15 grain refiner in the case when the melt was grain refined with both grain refiners. The result suggests that the use of both grain refiners allows the possibility to control the fading performance of a grain-refined melt.

Key words: aluminium alloy, solidification, grain refining, fading, stirring

1. Introduction

In industry, grain refinement is a common way of achieving a proper, uniform, fine grain structure in wrought aluminium alloys, since metals and alloys usually solidify with a coarse, columnar grain structure under normal casting conditions. The most widely used grain refiners are based on Al-Ti-B, notably Al-5wt.%Ti-1wt.%B (AlTi5B1). AlTi5B1 grain refiners are composed of an α-Al matrix, and Al_3Ti and TiB_2 particles [1]. Grain refiners based on Al-Ti-B are very effective, but they suffer from poisoning in the presence of Zr, and also some other elements [2]. For this and other reasons there is an increased interest in the use of alternative Al-Ti-C-based grain refiners, for example, Al-3wt.%Ti-0.15wt.%C. The AlTi3C0.15 grain refiner is composed of an α-Al matrix, Al_3Ti and TiC particles [3].

Many theories exist that explain the mechanism of grain refinement [1, 2, 4]. It is believed that the nucleation of the aluminium grains takes place directly or indirectly (with an intermediate layer between) on the TiB_2 or TiC particles. As well as the particles on which the nucleation of the aluminium takes place the solutes in the alloys also promote grain refinement [5]. The TiB_2 phase is known to be a thermodynamically stable phase in the aluminium melt. The TiC phase, on the other hand, is thermodynamically unstable under the typical conditions of grain refinement, but the rate of the replacement of TiC by Al_4C_3 is slow and does not significantly affect the grain refinement at low temperatures and for short holding times [6–8].

It is also known that the effect of the AlTi5B1 grain refiner is much smaller than expected if the grain-refined melt is held for a longer time. This phenomenon is usually called fading, and means that the number of potent nucleation sites decreases with holding time [2]. Several results in the literature [9–16] indicate that the fading of the Al-Ti-B grain refiners is a consequence of the settling of TiB_2 particles, which represent the nucleating particles, during the holding and remelting.

The presence of different particles in the melt is an important issue since the recycling of aluminium and aluminium alloys is a common practice in the aluminium industry. Scrap usually contains TiB_2 particles and, consequently, these particles are present in non-grain-refined, industrial aluminium and aluminium alloys. The AlTiC grain refiner could be ineffective in the presence of TiB_2 particles [17, 18]. It

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Table 1. Composition of the Al-Fe alloy in wt.%

Si	Fe	Cu	Mn	Cr	Zr	V	Ti	B
0.065	1.36	0.098	0.263	< 0.001	< 0.002	0.003	0.008	< 0.0005

Table 2. Titanium, boron and carbon contents in the grain refiners in wt.%

	Ti	B	C
AlTi5B1	5.1	1.01	
AlTi3C0.15	2.7		0.16

Table 3. Quantities of Al-Fe alloy and grain refiners used in the experiment

Melt	Al-Fe alloy	AlTi5B1	AlTi3C0.15
B	3.21 kg	3.21 g	
BC	3 kg	3 g	4 g

was found that the AlTiC grain refiner was ineffective only in the presence of "freshly" added TiB_2 particles due to the smaller undercooling needed for the free growth of the α-Al grains on the TiB_2 particles.

The aim of this work was to find out how fading and stirring affects the performance of the grain refinement in an Al-Fe melt grain refined with just the AlTi5B1 refiner and with the AlTi5B1 and AlTi3C0.15 refiners.

Table 4. Courses of the experiments

	Time	Samples
Addition of grain refiner		
Stirring	Immediately after addition	
First sample	2 minutes	B1, BC1
Second sample	15 minutes	B2, BC2
Stirring	24 minutes 30 seconds	
Third sample	25 minutes	B3, BC3

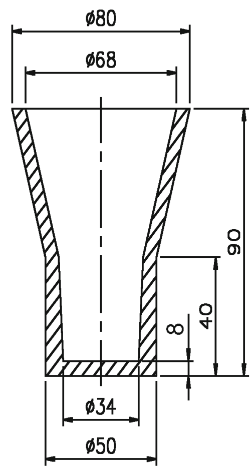

Fig. 1. Shape and dimensions of the bronze mould.

2. Experimental

An Al-Fe alloy was melted in an induction furnace with a graphite crucible. The melts were grain refined with the commercial AlTi5B1 and AlTi3C0.15 grain refiners in the form of 9.5-mm-diameter wire at a temperature of 705 ± 5 °C and stirred with a graphite stick after the addition of the grain refiners. The composition of the Al-Fe alloy and the contents of titanium, boron, and carbon in the grain refiners are presented in Tables 1 and 2. The quantities of Al-Fe alloy and grain refiners used for the preparation of the melts are presented in Table 3.

The first samples (B1 and BC1) were cast two minutes after the addition of the grain refiner into a bronze mould, as presented in Fig. 1 (cooling rate $\sim 15\,°C\,s^{-1}$). Meanwhile, the grain-refined melts were held at the temperature of 705 ± 5 °C. The second samples (B2 and BC2) were cast fifteen minutes after the addition of the refiners. Twenty-four minutes and thirty seconds after the addition of the refiners the melts were stirred again, and twenty-five minutes after the addition, the third samples (B3 and BC3) were cast. The courses of the experiments are presented in Table 4.

The castings from the bronze mould were cut 13 mm above the bottom for the microstructure analysis. The samples for the microstructure analysis (grain size measurement) were ground, polished and anodized for 2 minutes at 23 V in a 2.5 % water solution of HBF_4 for polarized-light microscopy. The average grain areas were calculated from the measured individual grain areas on the polarized-light microscopy images using commercial software for image analysis. The 95 % confidence intervals (95 % CI) for the measured average grain areas were determined according to ASTM E 1382-97. The average grain areas were converted to the mean linear-intercept lengths according to ASTM E 112-96. The term "grain size" in this paper corresponds to the mean linear-intercept length.

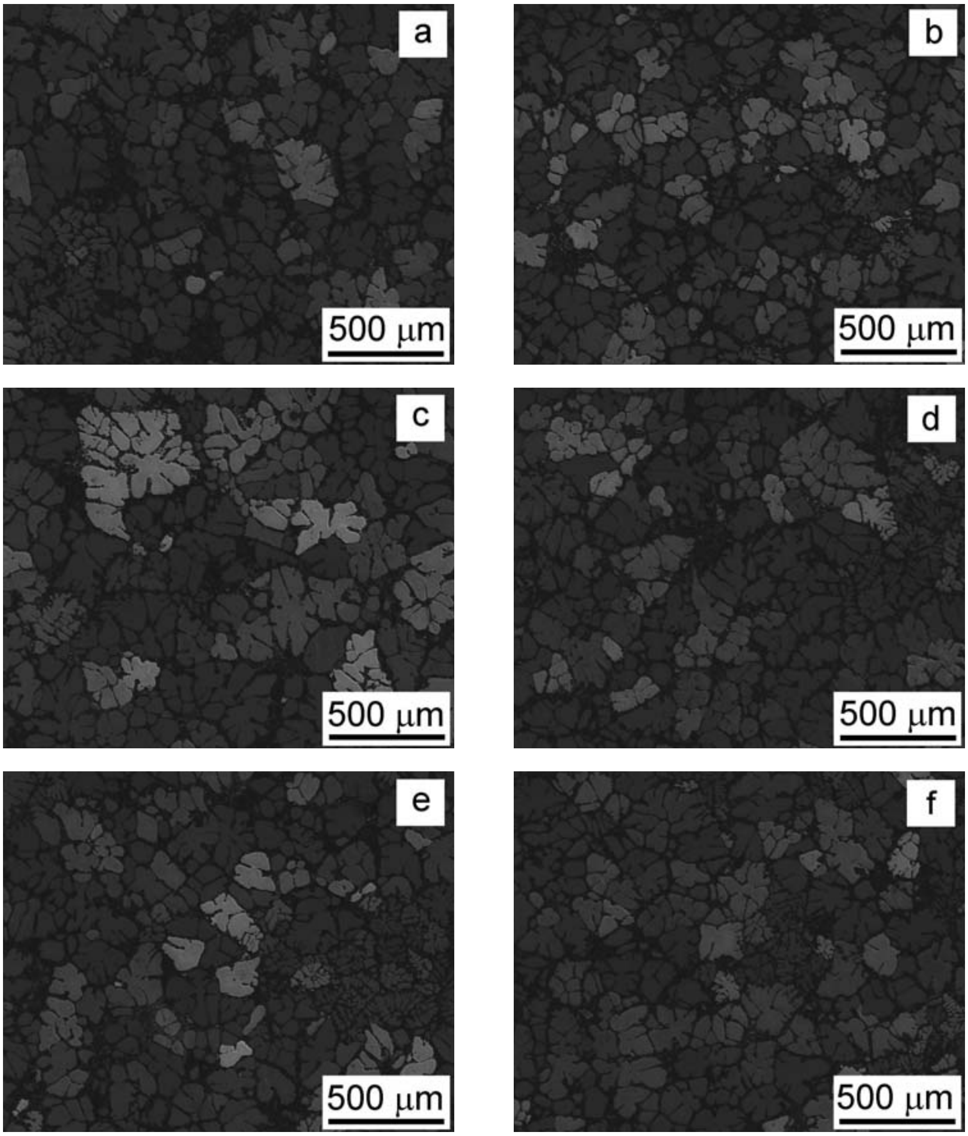

Fig. 2. Polarized-light micrographs of samples cast from the grain-refined melts a), b) two minutes, c), d) fifteen minutes, and e), f) twenty-five minutes after the addition of the grain refiners. The micrographs of the samples cast from melts grain refined just with the AlTi5B1 grain refiner are presented in figures a), c) and e), and those grain refined with both the AlTi5B1 and AlTi3C0.15 grain refiners, in figures b), d) and f).

3. Results and discussion

The microstructures and grain sizes in the samples cast from the grain-refined melts, two minutes, fifteen minutes and twenty-five minutes after the addition of the grain refiners are presented in Fig. 2 and Table 5. The results from Table 5 are also presented in the form of a diagram showing the variation of the grain size with respect to the holding time and stirring for the Al-Fe alloy with the addition of AlTi5B1 and for the Al-Fe alloy with the addition of both AlTi5B1 and AlTi3C0.15 grain refiners in Fig. 3.

The grain size two minutes after the addition of grain refiners shows that in the case of the addition of the AlTi5B1 grain refiner the grain size is

Table 5. Average grain areas, 95 % confidence intervals for the grain areas and grain sizes in samples cast from the grain-refined melts, two minutes, fifteen minutes and twenty-five minutes after the addition of the grain refiners

Sample	Grain area (μm^2)	Grain area 95%CI (μm^2)	Grain size (μm)
B1	27398	2689	147
B2	45146	6786	189
B3	24979	4085	141
BC1	22057	3311	132
BC2	25590	3151	142
BC3	21928	3573	132

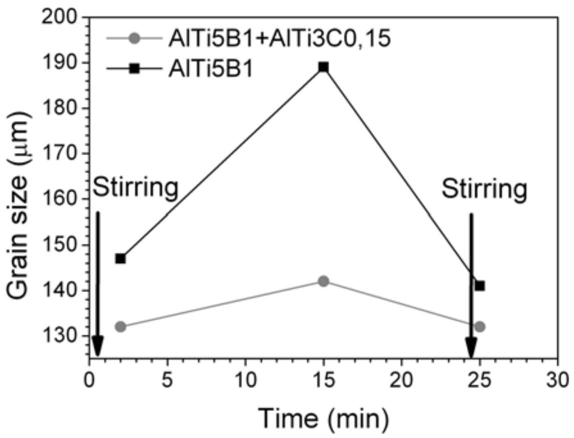

Fig. 3. Grain size with respect to the time after the addition of grain refiners for the Al-Fe alloy.

147 μm, and in the case of the addition of both the AlTi5B1 and AlTi3C0.15 grain refiners it is 132 μm. The results in Fig. 2 and Table 5 reveal that the grain size in the Al-Fe alloy with the addition of just the AlTi5B1 grain refiner increases with increasing holding time from 147 μm to 189 μm, while in the case of the Al-Fe alloy with the addition of both the AlTi5B1 and AlTi3C0.15 grain refiners it increases only from 132 μm to 143 μm. The grain sizes decrease after stirring, to 141 μm in the case of the Al-Fe alloy with the addition of the AlTi5B1 grain refiner, and to 132 μm in the case of the Al-Fe alloy with the addition of both the AlTi5B1 and AlTi3C0.15 grain refiners.

The grain size two minutes after the addition of the grain refiners is slightly smaller in the case of the addition of both the AlTi5B1 and AlTi3C0.15 grain refiners (sample BC1) compared to the addition of just the AlTi5B1 refiner (sample B1). It was found that in the case of the addition of both grain refiners the AlTi3C0.15 grain refiner is not effective and the nucleation of the α-Al grains takes place on the TiB_2 particles due to the temperature rise before melt undercools enough for the nucleation on TiC particles [18, 19]. A slightly smaller grain size in the case of the addition of both the AlTi5B1 and AlTi3C0.15 grain refiners (sample BC1) compared to the melt that was grain refined just with the AlTi5B1 refiner (sample B1) is most likely the consequence of the additional content of solute titanium brought in by the AlTi3C0.15 grain refiner.

The grain size markedly increases with increasing holding time in the case of the addition of just the AlTi5B1 grain refiner (melt B), while, on the other hand, only a slight increase in the grain size with increasing holding time is observed in the case of the addition of both the AlTi5B1 and AlTi3C0.15 grain refiners (melt BC). The reason for the marked increase in the case of the addition of just the AlTi5B1 grain refiner (melt B) is fading.

It has been reported that fading leads to a marked increase in undercooling on solidification [18, 19]. Cooling curves confirm that the recalescence fifteen minutes after the addition of the AlTi5B1 grain refiner, compared to recalescence two minutes after the addition in aluminium, becomes larger [18, 19]. The fading and the increase in the undercooling on solidification can be explained by the settling of the TiB_2 particles due to gravity. Greer et al. [20] showed that the nucleation was limited by recalescence and a smaller particle needed a greater degree of undercooling for free growth. It is also common for larger particles to settle faster. Consequently, after some holding time the size distribution changes in such a way that the fraction of the larger and more potent particles decreases. Such a size distribution will clearly lead to a less efficient grain refinement and greater undercooling on solidification.

If both grain refiners are present the grain size increase with increasing holding time is almost negligible. This can, again, be explained by the fading of the AlTi5B1 grain refiner [18, 19]. This fading of the AlTi5B1 grain refiner also indicates that the undercooling on solidification had increased [18, 19]. The previously mentioned change in the size distribution of the TiB_2 particles due to the settling of these particles leads to undercooling, which is large enough for the nucleation of the α-Al on the TiC particles. Consequently, the AlTi3C0.15 grain refiner can become effective in the presence of TiB_2 particles, since the fading of the AlTi3C0.15 refiner is not instantaneous [6–8, 15, 19]. This is the reason why the grain size increase with holding time is almost negligible in the case of the addition of both the AlTi5B1 and AlTi3C0.15 grain refiners (melt BC) compared to the case of the addition of just the AlTi5B1 grain refiner (melt B), where it is somewhat larger. It is expected that in the case of the addition of both the AlTi5B1 and AlTi3C0.15 grain refiners (melt BC), fifteen minutes after the addition of the grain refiners the nucleation of the α-Al grains also takes place on the TiC particles.

Both melts were stirred thirty seconds before the third samples were cast (samples B3 and BC3). The grain sizes in both samples were approximately the same as two minutes after the addition of the grain refiners. The result in the case of the addition of just the AlTi5B1 grain refiner (melt B) shows that stirring completely restored the grain-refinement effect that was lost with fading. It is assumed that stirring of the melt redistributed the settled TiB_2 particles throughout the melt and that these particles could again participate in the process of the nucleation of the α-Al grains on solidification and result in a similar grain-refinement effect as before the fading.

It has been reported that fading in the case of the

AlTi5B1 grain refiner, in addition to a decreased effectiveness of the grain refiner, leads to a marked increase in recalescence (undercooling) on solidification [18, 19]. As was mentioned previously, it is common for larger particles to settle faster and, consequently, the size distribution changes in such a way that the fraction of the larger and more potent particles decreases. Such a size distribution would clearly lead to a less efficient grain refinement and a larger undercooling on solidification. Since the grain-refinement effect was restored with the stirring we assume that the recalescence was decreased due to the redistribution of the settled fraction of the larger and more potent TiB_2 particles. The situation in sample BC3 would then be similar to that in the sample BC1, and it would again be expected that in sample BC3 the nucleation of the α-Al grains takes place on the TiB_2 particles.

These results also indicate that the use of both grain refiners leads to an altered fading performance of the grain-refined melt. In comparison to the AlTi3C0.15 grain refiner, the AlTi5B1 grain refiner fades more rapidly (at shorter holding times). The addition of the AlTi3C0.15 grain refiner to the AlTi5B1 refiner would not have a marked impact on the grain-refinement performance at short holding times, where the AlTi5B1 refiner is very effective. On the other hand, the addition of the AlTi3C0.15 grain refiner to the AlTi5B1 refiner would become quite obvious for longer holding times, where the performance of the AlTi5B1 refiner rapidly fades and the AlTi3C0.15 refiner becomes active due to its different fading performance. In this way it is possible to control, to a limited extent, the fading performance of a grain-refined melt.

4. Conclusions

The Al-Fe alloy was in one case grain refined with the AlTi5B1 grain refiner and in the other case grain refined with both the AlTi5B1 and AlTi3C0.15 grain refiners.

The grain size two minutes after the addition of the grain refiners was slightly smaller in the case of the addition of both the AlTi5B1 and AlTi3C0.15 grain refiners, probably due to the additional content of solute titanium brought in by the AlTi3C0.15 grain refiner. The grain size increase with the increasing holding time was almost negligible in the case of the addition of both the AlTi5B1 and AlTi3C0.15 grain refiners, while in the case of the addition of just the AlTi5B1 grain refiner it was relatively large. It is assumed that the almost negligible grain size increase with increasing holding time found in the case of the addition of both the grain refiners is due to the fading of the AlTi5B1 grain refiner and the consequent effectiveness of the AlTi3C0.15 grain refiner in the presence of the TiB_2 particles. The stirring of both melts led to approximately the same grain size as was observed for two minutes after the addition of grain refiners. The results indicate that stirring completely restored the grain-refinement effect of the AlTi5B1 grain refiner, which was lost with fading and led again to a similar situation that was observed two minutes after the addition of the grain refiners. The results also indicate that it is possible, with the use of both grain refiners, to control the fading performance of a grain-refined melt to a limited extent.

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