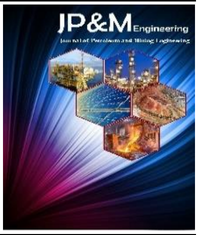
Journal of Petroleum and Mining Engineering 22(2)2020 DOI: 10.21608/jpme.2020.33579.1037

JP&M Engineering

Journal of Petroleum and Mining Engineering



Joint Addition of Zirconium, Titanium and Chromium to Commercial Pure Aluminium

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Article Info

Received 23 Jun. 2020
Revised 11 Jul. 2020
Accepted 23 Jul. 2020

Keywords

joint addition, Zr-Ti-Cr, grain refinement, aluminium.

Abstract

The effect of joint addition of Zr, Ti and Cr on the grain refinement of commercial pure aluminium (99.7% Al) has been investigated by optical microscopy and scanning electron microscopy (SEM) as well as Energy Dispersive X-ray Spectroscopy (EDS). It was found that joint addition of 0.15 wt% Zr and 0.025 wt% Ti to Al can result in a remarkable refinement with an average grain size of 102 µm. It was found the optimum addition level of Ti to be 0.025 wt% in the presence of 0.1 % Zr and any increase in the Ti beyond 0.025wt% results in coarse grain size. Joint additions of 0.15 wt% Zr, 0.025 wt% Ti and 0.15 wt% Cr to Al facilitate better grain refinement and the average grain size was 75 µm. The grain refining performance of joint addition of 0.1 wt.% Zr and different additions of either Ti or Cr is higher than refining with zirconium alone. EDS and SEM analysis of the precipitated phases observed at or near the centers of the refined aluminium with joint addition of Zr and Ti was found to be $Al_3(Zr_{1-x}Ti_x)$. These $Al_3(Zr_{1-x}Ti_x)$ particles act as heterogeneous nucleation sites for α-Al during solidification and resulted in better grain refinement.

Introduction

Aluminium is the most abundant element in the earth's crust. Pure aluminium possesses many advantages to be used extensively in industry, such as good electrical conductivity which superior to copper, better heat conductivity, lower density and higher plasticity [1-3]. Depending on the conductivity of the aluminium alloy, the current carrying capacity of some aluminium alloys is about twice (per unit mass) compared to copper [4].
The wide applications of aluminium and its alloys in industry are limited for the coarse grain size which degrades the mechanical properties [5,6,7]. Grain refinement is a favorite technique to improve simultaneously the plasticity and strength of metallic materials. So that grain refining of aluminum is a key technique in aluminum processing. Now a days, there have been a number of techniques for aluminum grain refining. Grain refining techniques can be classified into four categories as follows: the addition of grain refiner, grain refining by stirring and vibration during solidification, rapid solidification and severe plastic deformation [8].

The widespread usage of grain refinement in the aluminium industry is due to that grain refinement is one of the predominant techniques in controlling the quality of Al castings [5,6-9] . Formation of fine equiaxed grains during solidification is a result of grain refinement. Fine equiaxed grains leads to many benefits such as, uniform and improved mechanical properties throughout the material, imparts high yield strength, good extrudability, reduced ingot cracking and improved resistance to hot tearing, and gives a more uniform distribution of secondary phases and microporosity on a fine scale [10-14]. The most common grain refiners used for aluminium and aluminium based alloys are the transition metals such as titanium (Ti), vanadium (V), zirconium (Zr), tantalum (Ta) etc. These transition metals are used to refine aluminium and its alloys in the form of master alloys of sort Al-B, Al-Ti-B, Al-Ti-C of different Ti/B and Ti/C ratios, Al-Zr and Al-Sc [8, 15-18]. The grain refinement mechanisms are based on heterogeneous nucleation of aluminium grains on inoculant particles. According to the heterogeneous nucleation theory, the effectiveness of grain refining is governed by two factors: number of particles nuclei in a unit of melt volume and effectiveness of the inoculate action of particles nuclei. The latter factor depends on the similarity of crystal lattices of a particle-nucleus and matrix in terms of size and structure. The similarity in lattice types plays a decisive role in grain refining. Increasing the number of nucleating particles results in a fine-grained structure, so that a large number of crystals are formed, which soon impinge on each other and prevent each other from further growth [14,19,20]. Grain refinement of Aluminium is affected by the percentage and type of grain refiners, the holding time [21] and the size of added grain refiner [22].

The aim of this work is to investigate the performance of joint additions of Al-Zr-Ti and Al-Zr-Ti-Cr as grain refiners for commercial pure aluminium (99.7%).

Experimental procedures

Commercial pure aluminium (99.7%) was the starting material for all grain refinement experiments. Al-Zr master alloy was used as the grain refiner. This master alloy was prepared by in-situ reduction of zirconium oxide (ZrO_2) with excess aluminium in the presence of cryolite flux. Al-Cr master alloy was obtained from Aluminium Company of Egypt (EGYPTALUM). A commercially pure Al was melted in a graphite crucible using electrical resistance furnace at 740 °C. After addition of the grain refiners to molten aluminium, the melt was stirred with a graphite rod for 60 s to homogenize the melt. The molten aluminium was kept for 90 s in the furnace then poured into a steel ring of 75-mm diameter, 4 mm wall thickness and 25 mm height with a base of refractory brick. After solidifying and cooling, the specimens were prepared for macrograph by grinding, polishing and etching in solution contains 15mm HF, 15mm HNO_3, 45 mm HCl and 25mm distilled water. For measuring the grain size of the grain refined specimens, specimens were ground and polished then the micrograph was revealed using Keller's reagent to reveal their grain boundaries. At least 40 pictures were taken for each sample using Olympus PME M021 0ptical microscope attached with digital camera, which were used in measuring the grain size with the linear intercept method (ASTM-E112-13).

SEM image was examined by a Scanning Electron Microscope (SEM) using a Joel- JSM-5400 LV -SEM equipped with EDX analyzer at Electron microscopy unit-Assiut university.

Results and Discussions

Joint Addition of 0.1 % Zr and Various Ti Additions

Figure 1 (a) shows the macrostructure of unrefined commercial pure aluminium with average grain size of approximately 1100 µm. Unrefined aluminium exhibits fine columnar structure at the periphery and coarse equiaxed grains at the center of the specimen. Figure1 (b-f) shows the macrostructures of commercial pure aluminium specimens inoculated with joint addition of 0.1 % Zr and various additions of Ti at 740 C^o and holding time of 90 seconds. It is obvious that the macrostructure of the commercial pure aluminium grain refined with joint addition of Ti and Zr shows equiaxed, fine and uniform grains. Wang et al [23] found that Al_3Zr particles formation are highly potent nucleants for Aluminium.

The precipitated phases observed at or near the centers of the refined commercial pure aluminium grains refined with joint addition of Zr and Ti were studied by SEM micrographs at larger magnification (5000x) as shown in Figure 2(a). It is clear that these particles exhibit petal-like morphology. Figure 2(b) shows **EDX** analysis for the phase. The composition of these particles was found to fit to $Al_3(Zr_{1-x}Ti_x)$. These particles are to act as heterogeneous nucleation sites for Al during solidification.

The reason of fine structure is related to formation of $Al_3(Zr_{1-x}, Ti_x)$ particles which act as potent nucleating sites for Al. The average grain size decreases as the addition level of Ti increases from 0.01 to 0.025 wt.% then slightly increases at higher Ti additions. The grain refining performance of joint addition of 0.1 wt.% Zr and different additions of Ti is higher compared with refining with zirconium alone [24,25]. Figure3 (a-e) shows the microstructures of commercial purity aluminium with joint addition of 0.1 wt. % Zr and the different addition levels of Ti (0.01, 0.015, 0.02, 0.025, and 0.03 wt. %).

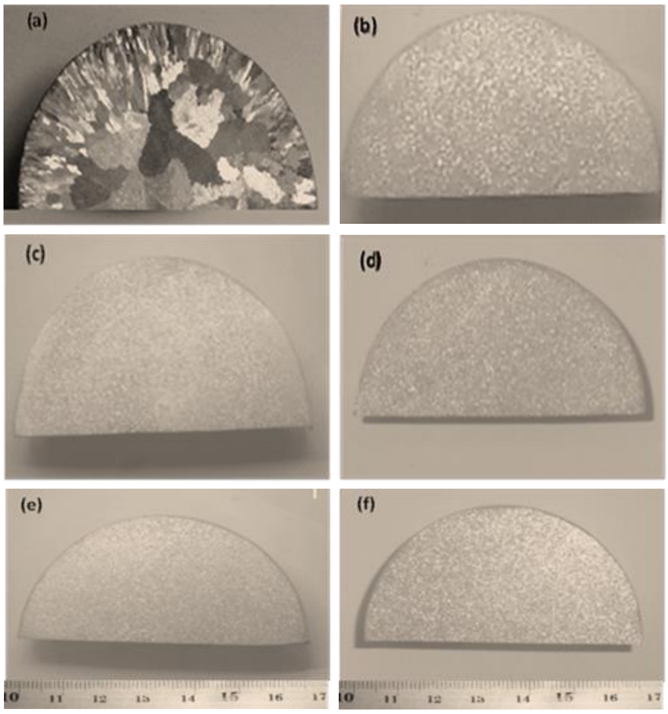

Figure 1 Optical macrographs of commercially pure Al grain refined with 0.1 wt. % Zr and different addition levels of Ti: (a) unrefined; (b) 0.01; (c) 0.015; (d) 0.02; (e) 0.025 (f) 0.03 wt.% Ti.

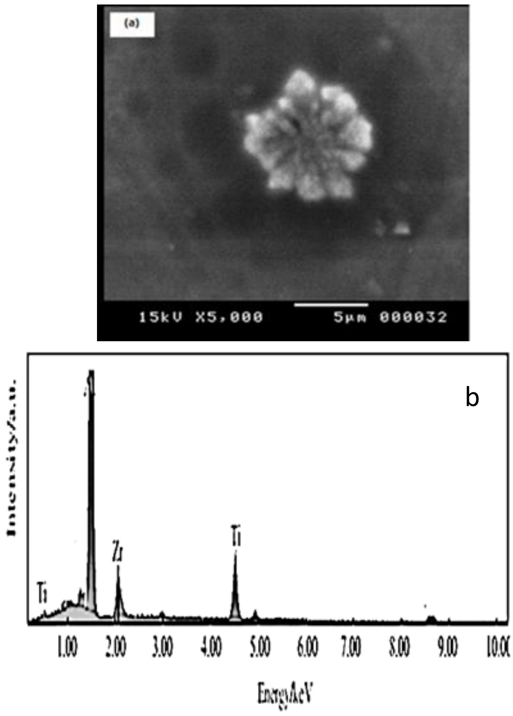

Figure 2 (a) SEM image of $Al_3(Zr_x,Ti_{1-x})$, (b) EDX spectrum at the particle center.

It can be seen from Figure 1 that there is a significant conversion from columnar grain structure of the commercial purity aluminium to equiaxed grain structure. Increasing the addition of Ti simultaneously with 0.1 wt.% Zr, the aluminium microstructure get more refined. Figure 4 displays the average grain size variation of the grain refined aluminium as a function of addition level of Ti, which clearly shows the grain refining performance of joint addition of titanium and zirconium. It can be noted that, the average grain size of the refined aluminium with constant addition of Zr (0.1 %) is decreased to 155, 144, 135, 125 µm, respectively with increasing the percentage of Ti to 0.01, 0.015, 0.02, and 0.025, respectively. The optimum addition level of Ti with 0.1 % Zr is 0.025 % at which the average grain size of aluminium is 125 µm. At higher concentration of Ti the average grain size of aluminium increases. This behavior agrees with the result which obtained by Jaradeh et al. [26], where not all Ti_3Al particles are dissolved. Zhang et al [27] found that excess Ti addition tend to form large particles of Ti_3Al intermetallic.

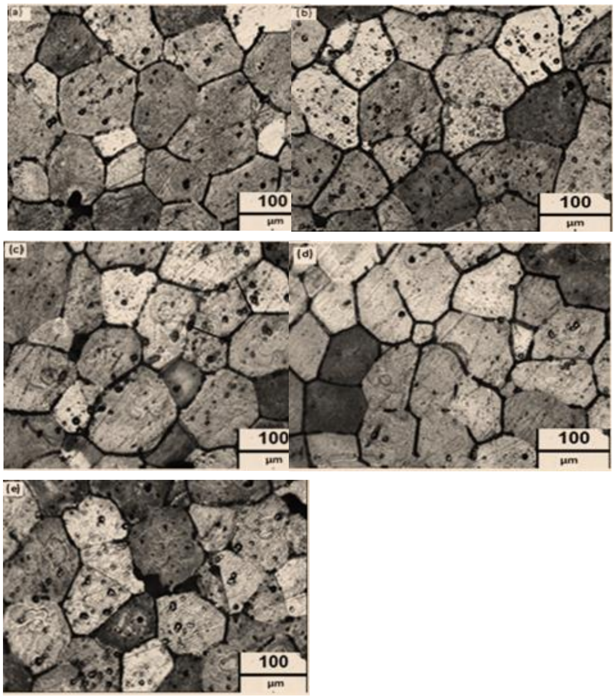

Figure 3 Optical micrographs of grain refined aluminium by addition of 0.1 wt. % Zr and different addition levels of Ti: (a) 0.01; (b) 0.015; (c) 0.02; (d) 0.025; (e) 0.03 wt. % Ti.

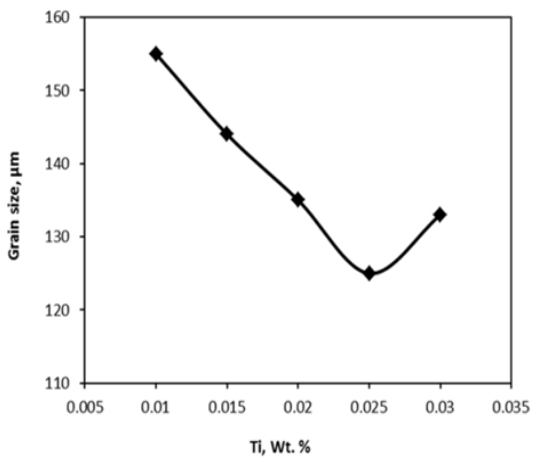

Figure 4 Effect of joint addition of 0.1 wt. % Zr and various addition levels of Ti on grain size.

Joint Addition of 0.15 wt. %Zr and Various Ti Additions

The effect of joint addition of several contents of Ti and 0.15 wt. % Zr on the grain refinement of commercial purity aluminium is illustrated in Figure 5 (a-e), which shows the macrostructure of the refined aluminium. It can be noted that, the joint addition of 0.15 wt. % Zr and 0.01, 0.015, 0.02 and 0.025 wt. % Ti can greatly refine the commercial purity aluminium. The refining effect is markedly pronounced with the Ti addition of 0.01 wt. %, then slowly with increasing Ti content from 0.01 to 0.025 wt. %

Figure 6(a-e) indicates the micrographs of the grain refined aluminium by joint addition of 0.15 wt.% Zr and various addition levels of Ti (0.01, 0.015, 0.02 and 0.025 wt.%). It can be revealed that the refined commercial purity aluminium presents the equiaxed grain structure and the finest grain structure is obtained at joint addition of 0.15 wt.% Zr and 0.025 wt. % Ti as shown in Figure 5(d).

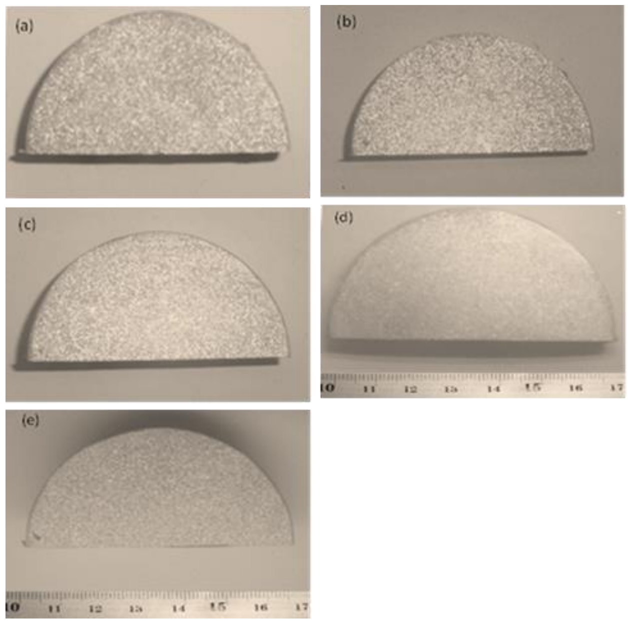

Figure 5 Optical macrographs of commercially pure Al grain refined with 0. 15 wt. % Zr and different addition levels of Ti: (a) 0.01; (b) 0.015; (c) 0.02; (d) 0.025; (e) 0.03 wt.% Ti.

It is important matter to determine the optimum addition level of the grain refiners, at which, a more uniform distribution of intermetallic phases and finer equiaxed grain structure in comparison with unrefined aluminium can be achieved. Figure 7 shows the effect of various concentrations of Ti with 0.15 wt.% Zr added to aluminium melt prior to solidification. By joint addition of 0.15 % Zr and 0.01Ti %, the average grain size of aluminium reduces from 1100 µm to 150 µm. Joint addition of 0.15 % Zr and 0.015Ti % decreases the average grain size of aluminium to 141 µm. Further addition of 0.15 % Zr and 0.02%Ti reduces the average grain size of aluminium to 126 µm. The optimum amounts of Zr and Ti added to aluminium melt are 0.15 % Zr and 0.025 % Ti. At this optimum addition level, the average grain size of aluminium drastically reduced from 1100 to 102 µm. Increasing the concentration of Ti to 0.025 % with 0.15 % Zr increases the average grain size of aluminium to 122 µm. The variation in grain size represents more produced effective nucleation sites in the grain refined aluminium specimens. The $Al_3(Zr_{1-x},Ti_x)$ particles act as potent nucleating sites for α-Al and resulted in finer grains which are caused by the grain refining effect of joint addition of Zr and Ti.

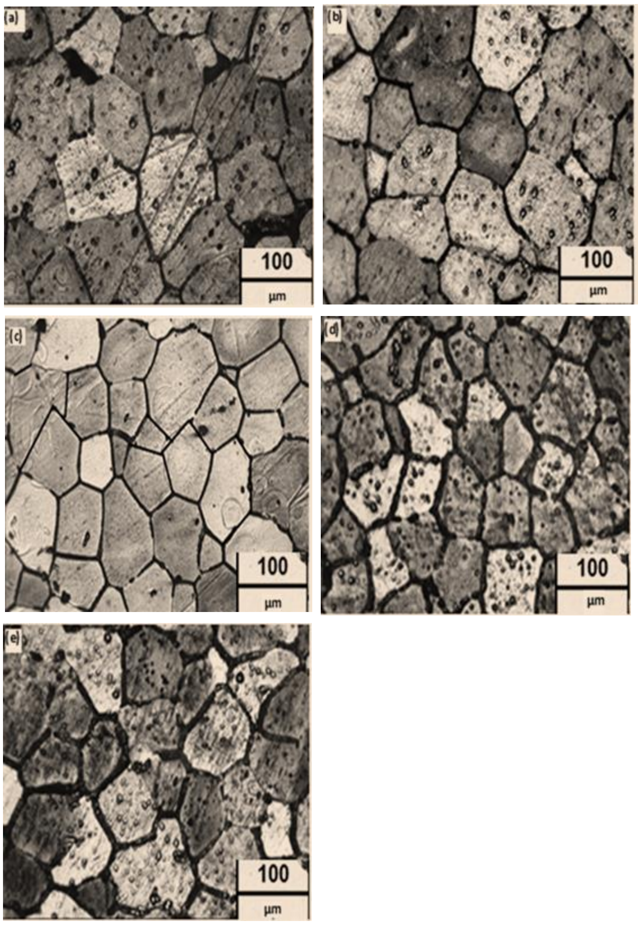

Figure 6 Optical micrographs of grain refined aluminium by addition of 0.15 wt. % Zr and different additions of Ti: (a) 0.01; (b) 0.015; (c) 0.02; (d) 0.025; (e) 0.03 wt. % Ti.

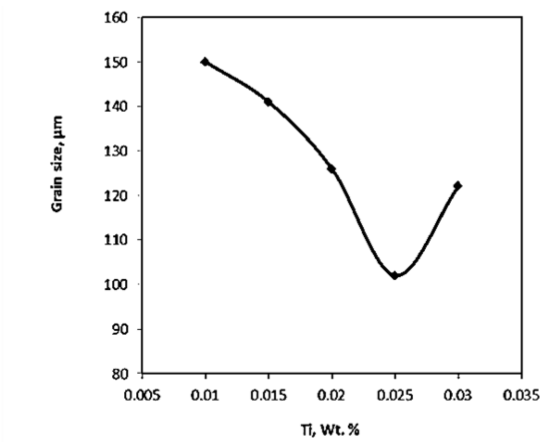

Figure 7 Effect of joint addition of 0.15 wt. % Zr and various addition levels of Ti on grain size

Joint Addition of 0.2 % Zr and Various Ti Additions

Figure 8(a-d) shows the macrographs of the refined aluminium with 0.2 wt. % Zr and different Ti additions (0.01, 0.015, 0.02 and 0.025 wt. %, respectively). It can be observed that, complete conversion of coarse columnar grain structure to fine equiaxed grains occurs. As the amount of Ti is increased from 0.01 to 0.02 wt. %, the average grain size became finer then increased slightly by further addition of Ti. It is clear that the macrostructure of the refined aluminium specimens by joint addition of 0.2 wt. % Zr and different Ti additions are coarsened slightly compared to that refined by 0.15 wt. % Zr and different Ti additions.

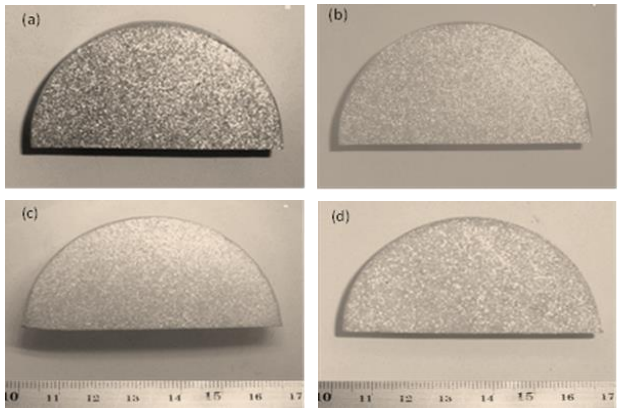

Figure 8 Optical macrographs of refined Aluminium with 0.2 wt. % Zr and different addition levels of Ti: (a) 0.01; (b) 0.015; (c) 0.02; (d) 0.025 wt.% Ti

The representative microstructures of grain refined aluminium by joint addition of 0.2 wt. % Zr and different amount of Ti are given in Figure 9 (a-d).

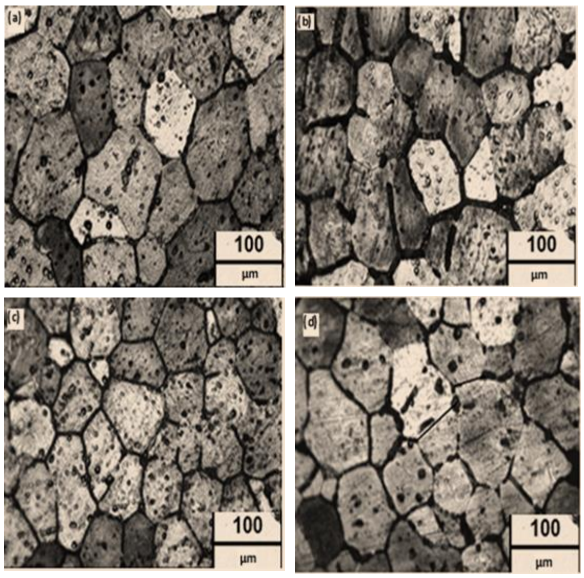

Figure 9 Optical micrographs of grain refined aluminium by addition of 0.2 wt. % Zr and different addition levels of Ti: (a) 0.01; (b) 0.015; (c) 0.02; (d) 0.025 wt. % Ti.

From these micrographs, it is clear that the grain morphologies of all the specimens exhibit an equiaxed microstructure and no dendrites were observed. The average grain size of refined aluminium with joint addition of 0.2 % Zr and Ti (0.01, 0.015, 0.02, and 0.025 %, respectively) is plotted in Figure 10. It can be noted that, with joint addition of 0.2 wt.% Zr and 0.01% Ti, the average grain size of aluminium reduced significantly to 140 µm. Increasing the addition to 0.2 wt. % Zr and 0.015 % Ti, the grains of aluminium can get a better refining effect and the average grain size reduced to 126 µm. By addition of 0.2 wt. % Zr and 0.02 % Ti the grain size reduced to 114 µm. Further addition of Ti and 0.2 wt. % Zr leads to a slight increase in the average grain size of aluminium.

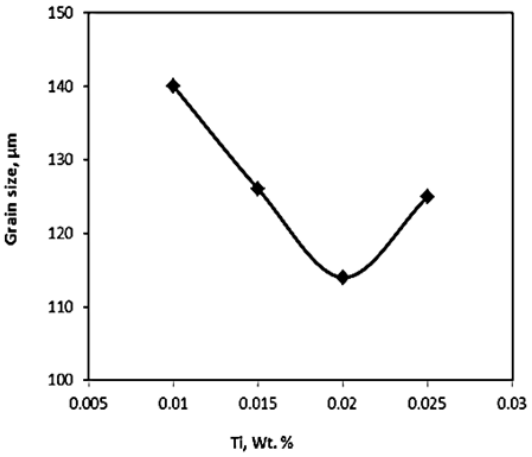

Figure 10 Effect of joint addition of 0.2 wt. % Zr and various addition levels of Ti on grain size.

Joint Addition of 0.15 wt.%Zr, 0.025 wt.%Ti and Various Cr Additions

Figure 11(a-d) presents the macrostructure of commercial Al specimens, which were refined by joint addition of 0.15 wt. % Zr, 0.025 wt. % Ti and different addition levels of Cr. It is shown that all refined specimens exhibit fine equiaxed grains. It was found that Cr dissolved in liquid Al in small quantities and alters the surface tension of Al to improve the wettability of the grain refining constituents of the grain refiners [28].

Figure 12 (a-d) shows the micrographs of refined aluminium with joint addition of 0.15 wt. % Zr, 0.025 wt. % Ti and different addition levels of Cr. It can be observed from Figure 12 (a-c) that, as the addition level of Cr increases up to 0.15 wt. %, the microstructure of Al specimens get more refined. Further addition of Cr slightly increases the grain size of the refined Al. This is due to that, increasing the addition level of Cr accelerates the dissolution rate of active nucleating sites [14,28].

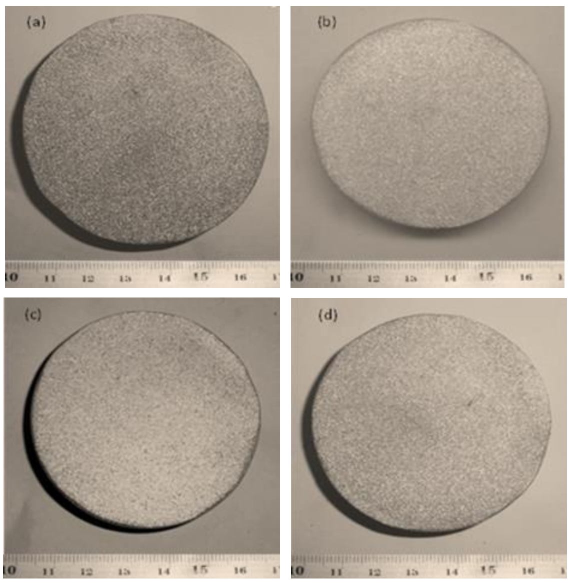

Figure 11 Optical macrographs of refined aluminium with joint addition of 0.15 wt. % Zr, 0.025 wt. % Ti and different addition levels of Cr: (a) 0.05; (b) 0.1; (c) 0.15; (d) 0.2 wt. %Cr.

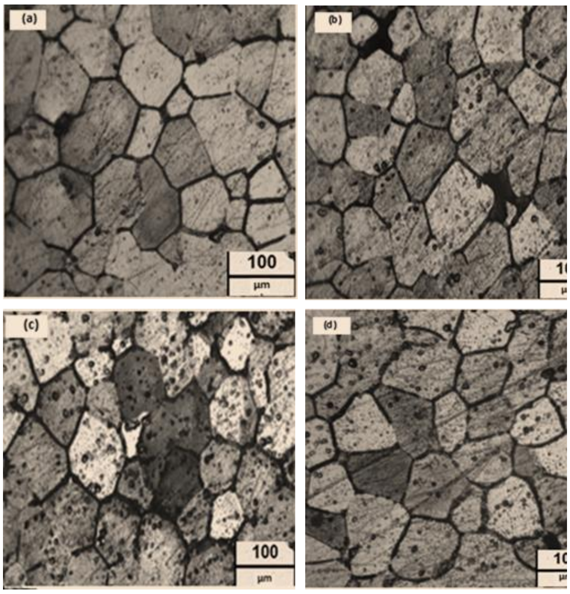

Figure 12 Optical micrographs s of commercially pure Al grain refined with joint addition of 0. 15 wt. % Zr, 0.025 wt. % Ti and different addition levels of Cr: (a) 0.05; (b) 0.1; (c) 0.15; (d) 0.2.

Figure 13 shows the addition levels of Cr versus the average grain size of the refined Al. It can be seen that, joint addition of 0. 15 Zr, 0.025 Ti and 0.05 wt. % Cr drastically reduces the average grain size of Al to 93 µm. Joint addition of 0.15 Zr, 0.025 Ti and 0.1 wt. % Cr facilitates better grain refinement, where the average grain size of Al is 82 µm. A remarkable refinement with an average grain size about 75 µm can be obtained with joint addition of 0.15 Zr, 0.025 Ti and 0.15 wt. % Cr. Joint addition of 0.15 Zr, 0.025 Ti and 0.2 wt. % Cr results in increasing the average grain size to 80 µm.

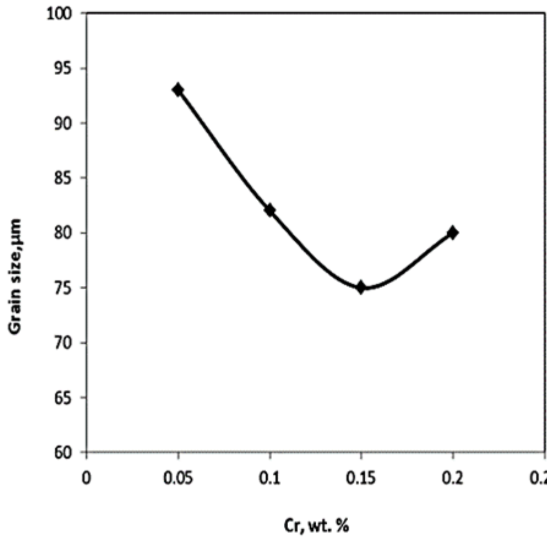

Figure 13 Effect of joint addition of 0. 15 wt. % Zr, 0.025 wt. % Ti and different addition levels of Cr on grain size.

Conclusions

The effect of joint additions of zirconium, titanium and chromium on the grain refinement of commercial pure aluminium (99.7%) is investigated by microstructure, macrostructure observation and grain size measuring. The following conclusions can be drowned:

1- $Al_3(Zr_{1-x}Ti_x)$ phase which precipitated at or near the centers of the refined grains in case of joint addition of Zr and Ti was observed by SEM and confirmed by EDX.
2- $Al_3(Zr_{1-x}Ti_x)$ phase has a petal like morphology and acts as a substrate for heterogeneous nucleation sites for the refined aluminium.
3- Joint addition of 0.15 wt. % Zr and 0.025 wt. % Ti effectively refined the microstructure of aluminium with average grain size of 102 µm. This is due to Ti substitutes for zirconium in Al_3Zr forming $Al_3(Zr_{1-x}Ti_x)$, which reduces the lattice parameters mismatch between aluminium and Al_3Zr improving the grain refining efficiency.
4- Joint addition of 0.15 wt. % Zr, 0.02 wt. % Ti and 0.15 wt. % Cr drastically reduces the average grain size of commercial pure aluminium from 1100 to 75 µm. This is attributed to that, Cr act as a grain refiner for aluminium and as a substrate for nucleating $Al_3(Zr_{1-x}Ti_x)$ particles.

Funding sources

This research received no external funding.

Conflicts of interest

There are no conflicts to declare.

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