Grain Refinement of Commercial EC Grade 1370 Aluminium Alloy for Electrical Applications

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Abstract The purpose of the present study was to investigate the effect of different grain refiners' additions on structure and electrical conductivity of commercially electrically conductive (EC) grade 1370 aluminium alloy. A series of controlled lab scale experiments were carried out by addition of 0.1 wt. percentage Al-5Ti-1B, Al-3Ti-1B, OPTIFINE, TiBloy, and Al-3Ti-0.15C, grain refiners to molten aluminium at 730 ± 5 °C. The macro structural analysis showed that 0.1 Wt % of TiBloy, Al-5Ti-1B, Al-3Ti-1B, and OPTIFINE did not grain refine commercial EC grade aluminium. The Al-3Ti-0.15C master alloy showed, however, good grain refining performance, giving an equiaxed structure. Fading effect was observed in the samples grain-refined by Al-3Ti-0.15C master alloy. The electrical conductivity of all the grain-refined samples decreased between 0.02-0.75 ± 0.3 % IACS, compared to the reference samples of commercial pure aluminium. However, the electrical conductivity increased slightly by holding the melt, after the addition of the grain refiners, for 90 minutes, that might be due to settling of the particles by grain refiner additions.

1. Introduction

Copper high-voltage transmission lines are replaced by aluminium for two reasons. First, aluminium is cheaper (per ton) as compared to copper, so it is more economical on those multi-mile distances. Second, fewer power pylons are needed since aluminium is much lighter than copper. In addition, decent mechanical strength and persistent corrosion resistance are other properties that have made aluminium a feasible choice for electrical conductivity applications [1, 2].

G. G. Gauthier investigated the effect of different elements on the electrical conductivity of high-purity aluminium (99.99%) [3]. It was shown that titanium, vanadium, magnesium and chromium have an adverse effect on the electrical conductivity of high-purity aluminium and reduce it significantly. The electrci conductivity reduction is due to electron scattering via thermal vibration of the crystal lattice (phonons) and the presence of structural imperfections [4]. Table 1 shows the influence of transition metal elements on the electrical conductivity of pure aluminium in the form of solute and precipitate impurities [5]. As shown in the Table 1, impurities in the form of precipitation particles cause minor reduction in conductivity.

Table 1. Influence of solute and precipitate impurities on electrical conductivity [5].

		Estimated average decrease in % (IACS* per Wt. %)	
Element	**Max solubility in Al (%)**	**In Solution**	**Precipitated**
Fe	0.052	29	1.2
Si	1.65	16	1.8
Ti	1.0	31	2.5
V	0.5	34	5.5
Cr	0.77	36	3.7
Zr	0.28	23	0.9

***International Annealed Copper Standard**

Normally the aluminium alloys used for electrical conductivity application are not grain-refined as the addition of commercial grain refiners may adversely affect the electrical conductivity by adding impurities, especially Ti, to the melt. However, non-grain-refined aluminium alloys may end up to bar fracture and cracks during post casting operations.

It is of great interest to aluminium industry to find out the level of grain refiner without having adverse effect on the acceptable levels of electrical conductivity. It is also very beneficial to identify an efficient grain refiner for the commercial electrically conductive aluminium grades, which not only effective to produce fine equiaxed microstructure, but also not decrease the electrical conductivity to under the acceptable levels for the electrically conducive grade aluminium. In other words, finding the optimum balance between the grain refiner addition and the electrical conductivity is desired to enhance the properties of the final product.

The objective of the current work is to investigate the effect of five commercially grain refiners for aluminium alloys (Al-5Ti-1B, Al-3Ti-1B, OPTIFINE, TiBloy, and Al-3Ti-0.15C) on structure and electrical conductivity of the commercial electrically-conductive grade 1370 aluminium alloy.

2. Experimental

The commercially pure aluminium (1370) and the grain refiners i.e. Al-5Ti-1B, Al-3Ti-1B, TiBloy, and Al-3Ti-0.15C, for the current work are provided by Norsk Hydro Karmøy and London Scandinavian Metallurgical (LSM), respectively. OPTIFINE (Special Al3Ti1B) was supplied by KBM. Table 2 shows the chemical composition of the commercial pure aluminium and the master alloys used in the experiments. Two kilograms of sectioned aluminium ingots were heated to 730 ± 5 °C (temperature of casting) in a resistance furnace. The melt temperature was controlled by Comark C9001 thermometer. A sample was taken before addition of the grain refiner. At 30, 60 and 90 minutes after the addition of the grain refiner three more samples were collected, as well. Details of grain refinement additions conducted in the present study are summarized in Table 3.

Table 2. Chemical composition of commercial pure aluminum and grain refiners (mass fraction, %).

Material	**Composition (Wt. %)**									
	Ti	**B**	**C**	**Fe**	**Si**	**V**	**Cu**	**Mg**	**Zn**	**Al**
Commercial pure Al	0.003	0.008	-	0.15	0.007	0.003	0.01	0.01	0.02	99.7
Al-5Ti-1B	5.0	1.0	-	-	-	-	-	-	-	-
Al-3Ti-1B	3.0	1.0	-	-	-	-	-	-	-	-
OPTIFINE	3.0	1.0	-	-	-	-	-	-	-	-
TiBloy	1.6	1.4	-	-	-	-	-	-	-	-
Al-3Ti-0.15C	3.0	-	0.15	-	-	-	-	-	-	-

Table 3. Addition level of the grain refiners and the time interval between taking the samples.

		Elemental addition level Wt.%		
Grain refiner	**Addition level of grain refiner, Wt. %**	**Ti**	**B or C**	**Time interval for sampling (min)**
Al-5Ti-1B	0.1	0.005	0.0001	0, 30, 60, 90
Al-3Ti-1B	0.1	0.003	0.0001	0, 30, 60, 90
OPTIFINE	0.1	0.003	0.0001	0, 30, 60, 90
TiBloy	0.1	0.0015	0.0013	0, 30, 60, 90
Al-3Ti-0.15C	0.1	0.003	0.00015	0, 30, 60, 90

A small graphite crucible with dimensions of 48 mm inside diameter, 50 mm outside diameter and 60 mm height was used for taking a small portion of the melt from the crucible inside the electrical resistance furnace. The graphite crucible was pre-heated in a furnace for 5 minutes at 730 °C and directly floated on the surface of the melt for 3-4 minutes. The molten aluminium was poured immediately into a cylindrical steel mould with the dimensions of 90 mm of outside-diameter, 40 mm of inside-diameter, 90 mm of height and 55 mm of inside bore depth. The collected samples were sawn transverse in two at the position of 30 mm above the bottom surface where the sample had contact with mould. The upper parts were used for structural analysis and the lower part were sawn longitudinally in two for the electrical conductivity measurements. The upper section plane was ground, polished and etched for macrostructure analysis. The etchant´s composition was 50 ml HNO3 + 25 ml HCl + 50 ml distilled water. Cutting of the samples causes micro and macro structural damages. To make sure that macro structural damages do not cause too much measurement errors with electrical conductivity meter a fine grinding was carried out with 320-grit paper. Electrical conductivity of the samples was measured by Auto-Sigma 3000 (EC meter), with the resolution of 10 to 110 % IACS (International Annealed Copper Standard) and the accuracy of ±0.5 % IACS at 100 % IACS reducing to ±0.1 % IACS at 10 % IACS at 20 °C. For each sample nine readings were performed, and the average electrical conductivity was calculated. For each grain refiner, the average electrical conductivity vs. time between taking the samples was plotted.

3. Results and discussion

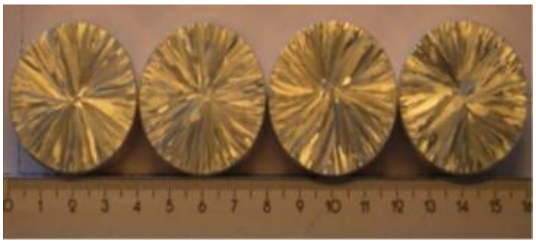
In the present work, the electrical conductivity and the structure of commercially EC grade 1370 aluminium (commercial pure aluminium) were studied under a series of controlled lab scale trails. Five different grain refiners (Al-5Ti-1B, Al-3Ti-1B, OPTIFINE, TiBloy, and Al-3Ti-0.15C) were added to the molten aluminium and samples were taken at defined time intervals. These samples were later sectioned, ground and macro-etched. Afterwards, the structure was analyzed by the use of a digital camera and the electrical conductivity was measured by Auto-Sigma 3000 (EC meter) at room temperature and was expressed as a percentage of the International Annealed Copper Standard (IACS %).

3.1. Structural analysis

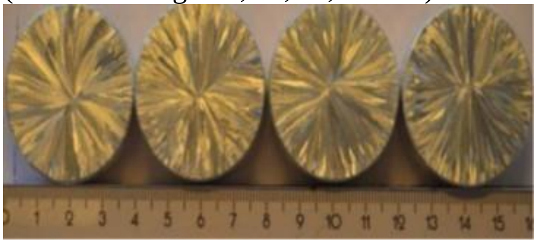
Fig. 1 (a-e) show the macrostructures of aluminium EC grade 1370 alloy grain-refined by Al-5Ti-1B, Al-3Ti-1B, OPTIFINE, TiBloy, and Al-3Ti-0.15C, respectively.

It is obvious that addition of 0.1% of Al-5Ti-1B, Al-3Ti-1B, OPTIFINE, and TiBloy doesnot have any noticeable grain refining effect on 99.7 % pure aluminium. However, Fig. 1 (e) shows that Al-3Ti-0.15C, with the same addition level (0.1%), is a more efficient grain refiner.

Jian-Guo et al. [6] showed that the grain refining performance of Al-5Ti-1B master alloy at the addition level of 0.2 % to the commercial pure aluminium was superior to that of Al-3Ti-0.15C. However, other researchers reported that Al-Ti-C master alloy is an efficient grain refiner for pure aluminium and its efficiency is comparable with that of the common type Al-5Ti-1B [7-10]. The present study supports the results of earlier studies [7-10], although the addition level of Al-Ti-C master alloy in the present study (0.1%) was lower than the addition of Al-Ti-C master alloys in these studies. The most important grain refinement mechanisms in aluminium may explain the role that TiC and TiB_2 particles play in the grain refinement of aluminium alloys. Cisse et al. hypothesized that the close lattice parameters between the solid matrix and the particle give a more efficient grain refiner [11]. TiC and α-Al have both fcc structure with the following crystal parameters and properties: Al lattice parameter = 0.405 nm, and density 2.70 $g\cdot cm^{-3}$; TiC lattice parameter = 0.43 nm, and density 4.91 $g\cdot cm^{-3}$ [7].



(a) 0.1% Al-5Ti-1B
(from left to right: 0, 30, 60, 90 min)



(b) 0.1% Al-3Ti-1B
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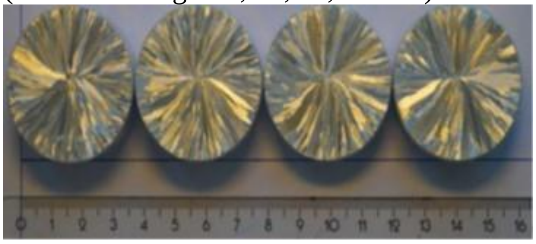
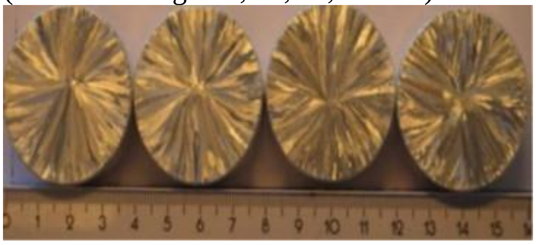
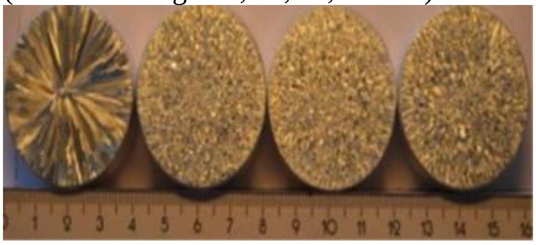


(c) 0.1% OPTIFINE
(from left to right: 0, 30, 60, 90 min)



(d) 0.1% TiBloy
(from left to right: 0, 30, 60, 90 min)



(e) 0.1% Al-3Ti-0.15C
(from left to right: 0, 30, 60, 90 min)

Fig. 1 (a-e). Macroscopic of the commercial aluminium grain-refined by the master alloys Al-5Ti-1B, Al-3Ti-1B, OPTIFINE, TiBloy, and Al-3Ti-0.15C.

TiB_2 has hcp crystal structure and lattice parameter of 0.303nm [7]. Comparing the crystal parameters and properties of TiB_2, TiC and α-Al suggests that TiC could be a better nucleating site for α-Al than TiB_2 particles are.

The common believe is that TiB_2 crystals are the nuclei for α-Al. Nevertheless, based on the Phase diagram/Peritectic theory, postulated by Crossley and Mondolfo [12], α-Al forms as a result of the peritectic reaction given below (Eq. 1) at 938 K (Fig. 2) [13].

$$L + TiAl_3 \rightarrow \alpha\text{-}Al \quad \text{Eq.(1)}$$

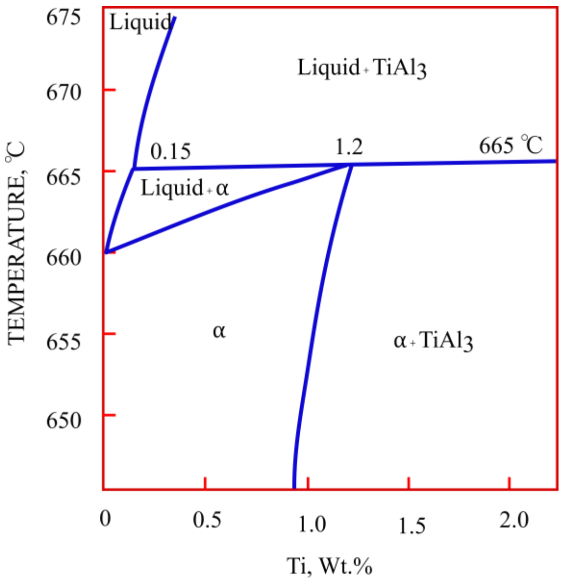


Fig. 2. Aluminuim rich end of Al-Ti binary equilibrium diagram [13].

Mohanty et al. [14] found that TiB_2 does not only nucleate grains, i.e. α-Al, but plays an indirect role in grain nucleation of aluminium alloys. They suggested that at Ti levels lower than 0.15 Wt. %, Ti diffuses towards the TiB_2 particles leading to segregation of Ti atoms on the TiB_2/melt interface. Consequently, a thin layer of $TiAl_3$ is formed on the surface of the TiB_2, which promotes the peritectic reaction and thereby nucleases the solid. In the present study the low concentration of Ti after addition of the grain refiners Al-5Ti-1B, Al-3Ti-1B, OPTIFINE and, TiBloy, could not form a sufficient activity gradient of Ti to promote the peritectic reaction.

It can be seen in the Fig. 1 (e) that by holding the molten aluminium fading happens. Holding the molten aluminium for a long time after grain refiner addition, leads to either particle settling and/or agglomeration.

3.2. Electrical conductivity analysis

The average electrical conductivity of the grain-refined aluminium as a function of time after the grain refining addition are plotted in the Fig. 3 (a-e). The time zero in each graph indicates the electrical conductivity of the reference sample (commercially pure aluminium before addition of the grain refiners). A trend line fits to the data points in the graphs.

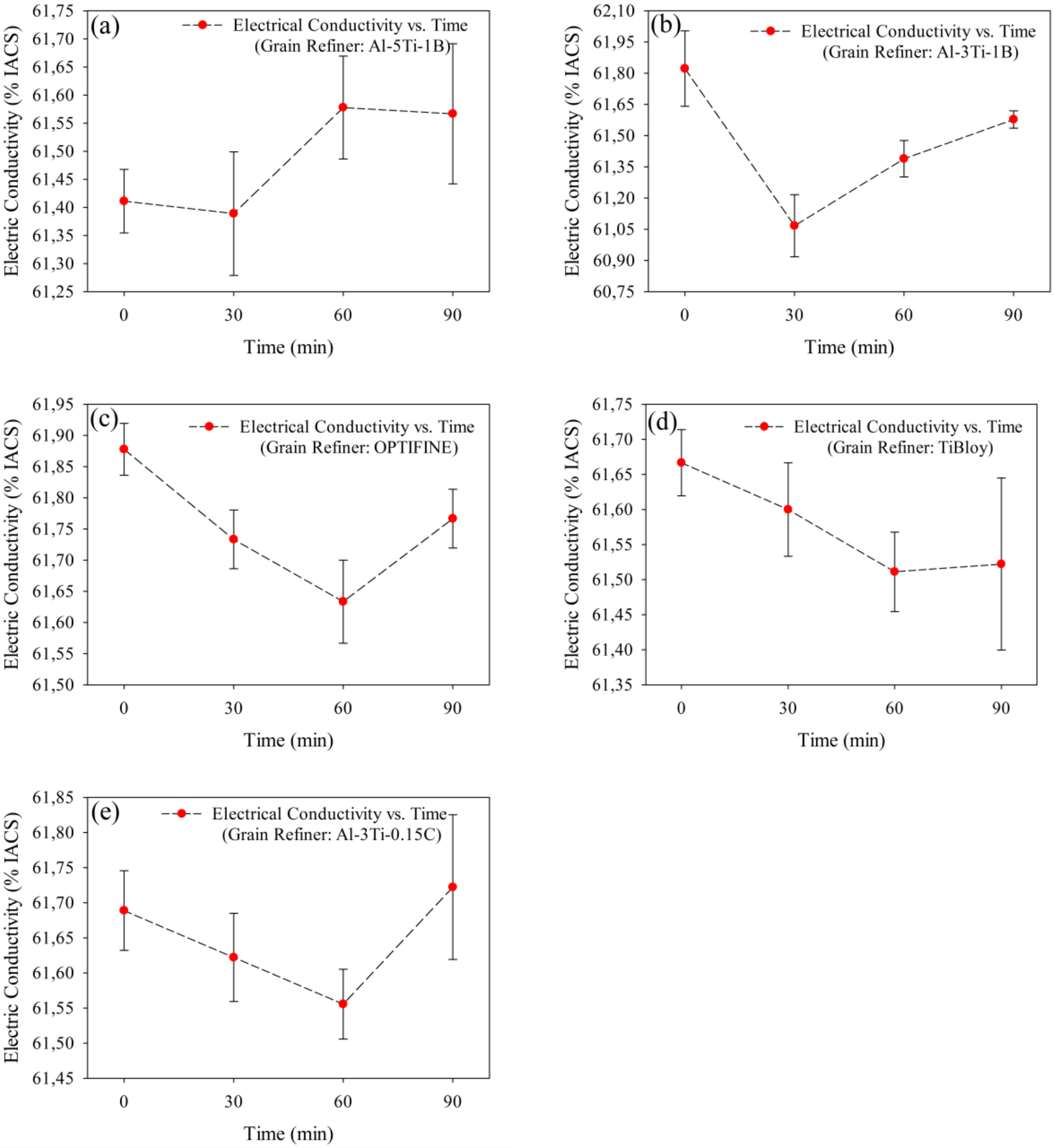


Fig. 3 (a-e). Electric conductivity of the grain-refined EC grade aluminium as a function of time after the addition of 0.1 % grain refiners (a) Al-5Ti-1B, (b) Al-3Ti-1B, (c) OPTIFINE, (d) TiBloy (e) Al-3Ti-0.15C

All the graphs show a slightly reduction of electrcial conductivity after addition of the grain refiners. However, the reduction of electrcial coductivity stopped after 30-60 minutes from the addition point and even increased moderately by holding the melt for a longer time. The reduction of electrcial conductivity may happen due to increasing concentration of dissolved Ti in the melt after addition of the grain refiners. Some of the released particles (TiB_2, TiC, $TiAl_3$ and AlB_2) in the molten aluminium may dissolve in the melt at high temperature and long holding times that leads to elevation of dissolved Ti concentration. In addition, these particles besides acting as heterogeneous nucleation sites, may also act as impurities and increase the resistivity of the EC grade aluminium alloys.

The primary decrease of electrical conductivity and the later increase might be due to the different settling behavior of the particles (TiB_2, TiC, $TiAl_3$ and AlB_2) introduced to the molten aluminium by different grain refiners. G. S. Vinod Kumar et al. [15] studied the settling behavior of TiB_2, TiC, $TiAl_3$ and AlB_2 particles and showed that TiC and TiB_2 had equal particle size distribution, 0.5-5 µm, but the TiB_2 particles were prone to form larger agglomerates than TiC and this led to their faster settling. It was also shown that $TiAl_3$ particles with wider size distribution, 2-50 µm, than TiB_2 and TiC particles, tend to settle in the first minutes of holding time. However, due to wider particle size distribution, the fine $TiAl_3$ particles takes longer time to settle in the liquid aluminium.

Al-5Ti-1B and Al-3Ti-1B master alloys normally contain crystals of TiB_2, $TiAl_3$ and AlB_2 [16]. The microstructural and elemental analysis of Al-5Ti-1B used in the present study confirms the presence of large blocky $TiAl_3$ and clusters of small TiB_2 particles in a matrix of α-Al (Fig. 4 and Fig. 5). As was mentioned earlier, these particles tend to settle very fast in the melt. As a result, the slight increase of the electrical conductivity after 30 minutes from the addition (in Fig. 3 (a) and 3 (b)), is assumed to be due to settling of the TiB_2 and $TiAl_3$ particles.

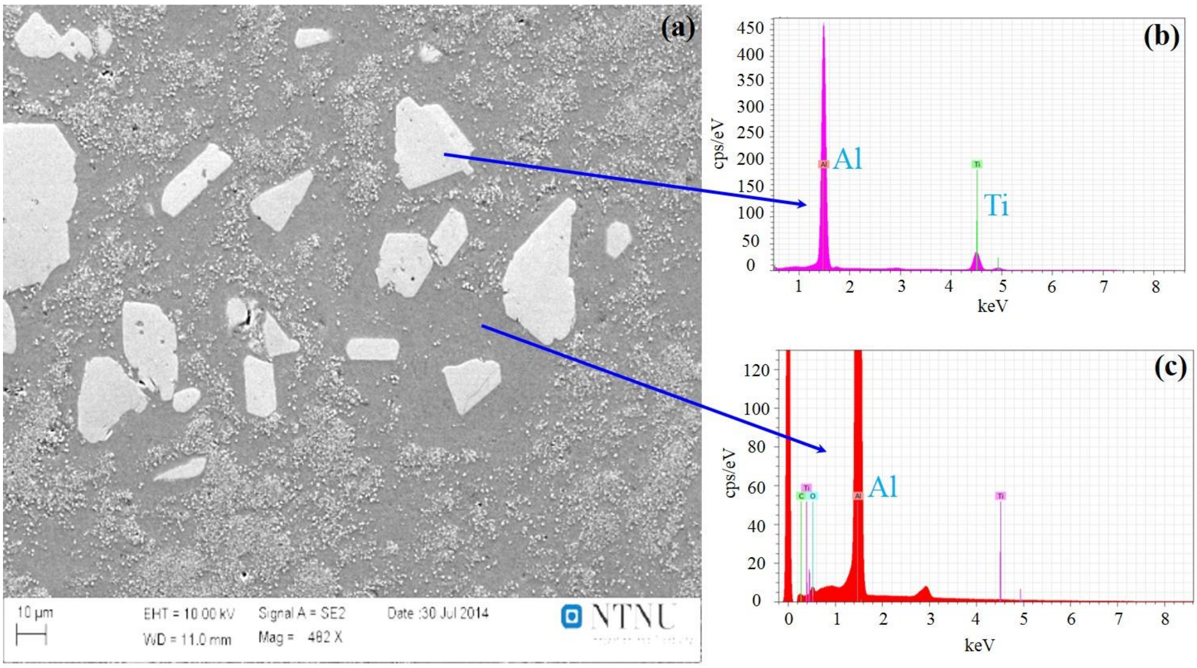


Fig. 4. SEM photomicrographs and Energy Dispersive Spectroscopy (EDS) of the matrix and large blocky particles in Al-5Ti-1B master alloy. (a) SEM photomicrographs of Al-5Ti-1B, (b) EDS of the large blocky particles, (c) EDS of the matrix.

The microstructural analysis of Al-3Ti-0.15C by SEM, Fig. 6 (a) and 6 (c), and their corresponding X-ray spectra (EDS), Fig. 6 (b) and 6 (d), show the presence of TiC and/or $TiAl_3$ particles in a matrix of α-Al. The present result is in agreement with the previous studies that reported the presence of the TiC and $TiAl_3$ particles in Al-Ti-C master alloys [7, 15, 17].

According to [15], the settling rate of TiC particles is lower than TiB_2 particles because they form smaller agglomerates. That might be the reason for the fact that electrcial conductivity of the samples grain-refined by Al-3Ti-0.15C decreased up to 60 minutes after grain refiner addition and then started to increase slightly when more particles settled down.

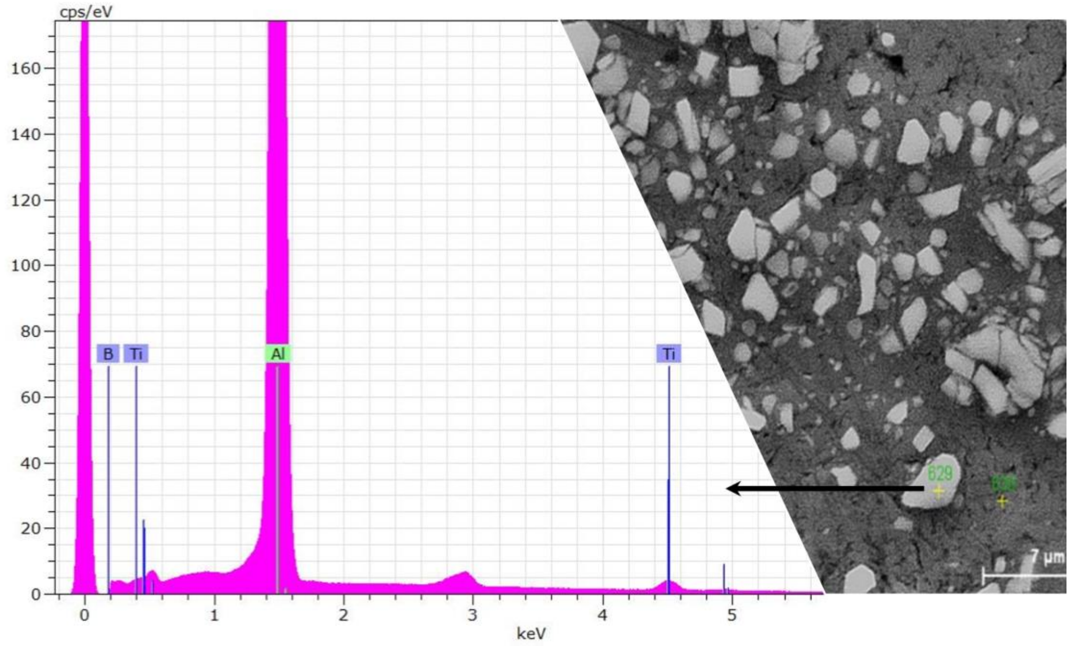


Fig. 5. SEM photomicrographs and Energy Dispersive Spectroscopy (EDS) of the small particles in Al-5Ti-1B master alloy.

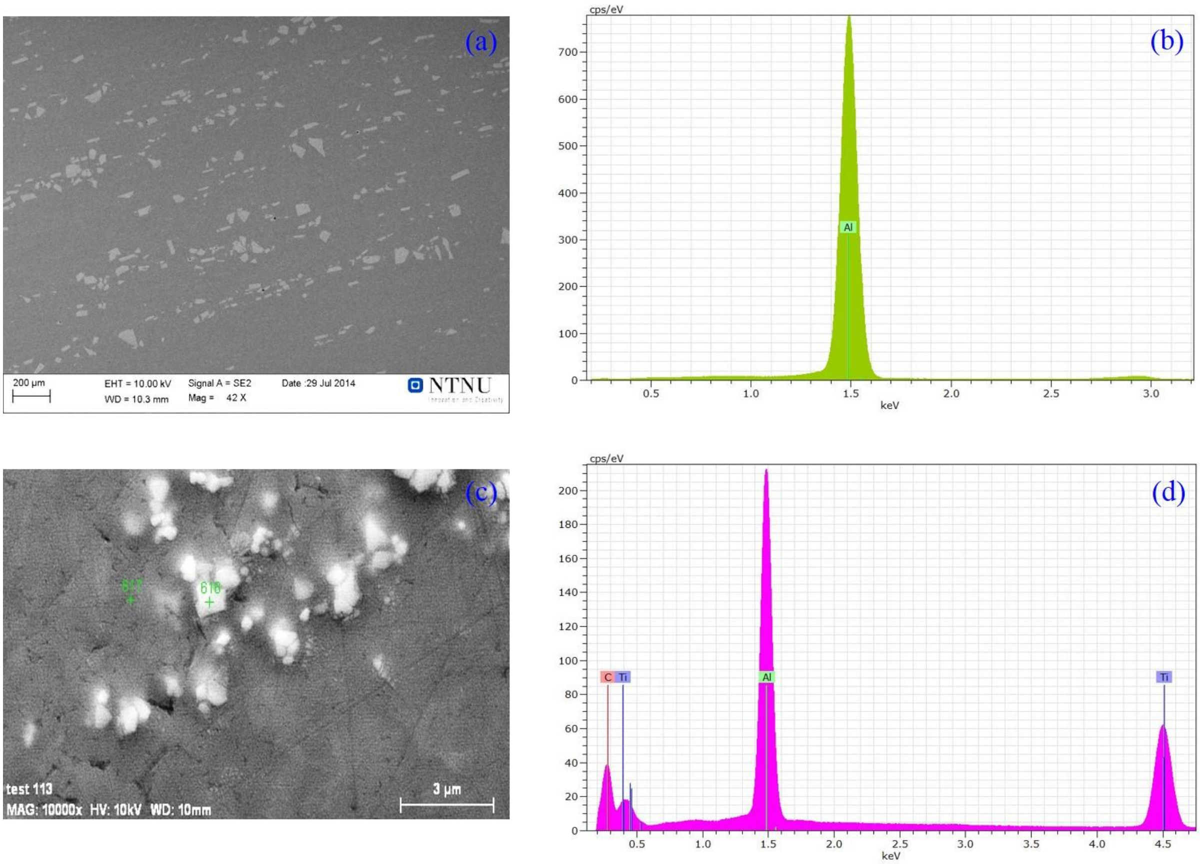


Fig. 6. SEM images and thier corresponding energy dispersive spectroscopy spectrums of Al-3Ti-0.15C master alloy showing TiC particles in a matrix of α-Al. (a) SEM of Al-3Ti-0.15C master alloy, (b) EDS of the matrix, (c) SEM of Al-3Ti-0.15C master alloy (d) EDS of the particles in Fig 6 (c).

TiBloy contains mixed boride particles, (Al,Ti)B_2 (instead of TiB_2 particles), which are smaller in size (0.05–1 µm) and have a lower density than TiB_2 particles in conventional Al–Ti–B master alloys [18]. Because of the aforementioned characteristics, boride particles in TiBloy probably settle more slowly than the TiB_2 particles in conventional Al–Ti–B master alloys [18]. This might be the reason for the reduction of electrical conductivity of the samples grain-refined by TiBloy up to 60 minutes.

OPTIFINE is a 3/1 type of Al-Ti-B based master alloy. It is been reported that on a relative scale, the efficiency of OPTIFINE is at least twice, sometimes up to thirty times higher than the standard refiners normally used [19]. This high efficiency is linked to a narrow range of boride particle sizes, which allows simultaneous nucleation on a large number of aluminium crystals [19]. The electrical conductivity of the commercial pure aluminium grain-refined by OPTIFINE shows the same trend as TiBloy and Al-3Ti-0.15C; a decrease that continues up to 60 minutes holding time and then a moderate increase. This may also be related to having narrow range of boride particle sizes that lead to low settling rate of the particles.

4. Conclusions

The present study gives insight on how small addition of different grain refiners (Al-5Ti-1B, Al-3Ti-1B, OPTIFINE, TiBloy, and Al-3Ti-0.15C) can influence the structure, as well as the electrical conductivity of commercial EC grade 1070 aluminium (commercial pure aluminium). The main findings are as follows:

1. The grain refining performance of Al-5Ti-1B, Al-3Ti-1B, OPTIFINE and TiBloy with the addition level of 0.1 Wt. % was weak and the grains after grain refining are more or less columnar.

2. The grain refining efficiency of Al-3Ti-0.15C master alloy was significantly better than Al-5Ti-1B, Al-3Ti-1B, OPTIFINE and TiBloy for grain refining of electrical conductive grade aluminium alloys at equal addition level of 0.1 Wt. %.

3. In the case of Al-3Ti-0.15C master alloy, slight fading occurs after long holding time (90 minutes).

4. The electrical conductivity of the commercial pure aluminium grain-refined by Al-5Ti-1B, Al-3Ti-1B, OPTIFINE, TiBloy, and Al-3Ti-0.15C master alloys, with the maximum addition level of 0.1 Wt.%, did not change remarkably.

5. The study of the macrostructure and the electrical conductivity of all the samples grain-refined by five different grain refiners, Al-5Ti-1B, Al-3Ti-1B, OPTIFINE, TiBloy, and Al-3Ti-0.15C, showed that Al-3Ti-0.15C was the most efficient grain refiner by giving fine grain structure and not reducing significantly the electrical conductivity of the commercially pure aluminium.

5. Acknowledgment

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