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DEVELOPMENT OF Al-TiC ALLOYS USING POWDER METALLURGY AS GRAIN REFINERS FOR ALUMINIUM AND ITS ALLOYS

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Grain refining, Al-TiC, Aluminium alloys, Powder metallurgy

Abstract

Al-Ti-C master alloys have been widely investigated for many years as grain refiner for aluminium and its alloys. In this work, the Al-Ti-C master alloys are synthesized using powder metallurgy technique through the mixing of aluminium and TiC powders with different TiC contents 3.75 (3), 5(4), 6.25(5) and 7.5(6) Wt% TiC(Wt% Ti). The mixing powders with different contents of TiC were pressed in cylinder shape. The pressed specimens were sintered from 450 ^{o}C in a tube furnace under argon atmosphere for 2 hrs. The produced alloys before and after sintering are examined using SEM, EDX and XRD. The results indicate that, the Al-TiC alloy containing fine TiC particles dispersed in all matrix was successfully prepared. The prepared Al-TiC alloys with different contents of TiC were evaluated using the KBI test mold as grain refiner for pure aluminum and its alloys. The results indicate that the prepared Al-TiC master alloy is high grain refining efficiency for pure aluminum and its alloys.

1. Introduction

Aluminium castshop widely uses different types of grain refiners to promote a fine equiaxed grain structure with improved mechanical properties [1]. The cast of Aluminium alloy ingots is the first stage process for producing wrought products. The uses of grain refiners to the molten aluminium prior to casting have supportive effects, particularly reduced cracking rates, improved mechanical properties, increased casting speeds, removal of feather crystal, and formation of an equiaxed cast structure. [2]. The used grain refiners for casting aluminium alloys are Al-Ti, Al-Ti–B and Al-Ti-C, however Al-Ti–B alloys are more commonly used than the other alloys [3]. Although only a small amount of grain refiner add into the melt causes a dramatic effect on the cast structure, however the grain refining effect fades with increasing holding time [3, 4]. There are various explanations about this fading phenomenon and its causes. One theory of fading is the idea that fades occurs due to the equilibration of Ti between the aluminide and the bulk melt. Another theory suggested that in the presence of boron, Al-Ti-B alloys perform better due to, the shift in the peritectic point towards lower Ti in the Al-Ti phase diagram [5]. Also the effect of alloying elements enhances or fades the grain refining efficiency, elements such as Mg, Si, Zn and Cu enhance grain refinement by constitute under cooling mechanism, whereas Zr and Cr have a poisoning effect while Mn has little effect in grain refining properties [6].

During the working in DC and continuous casting, the grain refining master alloys are added to molten aluminium at about 720 ^oC. The grain refining master alloys are added in the form of wire 9.5 mm in diameter. The rod is fed into the casting counter currently to the metal flow. The contact time usually between 30 sec to 10 min [7]. The type and composition of the used grain refiners are an important factor for refining process. Recently, Al-Ti-C master alloy as a grain refiner for aluminum and its alloys has been widely investigated to be an acceptable replacement for Al-Ti-B master alloys [8, 9]. Al-Ti-C grain refiners based on TiC are less prone to the disadvantages associated with TiB_2, which causes some quality problems in different products [10, 11]. the Al-Ti-C system has become of great interest in the grain refining industry, because it may be a viable alternative to the Al-Ti-B system.[12]. More recently there has been a growing interest in the development of technologies for Al composites reinforced with TiC particles because of good wettability with Al. Selcuk, et al., reported that, ‚Al–TiC composites have been manufactured by the dispersion of a master alloy in molten Al. The master alloy was made by the reaction of Al, Ti and C elemental powders where the Al content was controlled to form, isolated, spherical, micron-sized TiC particles [13]. The present work aims to prepare Al-TiC master alloys by powder metallurgy. The prepared alloys is examined for grain refining of aluminium and its alloys. An attempt to compareing between the produced alloys by this method and the commonly used Al-Ti-B master alloys.

2. Experimental methods

The materials used in this study are titanium carbide powder (TiC) and aluminium powder (Al) in the average size of 10 μm, and 59 μm respectively. The TiC and Al powders was mixed with a different content of TiC powder, 3.75, 5, 6.25 and 7.5 wt% TiC powder (3, 4, 5, and 6 Ti). The powder was uniformly mixed in cylindrical polyethylene bottle using Al_2O_3 ball with ball-to-powder ratio of 6:1 using horizontal mixing machine (Mechanical mixer, ABB ACS100), 150 rpm speed mixing time 6 hours. The mixed powders were pressed into a cylindrical form (10 mm in diameter, and up to 20 mm in height). The specimens were sintered at 450^oC for 2 hr in an argon atmosphere using a muffle furnace provided with self-propagating mode with a heated tungsten wire located on the top of the specimens. The sintered specimens were examined to determine the phase components and microstructures. The different kind of the synthesized Al-TiC master alloys wer xamined by X-ray diffraction (XRD) and scanning electron microscopy (SEM) provided with EDS for element analysis and back scattered technique.

For evaluating the efficiency of the prepared Al-TiC master alloy; appropriate quantities of pure aluminum 99.7% were charged in a silicon carbide crucible and placed in a verticle muffle furnace. The aluminum was melted at the required temperature, 720 ^oC, and held at this temperature for a prearranged time to ensure the homogeneity of temperature throughout the charge. The crucible was then taken out of the furnace and any oxides on the surface were skimmed off. A predetermined quantity of the prepared Al-TiC master alloy at a required titanium percentage was added to the molten aluminum, and stirred in the melt using a graphite rod for 10 seconds. The crucible was then put back into the furnace to keep the temperature of the melt, being held for the required holding time: 30 sec, 2 min, 5 min, 15 min, and 30 and then taken out of the furnace and poured in the KBI ring mold placed on the top of a silica foam block. After solidification is completed, the solidified samples are quenched in water and dried. The sample was etched for macrostructure examination using etching solution consists of HNO3, HCl, and HF; 43: 43: 14 respectively and then examined microscopically to determine the grain size using the linear intercept method, which is based on the number of grains that intercept with four crossing rays, which were counted using an optical microscope. A series of experiments were carried out to investigate the efficiency of the prepared Al-TiC master alloys. Four produced Al-TiC master alloys, TiC1, TiC 2, TiC3 and TiC4 (chemical composition shown in Table 1), were examined using a KBI ring mold test for the grain refining of commercial pure aluminum.

Table 1. The prepared Al-TiC master alloys with different Ti content

No.	Ti	C	TiC	Al%
TiC1	3	0.75	3.75	Rem.
TiC2	4	1.0	5	Rem.
TiC3	5	1.25	6.25	Rem.
TiC4	6	1.5	7.5	Rem.

3. Results and discussion

The X-ray diffraction analysis for the produced Al-TiC master alloy containing 7.5%TiC was carried out from 30 to 80 degree. Figure 1 shows the phase identifications for this pattern indicated that only two phases were appeared; pure Al and TiC. Although the mixing between Al powder and TiC powder was don for 6 hr, but no reaction or phase transformation was occurred.

Figure 2 shows SEM micrographs of the prepared Al-TiC master alloy containing 3.75%TiC by powder metallurgy. From this figure, it can be seen that a homogeneous distribution of TiC particles (White) dispersed within dark gray matrix. The size of TiC particles within Al-TiC master alloys ranged from 0.5 to 3 μm. The point analysis of the white phase at point 1 using energy dispersive spectrometer (EDS) Figure 3 proved that the chemical analysis of the white phase is 80.23 Wt% Ti and may be the reminder is carbon (19.77%), this analysis is much closer with the chemical composition of the TiC compound. The result confirmed with the the result obtained from XRD analysis. While the point analysis of the dark gray phase at point 2 using EDS Figure 3. is 0.3 Wt% Ti and 99.7 aluminium. This means that the white phase is TiC and the dark gray phase is aluminium matrix.

Fig. 4 indicates the X-ray mapping images and the distribution of Al, and Ti in the Al-TiC master alloy prepared by powder metallurgy. From this figure it can be seen that SEM photograph containing TiC within the aluminium matrix as mentioned in Figure 4 (a). But the left micrograph Figure 4 (b) indicates the only Al particles or atom (gray color) and the place of Ti particles (black) like shadow. The bottom micrograph Figure 4 (c) indicates only Ti particles (white color) and the place of aluminium particles black color. The carbon is not included because its atomic number is less than 7 and the EDX instrument measures more than atomic number 7. The all matrix analysis using EDX proved that the analysis of aluminium is 94.75 wt% Al and the Ti is 4.31 Wt% Ti and may the carbon is the reminder.

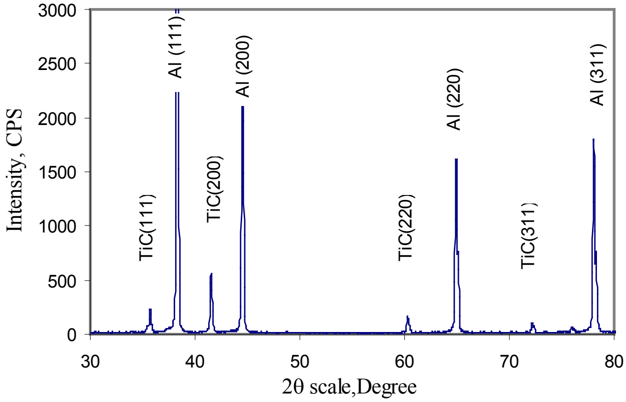

Figure 1. XRD pattern for the prepared Al-TiC containing 7.5 wt% TiC

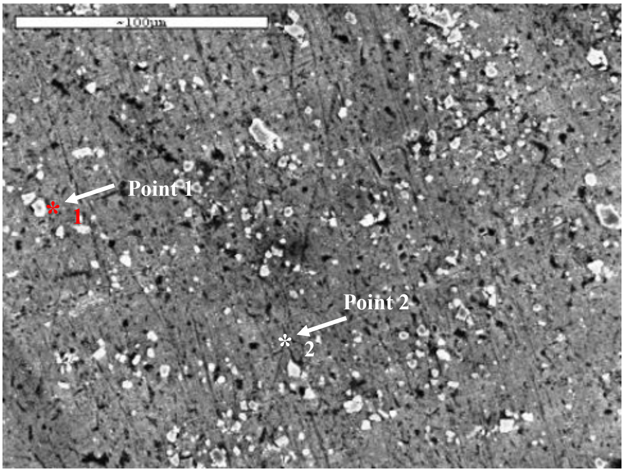

Figure 2. SEM micrograph for the prepared TiC1 containing 3.75Wt.% TiC

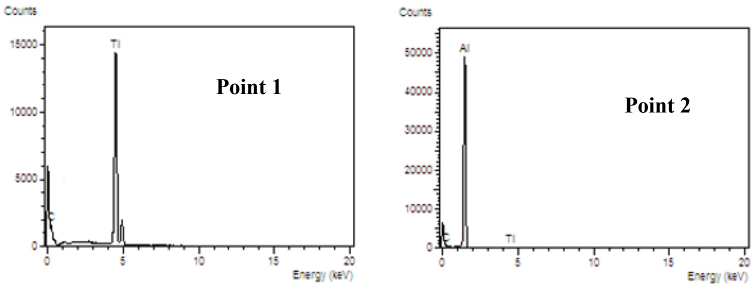

Figure 3. :EDS analysis for the prepared Al-TiC at point 1, 2 indicated above in Fig.2

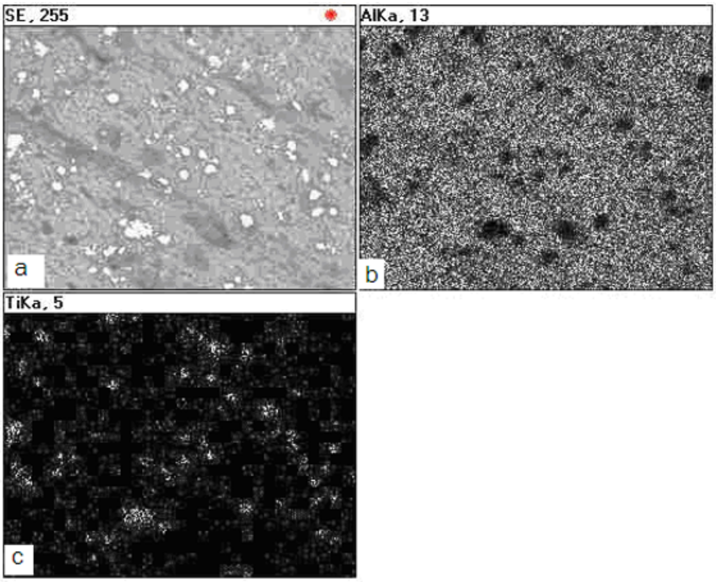

Figure 4. X-ray mapping images for the prepared TiC2 containing 5%TiC

<u>Evaluation of the Al-Ti-C Master Alloy</u>

<u>1- Comparison between the produced Al-TiC master alloys</u>

The comparison between the produced Al-TiC master alloys using powder metallurgy represented (TiC1, TiC2, TiC3, and TiC4) and the common type Al-5Ti-1B master alloys as grain refiners. The samples examined as grain rfiners for commercially pure aluminum (99.7 %Al). The choosing of pure Al for refining test because the refining of pure aluminium is more difficult than the other types of aluminium alloys such 6063 and 5052. The processing parameters of refining test are; pouring temperature of (720 ^oC), addition of Ti weight ratio of 0.01 %, and holding time of 2 minutes. The choice of these conditions is in accordance with previous results, which could be the most suitable conditions for the grain refining processes [15]. Figures 5, 6 shows the photomacrographs and the variation of the grain sizes of the commercial pure aluminum (99.7 % Al) refined with different types of the prepared grain refining and comparing with Al-5Ti-1B grain refining. It can be see that, the grain sizes of pure aluminium refined using TiC ranged from 90 μm for TiC2 to 120 μm for TiC4, and then the best grain refining efficiency is TiC2. Because the decreasing of the concentration of TiC in the prepared master alloys increase the solubility of the grain refiner in the molten aluminium leads to increasing of nucleating sites in the molten aluminium. While, the value of grain size of pure aluminium refined using Al-5Ti-1B is 170 μm is more than the grain sizes of pure Al refined by the prepared Al-TiC master alloys. The increasing of the grain refining efficiency of the produced Al-TiC master alloys than the fficency of Al-5Ti-1B master alloy is due to the decreasing of particles size of TiC in the prepared Al-TiC ranged from 0.5 to 3 μm as in Fig. 2, but the TiB_2 particles agglomerates with other leads to decrease the nucleating sites. Another reason, TiC is a good nucleating for Al, and this could be due to the fact that TiC has the same FCC structure [15].

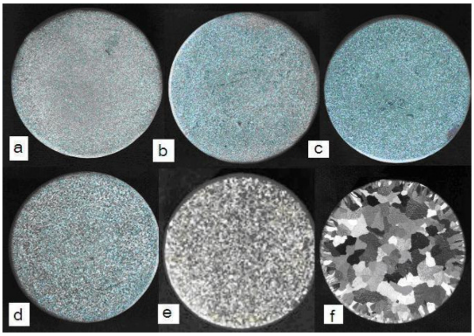

Figure 5. Photomacrographs for the refined commercially pure Al using :
a) TiC1 b) TiC2 c) TiC3
d) TiC4 e) Al-Ti-B f) Without refining

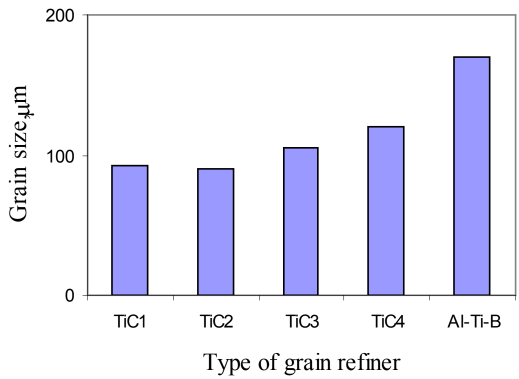

Figure 6. The effect of grain refining type on the grain size, of pure aluminium

<u>2. Effect of addition rate</u>

The effect of the addition rates of TiC2 and Al-Ti-1B master alloy to molten pure aluminium on the grain refining efficiency was studied at different Ti contents. A series of experiments were conducted at titanium contents of (0.0025, 0.005, 0.0075, 0.01, and 0.015 Wt% Ti in the refined pure aluminium at constant temperature (720 ^{o}C) and holding time 2 minutes. Fig.7 shows the photomacrographs of the refined commercially pure aluminum (99.7 Wt% Al) using TiC2 and Al-Ti-B grain refiners. It can see the difference in grain size of the refined aluminum at different Ti contents and diferent grain refiners compared with the as-cast aluminum without refining. Fig. 8 shows the variation of the grain sizes of the commercial pure aluminum (99.7 % Al) refined with different addition rate of TiC2 and Al-Ti-B master alloys. It can be noticed that, the grain size decreases almostly at the same trend with the increasing of addition rate at using the two kinds of grain refiners TiC2 and Al-Ti-B. It may be due to the increase of the nucleating sites with the increase of the Ti content according to the carbide and pretectic theories. Also from Fig. 8, the comparison between the common type Al-Ti-B and the prepared TiC2 grain refiners at the same conditions indicated that the prepared TiC2 grain refinr is more efficient than the Al-Ti-B grain refiner this due to the same couses before mentioned.

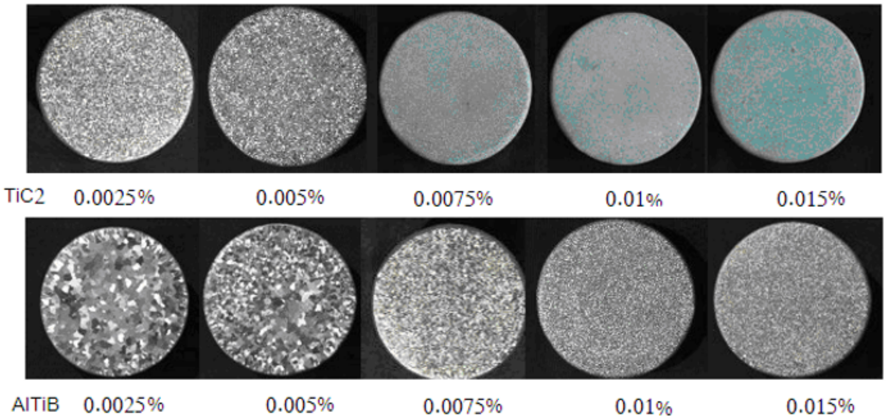

Figure 7. Photomacrographs for the refined commercially pure Al using TiC2 master alloys with different addition ratios.

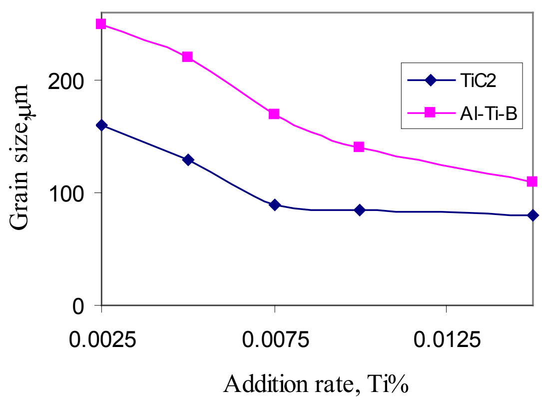

Figure 8. Effect of addition rate on the grain size of the pure aluminum refined with the prepared TiC2 and Al-Ti-B master alloys

3. Effect of holding time

The effect of holding time on the grain refining efficiency of commercially pure aluminum (99.7 Wt% Al) using TiC2 grain refiner was studied in a range of 0.5 to 30 minutes. The experiments were carried out at constant temperature 720^{o}C, addition rate 0.01% Ti with different times of 0.5, 2, 5, 15, and 30 minutes. Figure 9 shows the photomacrographs of the refined commercially pure aluminum (99.7 Wt% Al) using TiC2. It can see the difference in grain size of the refined aluminum at different holding times. Figure 10 illustrates the relationship between the grain size of the refined commercially pure aluminum using TiC2 vs holding time. At the start of nucleation, the grain size decreases sharply with the holding time and then increases slowly. This means that the nucleating sites start to disperse into the molten aluminium from 0 minutes reached to maximum dispersion at 2 minutes, and then Al-TiC grain refiner fades with the increase of holding time as results of precipitation or agglomeration of the nucleating sites.

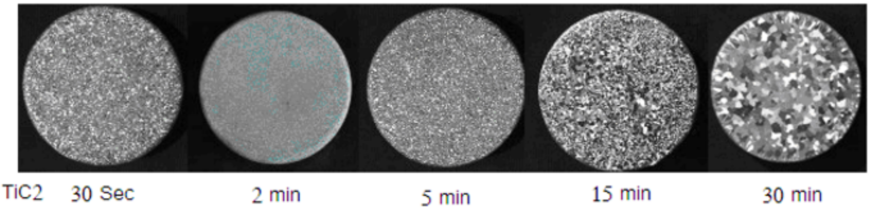

Figure 9. : Photomacrographs for the refined commercially pure aluminum using TiC2 master alloys with various holding times.

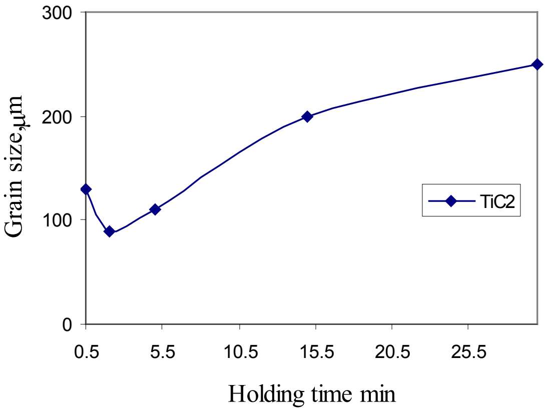

Figure 10. Effect of holding time on the grain size of 99.7 %Al refined with TiC2 master alloys.

From the previous results, the size of TiC particles within Al-TiC master alloys ranged from 0.5 to 3 μm, it play the same role as the TiB_2 particles in Al-Ti-B master alloys, to act as nucleating sites in the aluminum matrix. In addition, TiC is a good grain refiner for aluminium and its alloys, and this could be due to the same FCC structure [15]. TiC are less level to the disadvantages coupled with TiB_2 present in Al-Ti-B grain refiner such as some damages or scratches on the surfaces of extruded sections caused by agglomerates of TiB_2 particles [14]. Thus, Al-TiC grain refiner could be preferred in some applications and practices over Al-Ti-B grain refiner. The holding time plays an important role in the case of both dissolution and settling of TiC particles. The results make the Al-TiC master alloys prepared by powder metallurgy very suitable to be used in casting practices with very small holding times such as in case of DC casting

4. Conclusion

1. A reasonably novel technique has been successfully prepared Al-TiC master alloys using powdered aluminium and powdered TiC by powder metallurgy process.
2. According the XRD, SEM, X ray mapping, the TiC particles are homogeneous distribution and dispersed within aluminium matrix, and the size of TiC particles within Al-TiC master alloys ranged from 0.5 to 3 μm.
3. The prepared Al-TiC master alloys are an efficient grain refiner for pure aluminum and its alloys compared with the common type Al-5Ti-1B master alloy.
4. The proper conditions for the evaluation of the efficiency of Al-Ti-C master alloys to obtain a minimum grain size are temperature (720 ^{o}C) and holding time 2 minutes. And addition rate 0.01 %Ti.

5. References

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