# Effects of Residual TiB<sub>2</sub> and TiAl<sub>3</sub> Nucleant on Grain-Refinement of Recycled Aluminum-Silicon Alloy Castings

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#### Abstract

Recycling of return scraps is a general practice in aluminum foundries. These return scraps were usually grain-refined using AI-5Ti-1B grain refiners in the previous process. As a result, concentration of Ti and B can be found to be high even without any addition of AI-5Ti-1B in the latter melt treatment. However, this high Ti and B contents may not necessarily guarantee that the resultant melt will have an appropriate grain size. This research investigates the effectiveness of TiB<sub>2</sub> and TiAl<sub>3</sub> nucleant particles existing in return scraps previously grain-refined. The work was undertaken by recycling the grain-refined castings for five cycles. Two silicon levels were tested: 6.7 and 9.5 wt. %. It was found that the concentration of both Ti and B gradually decreased by repeated use. However, the loss of grain refinement efficiency was found to be greater than the chemical changes, especially in the batch with higher Si content. It was suggested that presence of high Ti and B contents in a returned scrap as a result of previous grain-refining does not guarantee fine grains in castings.

## 1. Introduction

Grain refiners in aluminum alloy are important for successful castings. For example, mechanical properties can be improved [1, 2], hot tearing susceptibility is reduced [3] and fluidity is improved [4, 5]. There are several ways to introduce grain refiner particles into the melt. The most widely accepted method is to add commercial Al-Ti-B master alloys into the melt in the form of rod coil or waffle plate. This method provides many benefits over other methods, including emissions reduction (compared with use of salt tablets) and better composition control [1].

Recycling of return scraps is a general practice in aluminum foundries. These return scraps were usually grain-refined using AI-Ti-B grain refiners in the previous casting process. As a result, concentration of Ti and B can be found to be high even without any addition of AI-Ti-B in the latter melt treatment. Many foundries rely on the chemical analysis of Ti and B to decide whether or not additional grain refiner needs to be added.

However, this high Ti and B contents may not guarantee whether or not the resultant melt will result in a casting with an appropriate average grain size. From literature reviews, there was only one literature on the effect of recycling on grain refinement efficiency [6]. The present research will investigate the effectiveness of  $TiB_2$  and  $TiAI_3$  nucleant particles retained in using grain-refined return scraps. In addition, the research will also examine that the analysis of Ti and B chemical compositions may not be used as the only way to evaluate the necessity to add additional grain refiner into the melt.

# 2. Experimental Procedures

ASTM 356 aluminum alloy, with the chemical composition as shown in Table 1, was used in this study, because it represents one of the most common aluminum alloys for general casting processes. The 356 aluminum alloy was melted in a clay-bonded graphite crucible at 840 °C by a closed electric resistance furnace. Total weight of the 356 aluminum alloy at the beginning of each experiment is 1 kg. A covering flux (45 wt. % NaCl - 45 wt. % KCl -10 wt % NaF) was added at the beginning of melting. Upon completion of melting, 0.8 wt. %. of AI-5Ti-1B master alloy was added. Argon was later purged through a stainless steel tube (6 mm inside diameter) coated with titanium dioxide into the melt to degas hydrogen. The degassing time was 3 min in each experiment with flow rate 4 L/min at 0.2 MPa. Excessive argon purging and melt transferring also ensured that the added master alloy could be uniformly distributed throughout the melt. The melt was always kept under the covering flux of 0.5 wt. % of the melt. A 30-min contact time was used. Before pouring at 740 °C into three steel ring molds (preheated at 100 °C), the melt was agitated for one minute with argon gas to reduce the settlement of both TiAl<sub>3</sub> and TiB<sub>2</sub> every time before pouring. Each steel ring mold was made of steel, 35 mm outside diameter, 4 mm thick, and 35 mm high. The rest of the melt was cast in an ingot mold. The ingot was later recycled for four times in a similar way without any additional AI-5Ti-B. In order to demonstrate the effect of higher silicon content, another series of experiments was conducted using 356 with an extra Si (9.5 %Si). Macrographs were taken after etching with Tucker's reagent for about 10 sec. Grain size analysis was carried out using the linear intercept method. The concentrations of Ti and B were determined by using an emission spectrometer. Unfortunately, B could only be determined in terms of B intensity, not the absolute weight percent, because there was no certified reference standard with known B concentration for the present study.

Sample	Si	Fe	Cu	Mn	Mg	Ni	Zn	Ti	AI
356 (6.7 %Si)	6.715	0.387	0.069	0.040	0.233	0.008	0.011	0.018	Bal.
356 with an extra Si (9.5 %Si)	9.521	0.392	0.144	0.023	0.105	0.032	0.146	0.013	Bal.

Table 1: Chemical composition of samples. (wt.%).

# 3. Experimental Result and Discussion

Figures 2a-f show the macrographs of 356 sample with various casting conditions. Figure 2a shows the macrograph of 356 sample with no grain refiner addition. Figure 2b shows the macrograph of the 356 sample with AI-5Ti-1B grain refiner at 0.8 wt. % master alloy addition level. The smallest grain size was found in this sample. No additional AI-5Ti-1B was added when recycling the aluminum ingot for four times. It is quite clear to see that the grain size of the 4<sup>th</sup> recycled sample had the largest grain size, as shown in Figure 2f.

The more the number of recyclings, the larger the grain size of the sample was obtained. Similar experiments, conducted at the higher Si content (9.5 %Si), provide a similar trend but with even larger grain sizes as shown in Figure 3a-f. Result of the grain size analysis of the samples from different recycled ingots is shown in Figure 4. The average grain size increases with the number of recycles. Samples from 356 with an extra Si content (9.5 %Si) have larger grain sizes.

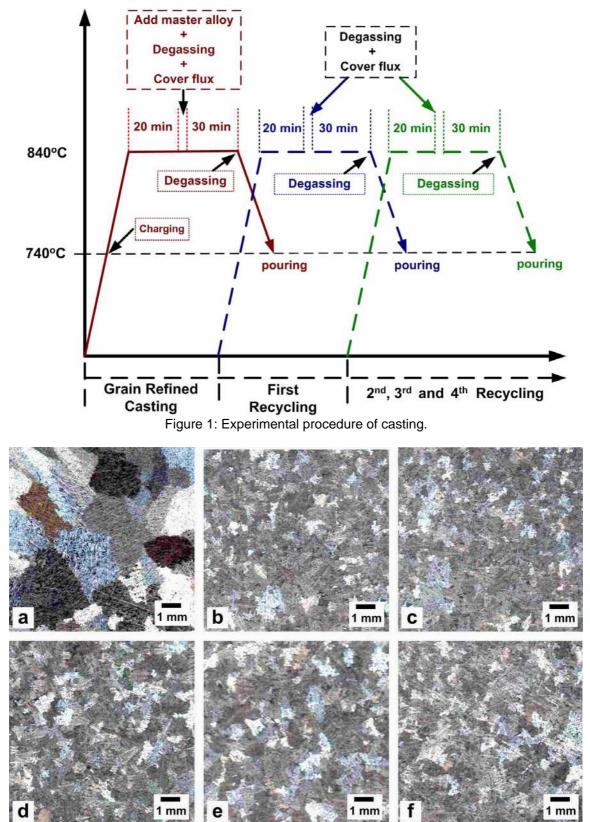


Figure 2: Macrograph of 356 (6.7% Si) a) Non-grain refined sample b) Grain refined sample c) Sample from 1<sup>st</sup> recycling d) Sample from 2<sup>nd</sup> recycling e) Sample from 3<sup>rd</sup> recycling f ) Sample from 4<sup>th</sup> recycling.

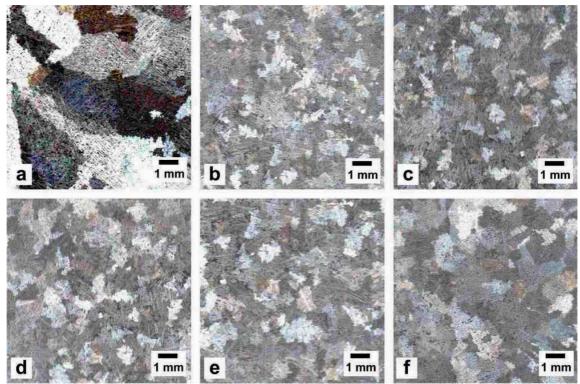


Figure 3: Macrograph of 356 with an extra amount of Si (9.5% Si) a) Non-grain refined sample b) Grain refined sample c) Sample from  $1^{st}$  recycling d) Sample from  $2^{nd}$  recycling e) Sample from  $3^{rd}$  recycling f ) Sample from  $4^{th}$  recycling.

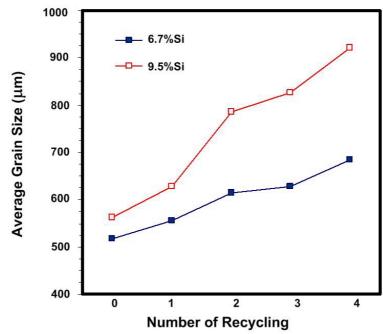


Figure 4: Average grain size of 356 samples with different Si contents after recycling.

Tables 2 shows the concentration of Ti and B in terms of count intensity found in both 356 and 356 with extra Si samples with 0.8 wt. % master alloy added. Figures 5 (a) and (b) show the composition of Ti and concentration intensity of B in the case of both 356 and 356 with extra Si. It was found that both Ti and B contents gradually decreased. The higher Si content results in faster fading. This fading phenomenon was believed to be from both Ti and B compound particles settling to the bottom as previously demonstrated elsewhere [7]. It can be seen that, after the fourth recycling, the relative decrease in Ti

contents was 16% and 19% in case of 356 and 356 with extra Si, respectively. The relative decrease in B contents was 9% and 7% in case of 356 and 356 with extra Si, respectively. On the other hand, grain sizes after the fourth recycle increased by 32% and 63% in case of 356 and 356 with an extra Si, respectively. Thus in term of relative changes in measurement, the observed effect of recycling was larger in grain size increase than in concentration decrease of both Ti and B. It is suggested that despite the relatively high residual concentration of Ti and B as analyzed, their grain refining effectiveness has been degraded by recycling, possibly by some changes in structure. It may even be possible that such changes may be harmful to some properties of the castings.

Sampla	Element	Number of recycling						
Sample	Element	0	1	2	3	4		
356 (6.7 %Si)	Ti (wt. %)	0.078	0.076	0.071	0.067	0.065		
	B (intensity)	5084	5013	4951	4652	4602		
356 with extra Si (9.5 %Si)	Ti (wt. %)	0.073	0.068	0.063	0.060	0.059		
	B (intensity)	5048	4959	4806	4765	4690		

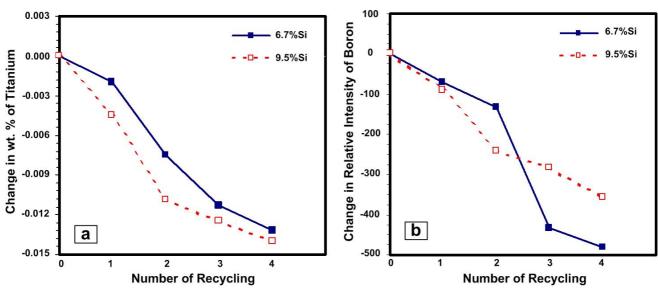


Figure 5: (a) Change in wt. % of Ti and (b) relative intensity of B of 356 samples with different Si content after recyclings.

#### 4. Summary

The major conclusions and suggestions drawn from the results of this work are as follows:

- 1. AI-5Ti-1B loses its effectiveness with repeated recycling.
- 2. Both Ti and B content decreases with repeated recycling. However, its degree of decreasing was found to be less pronounced than the increase in grain sizes.
- 3. We should not rely solely on Ti and B content as analyzed to predict the grain refinement effectiveness. After repeated recycling, Ti and B compound loses it effectiveness, especially in higher Si casting.

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### References

- [1] R. Cook, P.S. Cooper and M.A. Kearns. TMS Light Metals, 647, 1996.
- [2] D. Apelian, G.K. Sigworth and K.R. Whaler, Assessment of Grain Refinement and Modification of Al–Si Foundry Alloys by Thermal Analysis, American Foundrymen's Society, Inc, 297–307, 1984.
- [3] S.A. Metz, M.C. Flemings, A fundamental study of hot tearing, The Merton C. Flemings Symposium on Solidification and Materials Processing, USA publishers, Cambridge, MA, USA, 28–30 June, 2000, 181–188, 2001.
- [4] A.K. Dahle, L. Arnberg, P.A. Tondel and C.J. Paradies. Met. Trans. A, 8A, 2305-2313, 1996.
- [5] F.R. Mollard, M.C. Flemings and E.F. Niyama. J. Met, 11, 34-37, 1987.
- [6] P. Fisher and G.T. Campbell, Recycling The effect on grain refinement of commercial aluminium alloys, The 122<sup>nd</sup> TMS annual meeting & exhibition, Denver, CO, USA, Feb 21-25, 1993, 807-812, 1993.
- [7] C. Limmaneevichitr and W. Eidhed, Mater. Sci. Eng. A, 349A, 197-206, 2003.