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Grain Refining of Aluminium and 6063 Alloys Using Al-V Alloy Containing Al_3V Intermetallic Compound

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The grain refinement of aluminium alloys is one of the important processes in various industrial fields. This work aims to use aluminium-vanadium (Al-V) master alloy as a new grain refiner for grain refinement of aluminium alloys. The new refiner was prepared by reduction of vanadium pentoxide V_2O_5 using molten aluminium, then it was examined using SEM, EDS, XRD, and optical microscopy. The findings of these tests indicated that the developed master alloy contains blocky-shaped particles evenly distributed within the aluminium matrix Al_3V in the form of an intermetallic compound. Different levels of vanadium content were examined (3.5, 5.6, and 8.3) as grain refiners for commercial pure Al and 6063 alloys. It was found that the grain size was decreased from 2000 to 320 µm for commercial pure aluminium and decreased from 1500 to 300 µm for 6063 alloys. The best results were obtained by using vanadium content of 0.03 wt.% in the refined alloy and a holding time of 3 min. Based on the growth restriction factor (GRF), the new grain refiner Al-V master alloy performed well at vanadium contents more than 0.02 wt.% in the refined alloy.

Keywords: Al-V alloys, Al_3V intermetallic compound, grain refining, growth restriction factor.

1. INTRODUCTION

Aluminium alloys ingots represent the first step in the production of final products such as sheets, extrusion products, etc. To obtain good aluminium alloy products, a grain refiner must be added to the molten aluminium just before casting to produce semi-finished products such as cylinders or plates. These refiners have beneficial effects for aluminium alloys, especially in mechanical properties, increasing casting speed, and reducing casting defects [1, 2]. Several factors affect the macrostructure of the as-cast aluminium alloys through affecting the nucleation process during solidification of some solute elements on grain refinement of aluminium alloys [3, 4]. The performance of the grain refiners, as well as growth restriction factor (GRF) in the molten metal, can be influenced by the presence of alloying elements and impurities. Also, the influence of solutes can be measured on grain size evolution using three parameters, such as the GRF, Q; undercooling, P; and the freezing parameters [5 – 7]. The great importance for controlling the grain size of the cast aluminium alloy is the GRS, Q which is considered the main factor affecting the grain refining process [7]. It is depending on the liquidus line gradient, the concentration of solute in a binary alloy, and the solute partition coefficient [5, 8]. The transition elements have a high GRS in aluminium alloys such as titanium, tantalum and vanadium [9 – 11]. The grain refiners such as Al-Ti-C, Al-Ti, and Al-Ti–B are widely used for grain refining of aluminium and its alloys. Adding a small percentage of these grain refiners to molten aluminium and its alloys has a fantastic effect on the structure and grain size but after a period, this effect decreases, which is known as ingot fading or poisoning of grain refiner, especially when uses the Al-Ti-B alloy [12 – 14]. There are various interpretations of the phenomenon of poisoning and its cause. One of these theories of poisoning is that the poisoning occurs because of the molten aluminium coats by Al_3Ti and prevents its spread. The fading time in aluminium alloys depends on many factors, involving casting temperature, and constituting elements. The fading problem was solved by using other grain refiners such as Al-Ti-C [15 – 17]. The master alloy produced from aluminium with other transition elements such as Ta, Ti, V is a vital goal for the industry of aluminium [9, 18 – 21]. However, the Al-V master alloy production and use as a refiner of Al and its alloys are poorly published in the open literature. Generally, tri-aluminide intermetallic of transition-metal could provide a kind of reinforcement metal matrix composite from light metal [22-24]. In this work, an attempt will be made to use a new Al-V alloy for grain refinement of aluminium and 6063 alloy, the new refiner was prepared in a previous work by reduction of V_2O_5 by molten aluminium as explained elsewhere [25]. The other objective also aims at studying the parameters affecting the grain refiner efficiency on Al and its alloys to obtain the optimum grain refinement conditions such as addition rate and holding time.

2. EXPERIMENTAL PROCEDURES

2.1. Materials

The used materials in this work were: powdered Al (99.6 %) having a particle size of 59 µm, and powdered V_2O_5 (99.5 %) with particle size 178 µm and commercial pure Al 99.7 purity.

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2.2. Preparation of the Al-V master alloy

The powdered V_2O_5 was mixed with powdered Aluminium at different ratios. The mixtures were added to molten aluminium at a varying temperature from 700 – 850 °C in a vertical muffle furnace with mechanically stirred and pouring into cast-iron molds as explained elsewhere [25]. The produced prepared Al-V alloy specimens were characterized using XRD, SEM, X-ray mapping and analyzed the concentration of the constituent elements using EDS.

2.3. Evaluation method

To evaluate the prepared Al-V master alloysV1, V2, and V3; a calculated weight of commercially pure aluminium or 6063 alloy is melted in a crucible made of silicon carbide using a vertical muffle furnace at 720 °C until melting. The crucible containing molten Al withdrawn out the furnace and the formed slag is well removed. Then the calculated weight of the grain refiner alloy to be tested is added to the melt and stirred by a graphite rod for 10 seconds. The crucible is returned to the furnace by keeping it for the required holding time. Then, the crucible is taken out of the furnace and poured into KBI ring mould (3 in diameter and 1 in height) located on the top of a refractory base (fused silica block) [26]. When the solidification of the specimen has been completed, the solidified specimen is water quenched. Then, the bottom surface of the specimen is etched using an etchant consisting of a mixture of 43 % HNO_3, 43 % HCl, and 14 % HF until the grain boundaries of the specimen have appeared. The grain size of the etched surface is measured via the linear intercept method, which is based on the counting of grains intercepted by four cross rays under 10 X magnification.

A run of experiments was performed to study the effectiveness of the prepared Al-V master alloys as grain refiners on the grain refinement of commercially pure Al and 6063 alloys. Table 1 shows elementary analyses of different types of Al-V grain refiners, commercially pure aluminium 99.7 wt.%, and 6063 alloys used to evaluate the prepared master alloys grain refining.

Table 1. Elementary analyses of different types of Al-V grain refiners, commercially pure Al 99.7 wt.% and 6063 alloys

Composition / Alloy	Mg, wt.%	Si, wt.%	V, wt.%	Al, wt.%
V1, Grain refiner	–	–	3.5	Rem
V2, Grain refiner	–	–	5.8	Rem
V3, Grain refiner	–	–	8.3	Rem
Al 99.7 %	0.004	0.01	Other elements are nil	Rem
6063 alloy	0.57	0.36	Other elements are nil	Rem

3. RESULTS AND DISCUSSION

3.1. Characterization of the Al-V alloys

The produced Al-V alloy containing 5.8 % V was analyzed using X-ray diffraction, and the specimen was scanned from 20 to 70 degrees. The phase identification from XRD pattern as shown in Fig. 1 indicated that there are two phases, only pure Al and Al_3V appeared. Refers to the Al-V phase diagram indicated in Fig. 2, there are three metastable compounds, $Al_{21}V_2$, $Al_{45}V_7$, and $Al_{23}V_7$ existed below temperature 734 °C at composition about 16.2, 21.2 and 24.4 % respectively, and disappeared at temperature more than 734 °C.

While the intermetallic compound Al_3V existed at about 39 wt.% V and stable until 1249 °C as shown in Fig. 2 [8, 27]. The crystal structure of Al_3V obtained from X-ray diffraction data using PDF2, the program is body-centered tetragonal, and its lattice parameters are a, b is 3.78, and c is 8.322 λ.

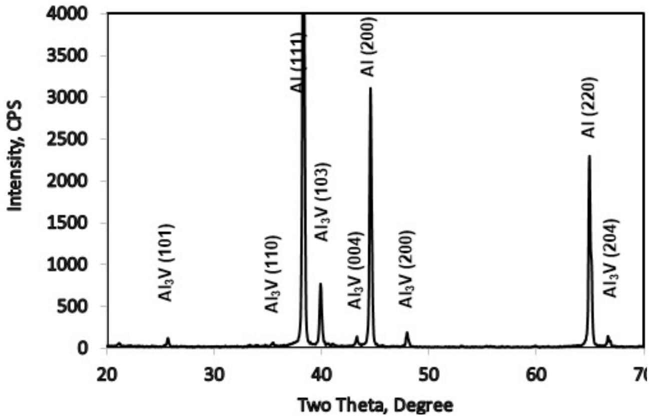


Fig. 1. XRD pattern for the prepared Al-V master alloy contains 5.8 wt.% V

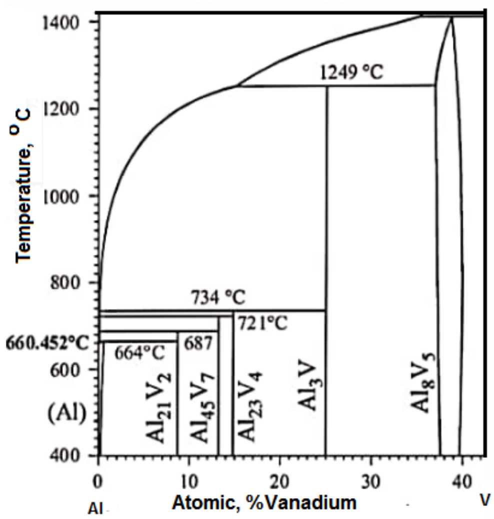
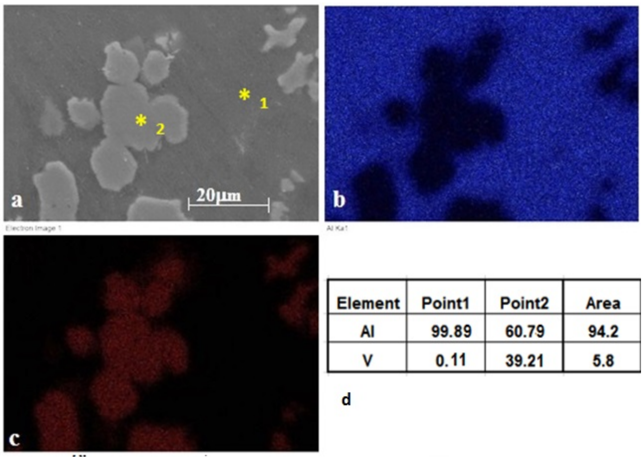


Fig. 2. Al-V binary phase diagram [25]

Fig. 3 shows the SEM micrographs analyzed by X-ray mapping of the produced Al-V grain refiner containing 5.8 % V. This figure indicates that the light grey phase is a homogeneous blocky shape distributed within a dark grey matrix Fig. 3 a. The analysis of the dark grey phase was analyzed at point 1 using energy dispersive x-ray (EDX), and it was 99.89 wt.% Al and 0.11 wt.% V. While the analysis of the dark grey phase (matrix) at point 2 was 60.79 aluminium and 39.21 wt.% vanadium. The analysis of the light grey phase at point 2 is much closer to the composition of the present Al_3V intermetallic compound vertical line at about 39 % as shown in the Al_3V phase equilibrium diagram Fig. 2 [8, 27], this finding was confirmed with the result of

XRD analysis. From the previous pieces of evidence, it is proved that the light grey phase and the dark grey phase are Al_3V intermetallic compound and aluminium matrix, respectively.

The area analysis of all matrices is 94.2 aluminium and 5.8 % vanadium. The dispersal of vanadium and Al atoms in the Al-V as indicated in Fig. 3 b illustrates only Al particles or atom (blue colour) and the place like a shadow is vanadium particles (black). Fig 3 c shows the vanadium particles (red colour) and the place of aluminium particles was the black colour. It could be concluded that the red block consists of a mix of aluminium particles and vanadium particles form Al_3V within the aluminium matrix blue colour, and the results confirmed with the results mentioned before.



Element	Point1	Point2	Area
Al	99.89	60.79	94.2
V	0.11	39.21	5.8

Fig. 3 SEM micrograph and X-ray mapping for the produced alloy contains 5.8 wt.% V: a – SEM micrographs; b – dispersal of Al atoms; c – dispersal of V atoms; d – points 1, 2 and area analysis

The image of backscattered electron for the prepared alloy contains 8.3 wt.% V as shown in Fig. 4.

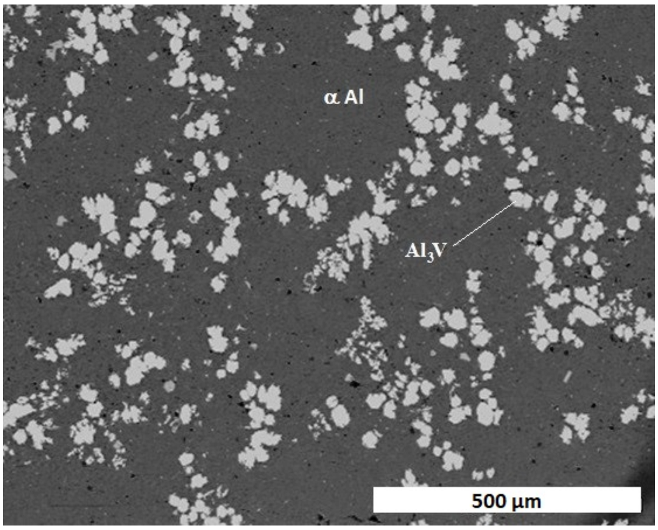


Fig. 4. Backscattered electron image of the prepared Al-V master alloy contains 8.3 wt%V

The figure indicates the distribution of irregular white particles like cotton precipitate around the grain boundary of aluminium particles(black). The Al_3V white particles are almost the same shape of Al_3V intermetallic compound surrounded the grain boundary of the black matrix (aluminium). This is confirmed with the published elsewhere [25] and declared before in both XRD and SEM also, from Fig. 4. The average grain size of the Al_3V intermetallic compound measured is about 10 – 30 µm.

However, Fig. 5 shows an optical micrograph indicating the distribution of Al_3V light grey particles around the grain boundaries of a relatively dark grey particle (α-Al). The same result was obtained from Fig. 4, and this result bears more absolute confirmation that the particles are intermetallic compound Al_3V and its particle size is about 15 – 40 µm.

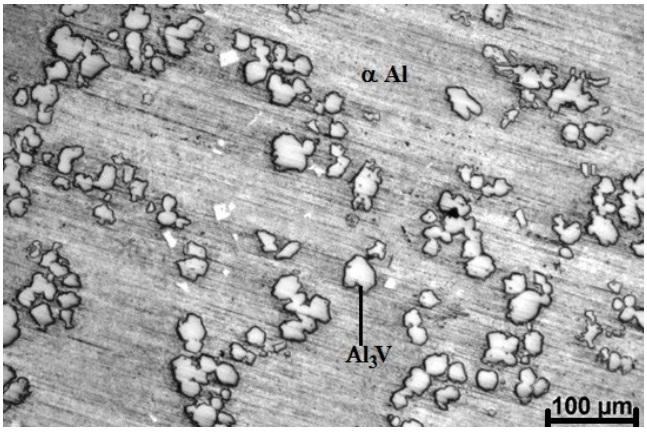


Fig. 5. The optical micrograph shows the dispersal of Al, and V in the prepared Al-V alloy contains 5.8 wt%V

3.2. Evaluation of the Al-V alloy as a grain refiner

The most vital parameters influencing the grain refining efficiency of Al and its alloys are addition rate and holding time. Three-groups of experiments were performed to study the effect of holding time and addition rate on the efficiency of grain refinement of the produced Al-V alloys at different grain refiners V1, V2 and V3 on the aluminium and 6063 alloys.

3.2.1. Effect of addition rate

The addition rate of the prepared Al-V grain refiners was investigated on the efficiency of grain refinement of commercially pure aluminium (99.7 wt.% Al) at addition rates of vanadium are: (0.01, 0.015, 0.02, 0.03, and 0.04) wt.% at constant temperature 720 °C and holding time 3 minutes, at different types of grain refiners (V1, V2 and V3).

Fig. 6 indicated the relationship between the vanadium contents and the grain size of pure aluminium refined with different types of Al-V alloy grain refiners. From this figure, the grain size of pure Al is sharply decreased as the V content in pure Al increases to 0.02 V wt.%. This is maybe due to the increase randomness of nucleation and consequently theincrease of the vanadium content in the pure Al, the nucleating rate increased according to the theory of peritectic reaction, this confirmed by the published elsewhere [7].

Then the grain size slightly decreases in the range from 0.02 to 0.03 wt.% V thereafter, some slight increases in grain size are observed in the range from 0.03 to 0.04 wt.%

V. The slight increase in grain size might be due to the agglomeration or precipitation that leads to poisoning of the refiner as occurs in the case of titanium boride as it has been published elsewhere [16]. The three types of grain refiners V1, V2 and V3 took the same trends, but the Al-V grain refiner master alloy V2 took the lowest grain sizes compared to the other types V1 and V3, and the largest one of the grain sizes belonged to the V3 grain refiner. The improvement of Al-V alloy V2 may be due to the concentration of the vanadium element inside it. However, the increasing in vanadium content inside the alloy (V3) may aggregate the Al_3V particles, and the decrease of vanadium content inside the alloy (V1) does not make it effective.

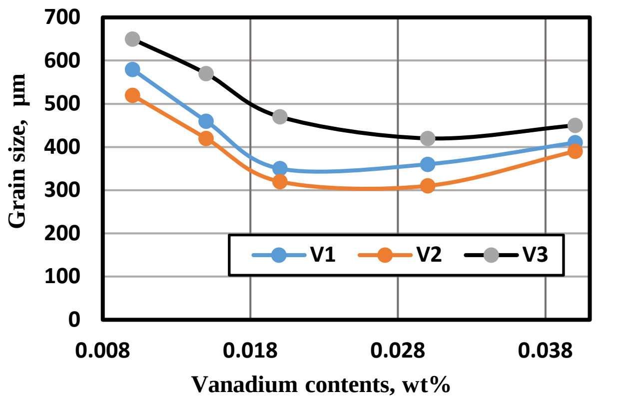


Fig. 6. The relation between vanadium contents, wt.% versus grain size of the pure aluminium refined from different types of Al-V master alloy grain refiners

Fig. 7 indicates the macrograph images of refined pure Al using Al-V master alloy V2 at different vanadium contents 0.01, 0.015, 0.02, 0.03, and 0.04 wt.%. From this figure, it can be noticed that the grain size reduced as the vanadium contents of the refined Al increase as shown in Fig. 7 a – d. This finding may be due to the increase in the rate of nucleating as the vanadium content increased according to the peritectic theory [12]. While Fig. 7 e indicates the grain size increases again as the vanadium content increases to 0.04 wt.% V.

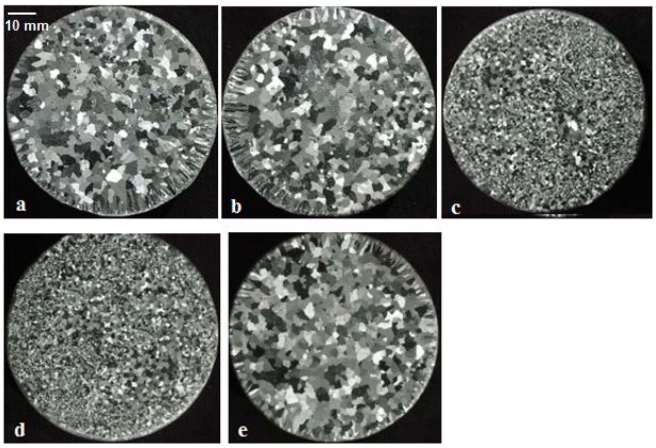


Fig. 7. The macrographs for the refined Al using Al-V master alloy V2 at different vanadium rate: a – 0.01 % V; b – 0.015 % V; c – 0.02 % V; d – 0.03 % V; e – 0.04 % V

The increasing of grain size as the vanadium contents increases to 0.04 wt.% V is due to the agglomeration or segregation of the Al_3V nucleus leading to fading of the Al-V master alloy grain refiner as mentioned in elsewhere published [16].

3.2.2. Effect of holding time

The influence of the holding time on the grain refining efficiency of the Al-V alloy in pure aluminium (99.7 wt.% Al) was studied from 1 min to 20 minutes at a temperature of 720 °C and vanadium contents 0.02 wt.% using different kinds of Al-V grain refiners master alloy (V1, V2 and V3) as shown in Fig. 8. From Fig. 8, it could be seen that, once the refiner is added, the grain size is decreased sharply as the holding time increased up to 3 minutes subsequently, the grain sizes are slightly increased. The behavior of those curves of the three refiners is similar. This means that the Al-V grain refiner wears out or fad at high waiting time. It could say that the holding time plays an important role in the dissolution of the Al_3V nuclei, which diffuse at the first time and then precipitate and agglomerate over time as published elsewhere [16].

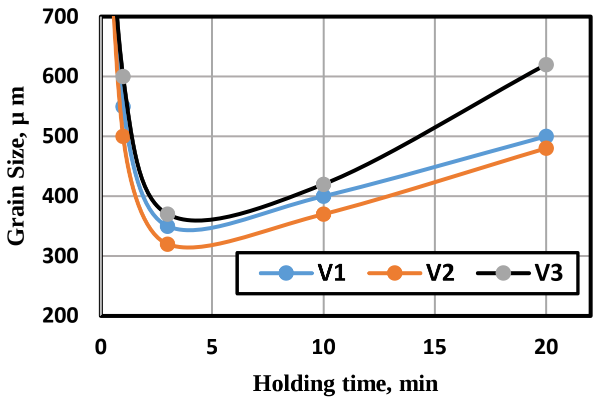


Fig. 8. The relation between the grain size of the refined commercially pure aluminium (99.7 wt.% Al) and holding time using different vanadium (V1, V2, and V3)

Fig. 9 shows the influence of holding time on the grain size of the refined 99.7 wt.% Al via V2.

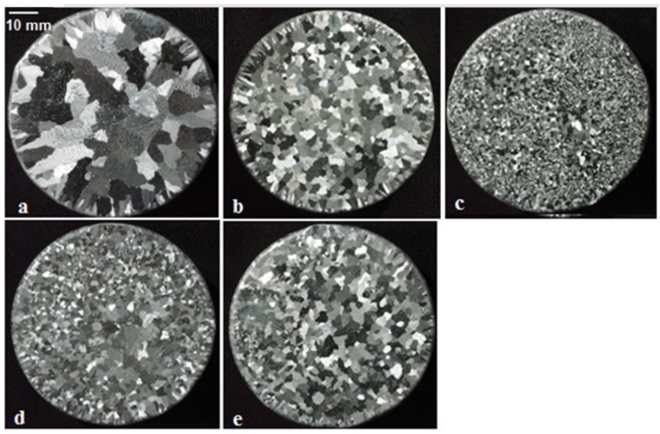


Fig. 9. Macrographs show the changing of the grain sizes versus holding time for the refined Al using V2 refiner: a – without refiner; b – after 1 min; c – after 3 min; d – after 10 min; e – after 20 min

From this figure, it could be detected that the sharp decreases in the refined Al grain size at varying holding times Fig. 9 a – d. Then the grain sizes are slightly increased after 3 min as result of alloy poisoning as mentioned in elsewhere[15, 16]. Fig. 9 a shows a macrostructure photo for pure aluminium without additives, there are large grains of, up to 2000 μm, but after adding 0.02 % of the produced Al-V grain refiner V2, for holding time 1 min., the size of the grains decreased sharply, reaching to 500 μm as shown in Fig. 9 b. Increasing the holding time to 3 min., the grain sizes are decreased to 320 μm, as illustrated in Fig. 9 c, and with more holding time to 10 and 20 min, it begins to increase again, reaching 370 and 480 μm respectively, as shown in Fig. 9 d and e. This increase is due to the fading or poisoning of the grain refiner as a result of many factors such as nuclei segregation or agglomeration of the A3V intermetallic compound, which leads to an increase in the nucleus size and reduce the nuclei number [16].

3.2.3. Effect of the types of refining on both pure Al and 6063 alloy

The effect of the different types of Al-V master alloy grain refiners (V1, V2 and V3) on the efficiency of the grain refinements of pure Al and 6063 Al alloy was studied at a temperature of 720 °C, V content of 0.02 wt.% and holding time of 3 minutes as shown in Fig. 10.

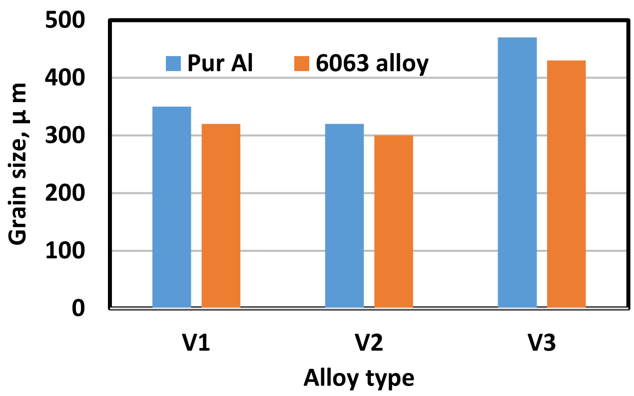


Fig. 10. Comparison between the grain refining efficiency for the pure aluminium and 6063 alloys using the different types of Al-V grain refiners master alloy

From the figure, the second type of Al-V grain refiner master alloy V2 was found the best composition for refining efficiency, which contains 5.8 wt.% V, followed by the type V1 contains 3.5 wt.% V, and then V3 contains 8.3 wt.% V. By comparing the efficiency of grain refinement of pure aluminium against the grain refining efficiency of 6063 alloy, it was found that the efficiency of grain refining for 6063 is improved than that of pure aluminium due to the existence of other alloy elements (solute) that increase the GRF and decrease the size of the particles, as mentioned elsewhere [9, 17].

It is obvious from Fig. 11 that the grain refiner master alloy V2 is the best refiner, where the size of the particles for commercially pure Al decreases from 2000 to 320 μm, and decreases from 1500 to 300 μm for 6063 alloy. While the grain size increases with using the other two types of grain refiners V1 and V3.

The role of alloying elements on estimating the grain size of aluminium is determined by three factors during cooling; i.e., GRF (*Q*), undercooling (*P*), and freezing range.

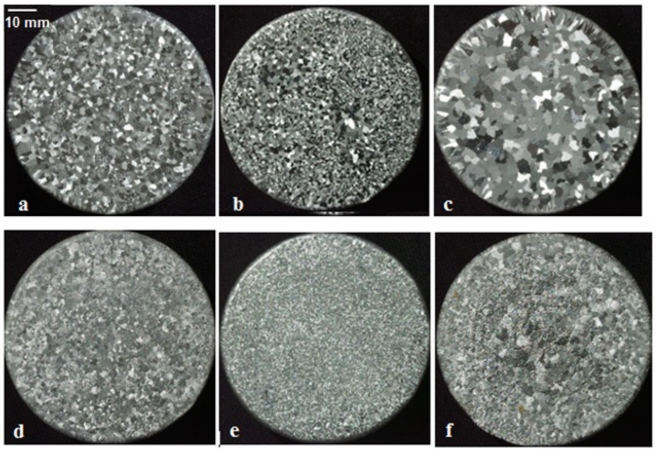


Fig. 11. Macrographs for the refined pure Al and 6063 alloy using different Al-V grain refiners (V1, V2, and V3): a – pure Al refined by V1; b – pure Al refined by V2; c – pure Al refined by V3; d – 6063 alloy refined by V1; e – 6063 alloy refined by V2; f – 6063 alloy refined by V3

These parameters are correlated to the grain refinement degree. GRF *Q* is calculated by [7, 8]:

$$Q = m\, C_o\, (k-1), \tag{1}$$

where *m* is the gradient of liquidus curve; C_o is the concentration of the solute; *k* is the partition coefficient at equilibrium.

Table 2 shows the GRF, and undercooling parameters, *P* required data for binary aluminium alloy calculation. From Table 2, the GRF of vanadium is 30 multiply by the amount of solute of vanadium in aluminium while the GRF for titanium is 220 multiplied by the amount of solute of titanium in aluminium.

Table 2. The growth restriction factor for frequently faced binary systems [7, 8]

Element	k_i	m_i	Max conc., wt.%	$m(k-1)$	$m(k-1)/k$
Ti	7-8	33.3	0.15	~220	~30
Ta	2.5	70.0	0.10	105	42
V	4.0	10.0	~0.1	30.0	7.5
Hf	2.4	8.0	~0.5	11.2	4.7
Mo	2.5	5.0	~0.1	7.5	3.0
Zr	2.5	4.5	0.11	6.8	2.7
Nb	1.5	13.3	~0.15	6.6	4.4

The high of *Q* value for titanium is believed to be why titanium when added in small amount,s decreases dramatically the grain size of Al [7]. According to the magnitude of *Q*, it can be calculated theoretically, the effect of titanium is 6 times the effect of vanadium on grain refining of aluminium. It can say that the addition 50 ppm of titanium to aluminium is equivalent to adding 300 ppm of vanadium to the aluminium to give the same effect on the grain size of pure aluminium. By the same way, according to the undercooling factor (*P*), the effect of titanium is 4 times the effect of vanadium on grain refining of aluminium.

Practically from Fig. 6, the grain size corresponding to addition of 300 ppm vanadium to the refined aluminium using a new Al-V master alloy grain refiner is about

320 μm, this result is closed with the published elsewhere when added about 50 ppm titanium to refind aluminium using Al-Ti or Al-Ti-B master alloys [12, 26, 28,]. It can be concluded that the effect of the new Al-V master alloy grain refiner on the grain refinement of aluminium and its alloys gives the same effect of about one-sixth the same amount of Al-Ti or Al-Ti-B alloys that used for grain refinement of aluminium and its alloys; at the same conditions of temperature and holding time.

4. CONCLUSIONS

A new Al-V master alloy grain refiner was prepared via reduction of V_2O_5 using molten aluminium. The new grain refiner was used for grain refinning for aluminium and 6063 aluminium alloy. The main results were summarized as follows:

1. The prepared Al-V master alloys containing 3.5, 5.6, and 8.3 wt%V were examined as grain refiners for commercial pure Al and 6063 alloy. The grain size of commercial pure aluminium were decreased up to 320 μm and decreased up to 300 μm for 6063 alloy.
2. The new Al-V grain refiner containing 5.8 wt.% V is the best one, and the best conditions were found; vanadium contents in the refined alloy 0.03 wt.% and holding time 3 min.
3. The effect of Al-V master alloy is roughly equivalent to the one-sixth effect of Al- Ti and Al-Ti-B master alloys on grain refinement of aluminium and its alloys at the same conditions of temperature and holding time.
4. According GRF, the new grain refiner Al-V master alloy performed well at vanadium contents in the refined alloy more than 0.02 wt.%.

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