Light Metals 2011 *Edited by: Stephen J. Lindsay*
TMS (The Minerals, Metals & Materials Society), 2011

Production of Al-Ti-C Grain Refiners with the Addition of Elemental Carbon and K_2TiF_6

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Keywords: Aluminum; Al-Ti-C Grain Refiners; Elemental Carbon; K_2TiF_6

Abstract

Al–Ti–B grain refiners are widely used as aluminum grain refiners. Despite the problems in application, Al–Ti–C refiners have an increasing demand in recent years. In the present work, Al-Ti-C grain refiners with different Ti:C ratios were produced by in-situ method with the addition of elemental carbon into the Al-Ti master alloy (master alloy method) and the addition of K_2TiF_6 and elemental carbon powder mixture into the commercially pure aluminum (flux method). The effects of production method and Ti:C ratio on the grain refinement process were investigated using Alcoa Cold finger Test and so optimum conditions were determined. The best performance was accomplished with the refiner produced at 1300°C with the master alloy method.

Introduction

Grain refinement has been an important technique in aluminum industry for many years to improve the soundness of aluminum products [1]. It is well known that a fine equiaxial grain structure can improve the mechanical properties, it reduces the size of defects such as micro porosity and second-phase particles[1,2] The most widely used grain refiners are based on Al-Ti-B which has α-aluminum matrix, Al_3Ti and TiB_2 particles[3]. On the other hand, the TiB_2 particles in Al-Ti-B master alloys lead to a number of problems such as streaking and porosity in foils, scratch-like linear surface, internal cracking in extrusion billets[4], suffering from poisoning in the presence of Zr, V and Cr[3,5]. In order to prevent such as harmful effects of Al-Ti-B, alternative grain refiners have been investigated [6]. The effectiveness of Al-Ti-C master alloys in the grain refinement of aluminum and its alloys have been widely investigated for many years [7]. In recent years, they have been of increasing importance in aluminum casting because, unlike conventional Al-Ti-B master alloys [8,9], they are believed to introduce a smaller volume fraction of insoluble particles into the melt. Al-Ti-C refiner are composed of an α-aluminum matrix, Al_3Ti and TiC particles. The existence of excess titanium in Al-Ti-C master alloys plays an important role on the refinement efficiency of TiC particles [10]. Al-Ti-C master alloys have been prepared by reaction of carbon with Al-Ti master alloy [11], melting the elemental blends, self propagating high temperature synthesis and reaction of molten Al with K_2TiF_6 and graphite powder [11,12]

In the present work, Al-Ti-C grain refiners were produced by master alloy method and flux method to compare the performance of refiners. Production parameters such as holding time, different Ti/C ratio and casting temperature were determined for most effective grain refiner.

Experimental Procedure

Commercial pure aluminum (AA 1080), Al–10%Ti master alloy, K_2TiF_6, Elftex 125 Cabot Carbon with average particle size of 10–20μm were used to produce Al–Ti–C grain refiners. Chemical compositions of AA1080 and Al-10%Ti alloy are given in Table I. Experiment sets were prepared by using different carbon ratios ranges changing from 0% to 0.90% as given in Table II and Table III. For each sample Ti ratio was fixed to 3%.

Table I. Chemical Compositions of AA 1080 and Al-10%Ti Master Alloy

Alloy	Al (%)	Fe (%)	Si (%)	Cu (%)	Ti (%)	Zn (%)	Bal (%)
AA1080	99.80	0.15	0.10	0.03	0.02	0.03	0.10
	Ti (%)		Fe (%)		Si (%)		Al (%)
Al-10%Ti	9.947		0.179		0.126		Rest

Table II. Grain Refiner Compositions and Experimental Parameters for the Flux Method

Grain refiner composition	Carbon and K_2TiF_6 addition temperature (°C)		Holding and casting temperature (°C)		Holding time (min)	
	Exp. 1	Exp. 2	Exp. 1	Exp. 2	Exp. 1	Exp. 2
Al-3%Ti	900	800	900	1300	20	20
Al-3%Ti-0.15%C	900	800	900	1300	20	20
Al-3%Ti-0.30%C	900	800	900	1300	20	20
Al-3%Ti-0.45%C	900	800	900	1300	20	20
Al-3%Ti-0.60%C	900	800	900	1300	20	20
Al-3%Ti-0.75%C	900	800	900	1300	20	20
Al-3%Ti-0.90%C	900	800	900	1300	20	20

Table III. Grain Refiner Compositions and Experimental Parameters for the Master Alloy Method

Grain refiner composition	Addition and casting temperature (°C)	Holding time (min)
Al - %3Ti	1300	20
Al - %3Ti - %0.15C	1300	20
Al - %3Ti - %0.30C	1300	20
Al - %3Ti - %0.45C	1300	20
Al - %3Ti - %0.60C	1300	20
Al - %3Ti - %0.75C	1300	20
Al - %3Ti - %0.90C	1300	20

For both methods, graphite crucible and stainless steel rod was coated with boron nitride to ensure that molten metal is purified from impurities. Graphite crucible, mold and foil wrapped carbon powder were preheated at 200°C for 1 hour.

In the master alloy method commercial pure aluminum and Al-%10Ti were melted together up to 1300°C. The melt was held at constant temperature for 15 minutes and after this period, carbon was added immediately by light stirring. The stirring was maintained for 5 minutes to obtain homogenous solution (stirring process was skipped for the refiner without elemental carbon (Al-3%Ti)). Subsequently, the melt was held at the same temperature (1300°C) for 20 minutes and then poured into the mold which had been preheated to 200°C. The aim of this holding process was to form TiC particles.

Flux method (addition of K_2TiF_6 and carbon) was investigated for two different techniques which were mentioned as Experimental 1 and Experimental 2 in Table II. For Experimental 1, carbon and K_2TiF_6 were mixed for 2 hours then powder mixture was preheated at 200°C for 1 hour for humidity elimination in powder mixture. In this method commercial pure aluminum was melted at 900°C, and powder mixture of carbon and K_2TiF_6 were added to the melt by light stirring. The temperature was held constant at 900°C during adding process. When the stirring process was completed, melt was held for 20 minutes at 900°C and casting was made into the preheated mold. For Experimental 2, the melting and addition temperature of powder mixture in melt was chosen as 800°C as a difference from Experimental 1. After addition of powder mixture, the melt temperature was increased to 1300°C and held stable at this temperature for 20 minutes before casting.

In grain refiner performance tests for both methods, commercial pure aluminum was melted in a boron nitride coated graphite crucible by induction furnace. Melt temperature was adjusted to 750°C throughout the experiment. Grain refiner additions were made with 0.2% weight of commercial pure aluminum and inoculated into the melt when desired temperature was reached and then was stirred for 2 minutes with boron nitride coated graphite rod. After stirring, melt was held for 3 minutes then casting was made into a plaster mold which was preheated to 750±5°C as indicated Alcoa Cold Finger (ACF) test procedure[13].

The samples which were prepared longitudinally by cutting into half were surface-machined. This surface was macro etched using Poulton's reagent. To measure the grain size, a sample was cut from 25.4 mm distance below the copper chill [14]. Prior to each experiment, the surface pretreatment of specimens were performed by mechanical polishing. Finally, these samples were etched by HF 0.5%. The grain size of each sample was studied to determine the most effective grain refiner.

Results and Discussion

The best performance was accomplished with the Al-3%Ti alloys prepared by the flux method. The grain refiners produced by the flux method did not give stable results. On the other hand, the results which were obtained by the master alloy method were quite stable. Figure 1 shows all the results for comparison.

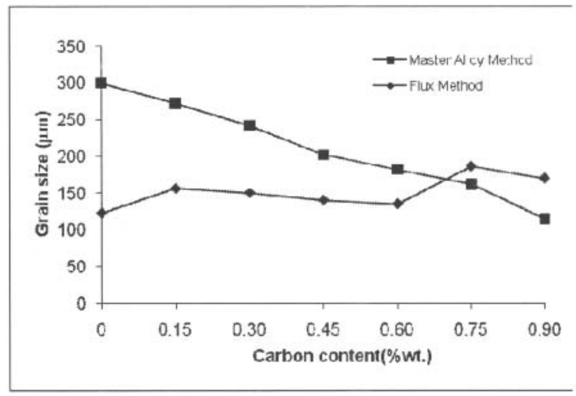

Figure 1. Grain size variation versus carbon content for different production method.

The micrographs shown in Figures 2-8, allow us to compare the effect of the grain refiners used in this study. Structural variables belonging to samples that were produced by both of the elemental carbon and flux methods can also be seen in these figures.

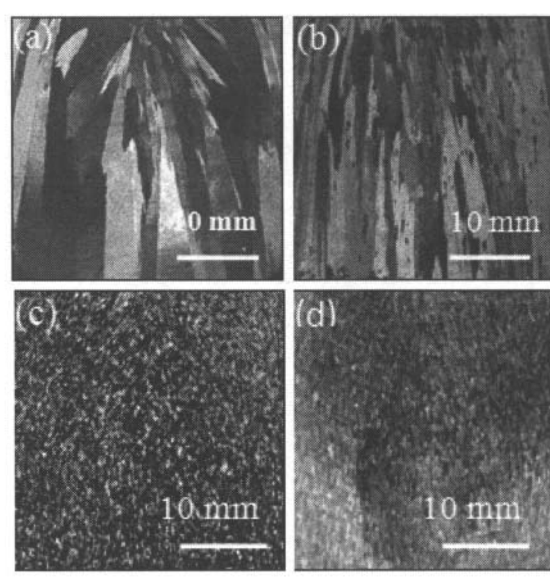

Figure 2. (a) Non refined AA1080 alloy, AA1080 including Al-3%Ti refiner produced at (b) 900°C, (c) 1300°C with the flux method, (d) 1300°C with the master alloy method

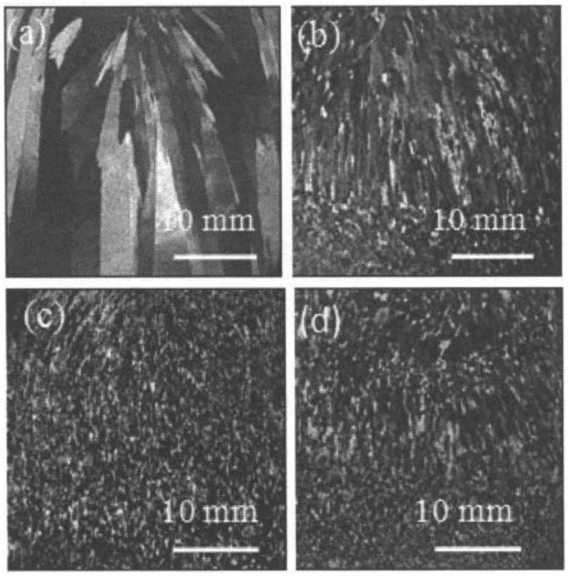

Figure 3. (a) Non refined AA1080 alloy, AA1080 including Al-3%Ti-0.15%C refiner produced at (b) 900°C, (c) 1300°C with the flux method, (d) 1300°C with the master alloy method

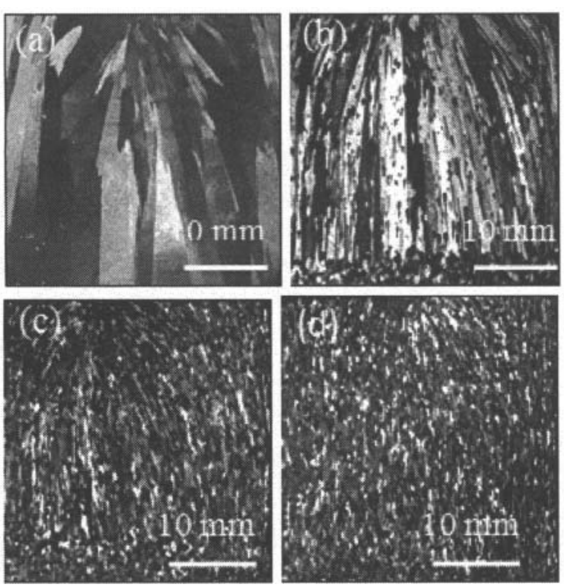

Figure 4. (a) Non refined AA1080 alloy, AA1080 including Al-3%Ti-0.30%C refiner produced at (b) 900°C, (c) 1300°C with the flux method, (d) 1300°C with the master alloy method

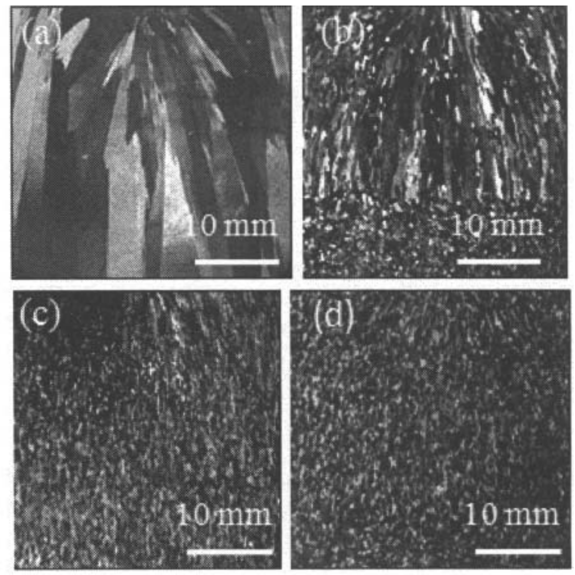

Figure 5. (a) Non refined AA1080 alloy, AA1080 including Al-3%Ti-0.45%C refiner produced at (b) 900°C, (c) 1300°C with the flux method, (d) 1300°C with the master alloy method

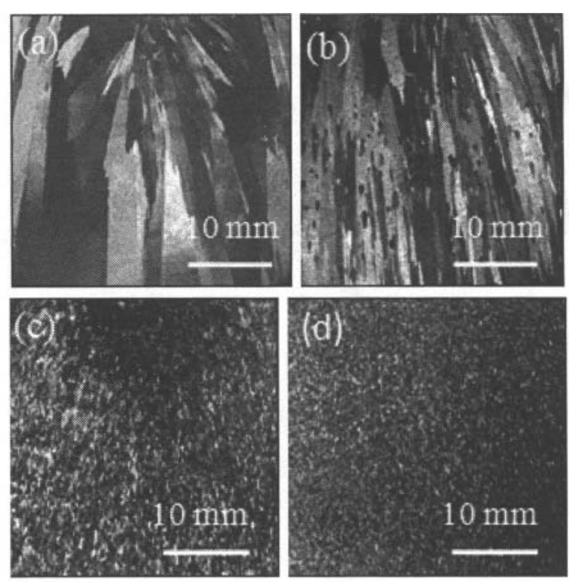

Figure 6. (a) Non refined AA1080 alloy, AA1080 including Al-3%Ti-0.60%C refiner produced at (b) 900°C, (c) 1300°C with the flux method, (d) 1300°C with the master alloy method

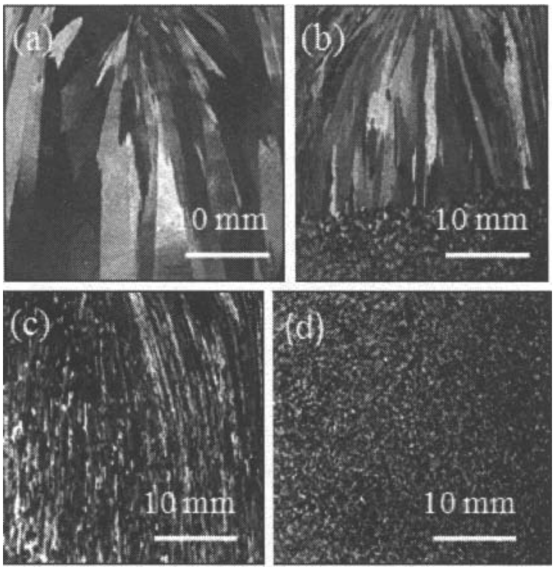

Figure 7. (a) Non refined AA1080 alloy, AA1080 including Al-3%Ti-0.75%C refiner produced at (b) 900°C, (c) 1300°C with flux method, (d) 1300°C with the master alloy method

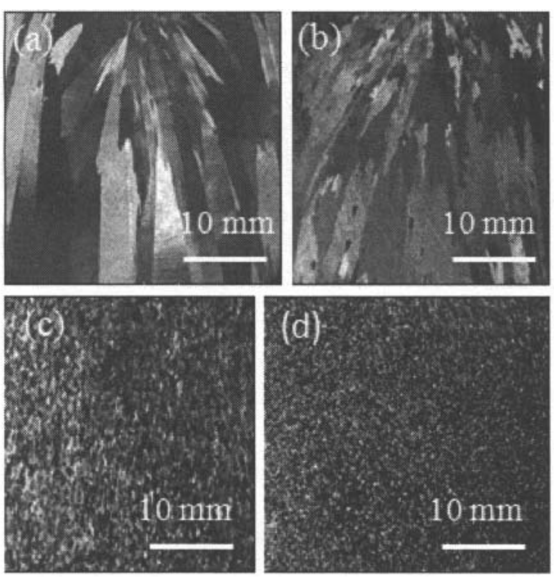

Figure 8. (a) Non refined AA1080 alloy, AA1080 including Al-3%Ti-0.90%C refiner produced at (b) 900°C, (c) 1300°C with flux method, (d) 1300°C with the master alloy method

The grain refiners produced by flux method at 900°C were not effective in reducing grain size. On the other hand, the grain refiners produced at 1300°C with the same method were found effective, but the grain reduction intensity was not dependant on the carbon content. Figure 1 implies this result. The production temperatures directly affect the grain refiner performance. Gezer et al.[6] have implied in their study that in the grain refiner samples produced at 900°C, had blocky type Al_3Ti morphology which is the result of low production and casting temperatures. This morphology changes to needle-like at high production temperatures as stated in [6]. The change of the morphology is related with dissolving behavior of the Al_3Ti particles with increasing temperatures.

In Figures 2-8 performance of all refiners are compared. Because of the lower amounts of Al_3Ti phase and by the absence of TiC particles, the refiners produced at 900°C had a poor refining performance. In the master alloy method, reduction of grain size is detected when the Ti:C ratio of the grain refiner is increased above the TiC stoichiometry. When the refiners produced in 1300°C is examined, the grain refiner becomes more efficient when the carbon amount increases. Increasing TiC amount can be the main reason for this situation. However, at grain refiner production small inefficiencies occurred during carbon addition.

Conclusions

In this study, Al-Ti-C grain refiners were produced by the master alloy method and the flux method. The grain refiner performances were compared. By these methods, effective grain refinement production is possible. Conclusions are as below:

- It is possible to produce Al-Ti-C grain refiners with the elemental carbon and flux methods,
- Grain refiners produced by the master alloy method had good performance when compared to flux method,
- Al_3Ti and TiC were detected as effective phases in grain refinement process,
- Refiners produced with flux method at 1300°C had relatively good performance when compared to 900°C productions by the presence of high amounts of Al_3Ti and TiC phases,
- In Al-Ti-C refiners produced with master alloy method at 1300°C, grain size decreased as the carbon content increased,
- Al-3%Ti-0.60%C, Al-3%Ti-0.75%C and Al-3%Ti-0.90%C compositions produced with master alloy method at 1300°C were determined as the most effective grain refiners.

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