I. NAGLIČ, A. SMOLEJ, M. DOBERŠEK

ISSN 0543-5846 METABK 48(3) 147-150 (2009) UDC – UDK 669.71:66.065:621.746=111

# THE INFLUENCE OF ALLOYING ELEMENTS IN ALUMINIUM ON THE GRAIN REFINEMENT WITH ALTI5B1

Received – Prispjelo: 2008-07-20 Accepted – Prihvaćeno: 2008-10-20 Original Scientific Paper – Izvorni znanstveni rad

This work deals with the influence of alloying elements in aluminium on the grain refinement with various additions of AlTi5B1. Grain-refinement tests were made at a cooling rate of 15 °C/s. The results revealed that in both aluminium and an Al-Fe alloy the grain size decreases with increasing additions of the AlTi5B1 grain refiner. We found that for the same boron content the grain size was smaller in the case of the Al-Fe alloy. The difference in the grain sizes for the same content of boron was approximately 15  $\mu$ m; this is considerably smaller than the difference between the grain sizes in samples with the same difference of growth-restricting factor made at slower cooling rates.

Key words: aluminium, casting, solidification, grain refinement

**Utjecaj legirajućih elemenata u aluminiju na usitnjavanje zrna s AlTi5B1.** Članak se bavi utjecajem legirajućih elemenata u aluminiju na usitnjavanje zrna s različitih dodatcima AlTi5B1. Ispitivanje usitnjavanja zrna je provedeno pri brzini hlađenja od 15 °C/s. Rezultati su otkrili da i u aluminiju i u Al-Fe leguri veličina zrna pada s povećanjem dodatka AlTi5B1 cjepiva. Utvrdili smo da je za isti dodatak bora veličina zrna manja u Al-Fe leguri. Razlika u veličini zrna za isti dodatak bora je bila približno 15 µm; što je značajno manje od razlike u veličini zrna u uzorcima s istom razlikom faktora ograničavanja rasta, pri manjim brzinama hlađenja.

Ključne riječi: aluminij, lijevanje, skrućivanje, usitnjavanje zrna

### 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.

Many theories exist to explain the mechanism of grain refinement [1, 2, 3]. Besides nucleant particles, like  $TiB_2$  and TiC, found in commercial grain refiners, solutes also play an important role in grain refinement [4]. The extent of the grain-refinement effect of an individual solute can be expressed by the growth-restricting factor (GRF):

$$GRF = mC_o(k-1) \tag{1}$$

where *m* is the slope of the liquidus,  $C_o$  is the concentration of the solute in the melt, and *k* is the equilibrium partitioning coefficient [1]. In the presence of a number of elements in the alloy the GRF is taken as the summation of the individual elements, which means the interactions between solutes are neglected.

It is known that the GRF is inversely proportional to the growth rate of the grains [5, 6]. When the GRF is large, the rate of growth (and hence the latent-heat evolution) is slow, allowing large numbers of substrates to be-

come active. The reverse is true when the GRF is small [1, 2]. Easton and St John [7] in their model confirmed that the GRF represents the rate of development of the constitutional zone with respect to the fraction of solid at a zero fraction of solid. In other words, it is a measure of how rapidly the constitutionally under-cooled zone is formed at the earliest stages of growth.

The influence of alloying elements in aluminium on the grain size has also been experimentally studied [5, 6, 8-14]. The data shows that the grain size decreases significantly with an increasing GRF. It is important to point out that the majority of the previously mentioned grain-refining tests were made at cooling rates ranging from 0,5 to 5 °C/s. The results of Backeroud and Johnsson [12] reveal that increasing the GRF shows a reduced effect of grain refinement at higher cooling rates. The aim of this work is to present the results of the grain refinement of samples with different GRFs made at faster cooling rates (15 °C/s) and compare them with the results from slower cooling rates.

## EXPERIMENTAL

Commercial purity aluminium (99,8 wt.% Al), an Al-Fe alloy and an AlTi5B1commercial grain refiner (in the form of 9,5-mm-diameter wire) were used in this study. The chemical compositions of the aluminium, the

I.Naglič, M. Doberšek, Institute of Metals and Technology, Ljubljana, Slovenia, A. Smolej, University of Ljubljana, Faculty of Natural Sciences and Engineering, Ljubljana, Slovenia

	Si	Fe	Cu	Mn	Cr	V	Ti	В	Zr	Al
Aluminium	0,031	0,081	-	-	-	0,001	0,0015	<0,0005	-	rest
Al-Fe alloy	0,065	1,36	0,098	0,263	<0,001	0,003	0,008	<0,0005	<0,002	rest

Table 1. Chemical co	mpositions of	aluminium a	and Al-Fe a	lloy in wt.%
----------------------	---------------	-------------	-------------	--------------

Table 2. Chemical composition of the grain refiners used in the aluminium and the Al-Fe alloy in wt.%

Grain refiner	Ti	В	
AlTi5B1 in aluminium	5,1	1,04	
AlTi5B1 in Al-Fe alloy	5,1	1,01	

Al–Fe alloy and the AlTi5B1 grain refiner are presented in Tables 1 and 2.

Three kilograms of aluminium and the Al–Fe alloy were melted in a medium-frequency induction furnace with a graphite crucible. Grain refiners were added at a temperature of  $705\pm5$  °C. The melts were stirred with a graphite stick and 2 min after the addition of the grain refiners they were cast into a bronze mould (cooling rate ~15 °C/s), as presented in Figure 1. The castings were cut 13 mm above the base for the preparation of samples for metallographic examination.

Samples for microstructure analysis were ground, polished and anodized for 2 minutes at 23 V in a 2,5 % water solution of HBF<sub>4</sub> for polarized-light microscopy. The average grain areas were measured on polarized-light microscopy images using commercial software for the image analysis. The average grain areas were converted to the mean, linear-intercept lengths in accordance with ASTM E112-96. The term "grain size" in this paper corresponds to the mean, linear-intercept length.

# **RESULTS AND DISCUSSION**

The results of the grain refinement of the aluminium and the Al–Fe alloy with the AlTi5B1 grain refiner are presented in Table 3 and Figure 2. The titanium and bo-



Figure 1. Shape and dimensions of the bronze mould

ron contents in the samples presented in Table 3 are based on the actual chemical analysis of each sample. The content of "free" titanium presented in Table 3 was calculated on the basis of the titanium and boron contents in the sample and the assumption that all the boron forms TiB<sub>2</sub>. The GRF was calculated according to equation (1). Besides the "free" titanium, the silicon, iron and vanadium contents were considered in the case of the aluminium, and the silicon, iron, copper, manganese and

	Addition of gra- in refiner / g/kg	Ti / wt.%	B / wt.%	Grain size / μm	"free" Ti / wt.%	GRF / K
Aluminium						
A1	0,88	0,0058	0,0008	149	0,0040	1,36
A2	1,71	0,0090	0,0012	133	0,0063	1,89
A3	2,55	0,0130	0,0019	123	0,0088	2,44
A4	4,28	0,0230	0,0035	98	0,0152	3,90
Al-Fe alloy						
F1	0,33	0,0103	0,0009	142	0,0083	6,65
F2	0,53	0,0110	0,0008	135	0,0092	6,86
F4	0,99	0,0127	0,0011	119	0,0103	7,09
F5	1,32	0,0140	0,0016	114	0,0105	7,14
F6	3,03	0,0230	0,0027	94	0,0170	8,62

 Table 3.
 Titanium and boron contents, grain size, "free" titanium content and GRF for the aluminium and the Al-Fe alloy with different additions of the AlTi5B1 grain refiner



Figure 2. Dependence of the grain size on the boron concentration for the aluminium and the Al-Fe alloy with the addition of the AlTi5B1 grain refiner

vanadium contents in the case of the Al-Fe alloy. The slopes of the liquidus m and the equilibrium partitioning coefficients k used in the calculation of the GRF were obtained from the work of Easton and St John [7].

With increasing boron content, as a consequence of the addition of the AlTi5B1 grain refiner, the grain size decreased in both the aluminium and the Al-Fe alloy (Figure 2). In the presented range of boron content the grain size is smaller in the case of the Al-Fe alloy. The difference in the grain size between the aluminium and the Al-Fe alloy for the same boron content is approximately 15  $\mu$ m (Figure 2). The boron contents in samples A2 and F4 were 0,0012 wt.% and 0,0011 wt.%, and are comparable (Table 3). The GRFs for the samples A2 and F4 were 1,89 and 7,09, and the grain sizes for these two samples were 133  $\mu$ m and 119  $\mu$ m. The increase in the GRF between the aluminium and the Al-Fe alloy, which

Table 4.Comparison of the effect of solutes on the<br/>grain refinement at different cooling rates

Sample	B / wt.%	Cooling rate / °C/s	Grain size /μm	GRF / K	Referen- ce
A4	0,0035	15	98	3,9	
F6	0,0027	15	94	8,6	
	0,0035	0,5	~550	3,9	12
	0,0035	0,5	~300	8,6	12
	0,0035	1	~450	3,9	11
	0,0035	1	~250	8,6	11
	0,0035	5	~300	3,9	12
	0,0035	5	~200	8,6	12
A3	0,0019	15	123	2,4	
F5	0,0016	15	114	7,1	
F6	0,0027	15	94	8,6	
	0,0020	3,5	~200	2,4	10,15*
	0,0020	3,5	~140	7,1	10,15*
	0,0020	3,5	~130	8,6	10,15*

\*Results of the Free-growth model [15] were used for estimation of the grain sizes at different GRF.

is 5,1 in the case of the samples A2 and F4, corresponds to a 14- $\mu$ m-smaller grain size.

A comparison of the effect of the solutes on the grain sizes at different cooling rates is presented in Table 4. A comparison of the influence of the same difference in GRF at similar boron contents and different cooling rates on the grain size shows that the difference in the grain size decreases with the increasing cooling rate. A significant decrease in the difference in grain size with increasing cooling rate was observed at 15 °C/s in comparison to the slower cooling rates.

The effect of the cooling rate on the efficiency of the grain refinement by the solutes also plays a role in the comparison of the different grain refiners, like AlTi5B1 and AlTi3C0,15 or AlTi5C0,2. The grain refiners AlTi3C0,15 and AlTi5C0,2 contain a larger content of "free" titanium in comparison to AlTi5B1 for the same overall content of titanium. Assuming that all the carbon in the AlTi3C0,15 grain refiner forms TiC and all the boron in the AlTi5B1 grain refiner forms TiB<sub>2</sub>, then 80 % of the titanium is "free" (unbounded in TiC) in the AlTi3C0,15 and only 56 % of the titanium is "free" (unbounded in TiB<sub>2</sub>) in the AlTi5B1 grain refiner.

A comparison of the efficiency of the grain refiners for the same level of titanium addition shows that the AlTi3C0,15 or AlTi5C0,2 grain refiners are more efficient at slow cooling rates and larger additions [16, 17], while at fast cooling rates the AlTi5B1 is much more efficient [18-20]. These results were also confirmed by the results of the performance of the AlTi5B0,2 and AlTi5C0,2 grain refiners in twin-roll strip casting [21, 22]. The results of the performance of the grain refiners at different cooling rates can be explained by the effect of the solutes on the grain refinement at different cooling rates. The grain refiners AlTi3C0,15 or AlTi5C0,2 contain larger contents of "free" titanium in comparison to the AlTi5B1, and consequently this solute titanium acts as a more efficient grain refiner at slower cooling rates than at faster cooling rates, as shown in Table 4.

## CONCLUSIONS

Aluminium and an Al-Fe alloy were grain refined with different additions of AlTi5B1 grain refiner at cooling rate of 15 °C/s. We found that the grain size decreased with increasing boron content in both the aluminium and the Al-Fe alloy. We also found that the grain size in the investigated range is, for the same boron content, smaller in the case of the Al-Fe alloy. A difference of approximately 15  $\mu$ m was found between the grain sizes for the same boron content in the aluminium and the Al-Fe alloy.

#### REFERENCES

 B. S. Murty, S. A. Kori, and M. Chakraborty, International Materials Reviews, 47 (2002) 3-29.

- [2] D. G. McCartney, International Materials Reviews, 34 (1989) 247-260.
- [3] K. T. Kashyap and T. Chandrashekar, Bull. Mater. Sci., 24 (2002) 345-353.
- [4] I. Maxwell, A. Hellawell, Acta Metall., 23 (1975) 229-237.
- [5] M. Johnsson, Z. Metallkd., 85 (1994) 781-784.
- [6] G. Chai, L. Backerud, and L. Arnberg, Materials Science and Technology, 11 (1995) 1099-1103.
- [7] M. A. Easton, D. H. StJohn, Acta Mater., 49 (2001) 1867-1878.
- [8] H. E. Vatne, Aluminium, 75 (1999) 84-90.
- [9] H. E. Vatne, Aluminium, 75 (1999) 200-203.
- [10] J. A.Spittle, S. Sadli, Materials Science Technology, 11 (1995) 533-537.
- [11] M. Johnsson and L. Backerud, Z. Metallkd., 87 (1996) 216-220.
- [12] L. Backerud and M. Johnsson, Light Metals, Edited by Wayne Hale. The Minerals, Metals and Materials Society, (1996) 679-685.
- [13] H. E. Vatne and A. Hakonsen, Light Metals, Edited by C. Edward Eckert. The Minerals, Metals and Materials Society, (1999) 787-792.
- [14] J. E. C. Hutt, A. K. Dahle, Y. C. Lee and D. H. StJohn, Light Metals, Edited by C. Edward Eckert, The Minerals, Metals and Materials Society, (1999) 685-692.

- [15] A. L. Greer, A. M. Bunn, A. Tronche, P. V. Evans, D. J. Bristow, Acta Materialia, 48 (2000) 2823-2835.
- [16] P. C. van Wiggen and J. K. Belgraver, Light Metals, Edited by C. Edward Eckert, The Minerals, Metals and Materials Society, (1999) 779-785.
- [17] P. C. van Wiggen and J. K. Belgraver, Aluminium, 75 (1999) 989-994.
- [18] I. Naglič, A. Smolej, M. Doberšek, B. Breskvar, Kovove Mater. 42 (2004) 353–362.
- [19] I. Naglič, A. Smolej, M. Doberšek, Metalurgija, 47 (2008) 2, 115-118.
- [20] I. Naglič, A. Smolej, M. Doberšek, P. Mrvar, Mater. Charact. 59 (2008), 1458-1465.
- [21] Y. Birol, S. Ucuncuoglu, M. Dundar, O. Cakir, A. S. Akkurt, Light Metals. Edited by W. Schneider. The Minerals, Metals and Materials Society, (2000) 923-929.
- [22] Y. Birol, Journal of Alloys and Compounds, 430 (2007) 179–187.

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