I. NAGLIČ

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THE INFLUENCE OF STIRRING ON THE FADING OF THE AlTi5B1 GRAIN REFINER IN AN Al–Fe ALLOY

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Preliminary Note – Prethodno priopćenje

An Al–Fe alloy, melted in an induction furnace with a graphite crucible, was grain refined with the AlTi5B1 grain refiner. The melt was stirred after the addition of the grain refiner. The first sample was cast two minutes after the addition of the grain refiner and the second sample fifteen minutes after the addition of the grain refiner. The melt was stirred again before the third sample was cast. The grain size was found to increase with the holding time. However, the grain size of the third sample, after stirring, decreased to a similar size to that of the first sample. The results indicate that the stirring of a grain-refined melt can restore the grain-refinement effect.

Key words: aluminium alloy, solidification, grain refinement, fading

Utjecaj miješanja taljevine na slabljenje učinka AlTi5B1 cjepiva u Al-Fe legurama. Al-Fe legura, taljena u grafitnom lončiću u indukcijskoj peći, je usitnjavana pomoću AlTi5B1 cjepiva. Taljevina je promiješana nakon dodatka cjepiva. Prvi je uzorak lijevan dvije, a drugi petnaest minuta nakon dodatka sredstva za usitnjavanje. Taljevina ja ponovo promiješana prije nego što je lijevan treći uzorak. Uočeno je da veličina zrna raste s vremenom zadržavanja. Veličina zrna se u trećem uzorku nakon promiješavanja smanjuje do slične veličine kao u prvom uzorku. Rezultati upućuju na to da promiješavanje cijepljene taljevine može povratiti učinak usitnjavanja.

Ključne riječi: aluminijska legura, skrućivanje, usitnjavanje, slabljenje učinka

INTRODUCTION

In industry, grain refinement is a common method for achieving a proper, uniform, fine grain structure in wrought aluminium alloys. 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, Al_3Ti and TiB_2 particles [1].

Many theories exist that explain the mechanism of grain refinement [1-3]. It is believed that the nucleation of the aluminium grains takes place directly or indirectly (with an intermediate layer between) on the TiB_2 particles. As well as the particles, on which the nucleation of the aluminium takes place, the solutes in the alloys also promote grain refinement [4].

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 prior to casting. This phenomenon is usually referred to as fading, and suggests that the number of potent nucleating sites decreases with the holding time [2]. This is usually attributed to either dissolution or the settling/floating (or both) of the nucleating particles during long holding periods [2, 5-9].

Continuous stirring has been found to prevent the fading in an Al-Ti-B grain-refined aluminium melt [6, 10]. The results of Wang et al., for example, showed that the content of Ti and B in an Al-Ti-B grain-refined melt decreased with the holding time and that stirring of the melt was able to recover the Ti and B contents in the melt [6]. The Ti and B contents were found to increase at the bottom of the crucible due to settling of the Al_3Ti and TiB_2 particles, but also that vigorous agitation of the melt using argon purging could partially return the particles back to the melt [7]. Our previous results have also shown that with remelting, the grain size increases, while the Ti and B contents decrease [11]. We also showed that the effectiveness of the same content of B in the form of grain refiner decreases with remelting [11].

The aim of this work is to determine how stirring affects the fading of the AlTi5B1 grain refiner in an Al–Fe alloy.

EXPERIMENTAL

An Al–Fe alloy (3,21 kg) was melted in an induction furnace with a graphite crucible. At a temperature of 705±5 °C the melt was grain refined with 3,2 g of commercial AlTi5B1 grain refiner in the form of 9,5-mm-diameter wire. The chemical compositions of the Al–Fe alloy and the grain refiner are presented in Tables 1 and 2. The melt was stirred with a graphite stick after the ad-

I. Naglič, Institute of Metals and Technology, Ljubljana, Slovenia

Table 1 **Chemical composition of the Al–Fe alloy / 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

dition of the grain refiner. In the first experiment the sample was cast into a bronze mould two minutes after the addition of the grain refiner, as presented in Figure 1 (cooling rate ∼15 °C/s). Meanwhile, the melt was held at a temperature of 705±5 °C. The second sample was then cast fifteen minutes after the addition of refiner. Next, twenty four and a half minutes after the addition of the refiner the melt was stirred again, and twenty five minutes after the addition, the third sample was cast. The course of the experiment is presented in Table 3.

The casings of the bronze mould were cut 13 mm above the bottom for the microstructure analysis. Metallographic samples for optical microscopy were ground and polished with diamond paste. The preparation of the metallographic samples was completed by polishing in a suspension of SiO_2. The metallographic samples for observation in polarized light with a lambda filter were anodized at 23 V for 2 min in a 2,5 % water solution of HBF_4.

The average grain areas were measured on polarized-light microscopy images using commercial software for image analysis. The average grain areas were calculated from the measured individual grain areas. 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 $\bar{l}$ according to ASTM E 112-96. The term “grain size” in this paper corresponds to the mean linear-intercept length.

RESULTS AND DISCUSSION

The grain size in the first sample, cast two minutes after the addition of the grain refiner, was 147 μm. The grain size increased with increasing holding time up to 189 μm in the second sample, cast fifteen minutes after the addition of the refiner. The grain size decreased with a longer holding time to 141 μm in the third sample cast twenty-five minutes after the addition of the refiner and stirred thirty seconds before casting. The variation of the grain size with the holding time and stirring is presented in Table 4 and Figure 2, and the microstructures of the cast samples are presented in Figure 3.

The large increase in grain size with holding time is to be expected since fading is a well-known phenomenon. The decrease in the grain size, after stirring, to approximately the same size as that obtained shortly after the addition of the grain refiner shows that stirring can restore the grain-refinement effect that is lost with fading. The results can be explained by the settling of the TiB_2 particles, since the density of the aluminium melt

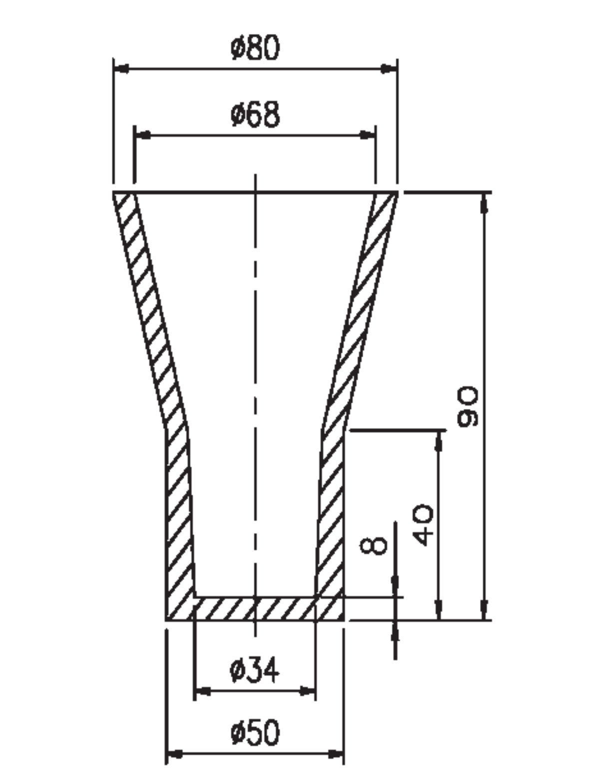

Figure 1 The shape and dimensions of the bronze mould

Table 2 **Titanium and boron contents in the AlTi5B1 grain refiner / wt.%**

	Ti	B
AlTi5B1	5,1	1,01

Table 3 **The course of the experiment**

	Time
Addition of grain refiner	
Stirring	Immediately after addition
First sample	2 minutes
Second sample	15 minutes
Stirring	24 minutes 30 seconds
Third sample	25 minutes

Table 4 **Average grain areas, 95 % confidence intervals (95 % CI) of the average grain area and grain sizes for samples cast 2 minutes, 15 minutes, and 25 minutes after the addition of grain refiner**

Time / min	Grain area / μm^2	Grain area 95 % CI / μm^2	Grain size / μm
2	27398	2689	147
15	45146	6786	189
25	24979	4085	141

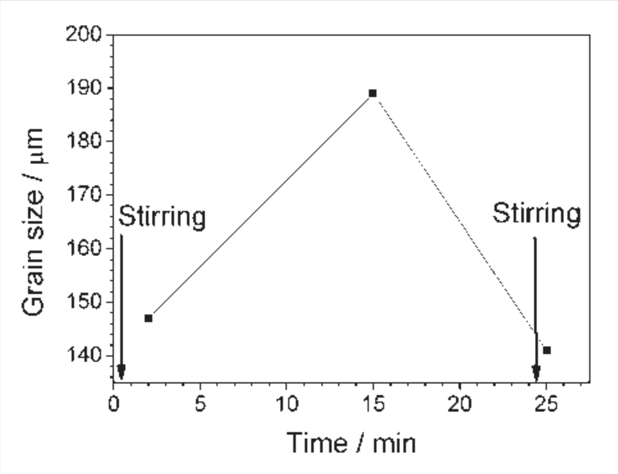

Figure 2 Variation of grain size with holding time and stirring for an Al–Fe alloy grain refined with the AlTi5B1 refiner

at 700 °C is ~ 2,4 g/cm^3 and the density of the TiB_2 phase is 4,5 g/cm^3. Several results in the literature [2, 5–12] indicate that the settling of the TiB_2 particles is the main reason for the fading. The TiB_2 particles represent the nucleation points for α-Al grains. The settling of these particles leads to a decrease in the number of newly formed grains and consequently to a larger grain size after the solidification. The stirring of the melt redistributes the settled TiB_2 particles throughout the melt and these particles can again participate in the process of nucleation of the α-Al phase on solidification and result in a similar grain-refinement effect to that before fading.

The results show that the stirring completely restored the grain-refinement effect lost with fading. An incomplete restoration of the grain-refinement effect after stirring is usually attributed to the agglomeration of TiB_2 particles [13]. The grain size in the third sample is even slightly smaller than in the first sample 2 minutes after the addition of grain refiner. This latter effect could be a consequence of the fact that the first sample was cast two minutes after stirring and the third only thirty seconds after stirring.

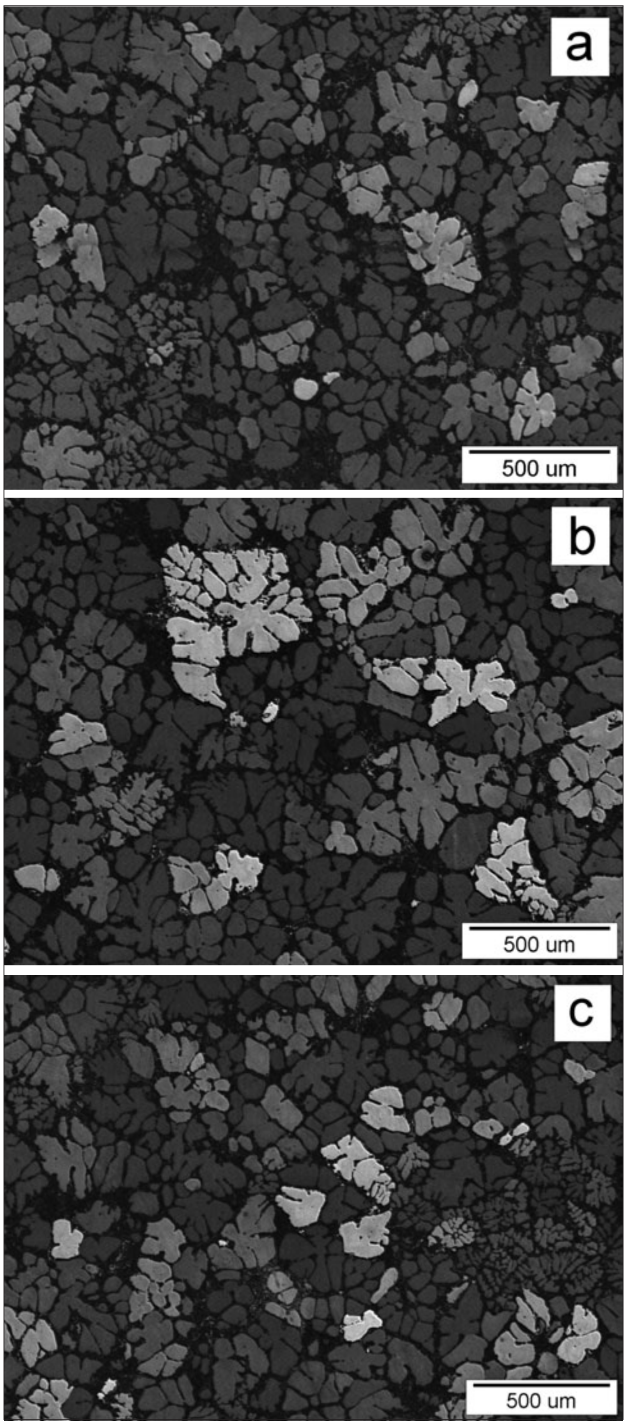

Figure 3 Microstructures of samples cast a) two minutes, b) fifteen minutes and c) twenty-five minutes after the addition of the AlTi5B1 grain refiner. The melt was stirred thirty seconds before sample c) was cast

CONCLUSIONS

An Al–Fe alloy was grain refined with the AlTi5B1 grain refiner. The effectiveness of the AlTi5B1 grain refiner was found to decrease with the increasing holding time. It was also found that stirring of the melt before casting can restore the grain-refinement effect of the AlTi5B1 refiner that is lost as a result of the fading. The results suggest that the settling of the TiB_2 particles due to gravity is the main reason for the fading of the AlTi5B1 grain refiner.

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Note: Linguistic Adviser / English language Paul Mc Guiness, Ljubljana, Slovenia.