The Effect of In-Situ Method Parameters in Size and Distribution of Particles for Aluminum Matrix Composites

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ABSTRACT

In this work, an in-situ (oxidation) method was used extensively to reinforce aluminum matrix composites with alumina particles. Multi parameters such as alumina particle size and its distribution were studied in details. Side hole diameter, stirring velocity and stirring method were studied also. XRD and SEM tests were used to characterize alumina particles. Results revealed it was obtained on aluminum matrix composites reinforced with alumina particles in Nano size via control side hole diameter and stirring velocity, where the side hole diameter is very effective factor on alumina particle size. The particle size decreases from several microns at (900µm) side hole DIA. to Nano size (≥100nm) at side hole DIA. 500µm with manual stirring. Increasing stirring velocity has led to decrease in alumina particle size which gives the ability to obtain alumina particles ≤50nm at stirring velocity 2280 RPM with side hole DIA. 900µm. The decreasing in the side hole diameter and increasing in stirring velocity lead to decrease in alumina particles size from several microns toward Nano size, but it has a negative influence on alumina particles distribution.

Keywords: In-Situ (Oxidation) Method, Nano Alumina Particles.

تأثير عوامل طريقة ال in-situ على حجم وتوزيع الحبيبات في المواد المتراكبة ذات اساس من الألمنيوم

الخلاصة:

في هذا البحث ، المواد المركبة ذات اساس من الألمنيوم قويت بحبيبات من الألومينا باستخدام طريقة **in-situ** (الأكسدة) . تأثير كلا " من قطر الثقب الجانبي ، سرعة وطريقة الخلط على حجم حبيبات الألومينا وتوزيعها ضمن المعدن الأساس قد درست بشيء من التفصيل . اختبارات حيود الأشعة السينية والمجهر الإلكتروني الماسح قد استخدمت لتشخيص حبيبات الألومينا . من خلال النتائج تبين انه من خلال السيطرة على قطر الثقب الجانبي وسرعة الخلط اصبح من الممكن الحصول على مواد مركبة ذات اساس من الألمنيوم مقواة بحبيبات بحجم النانو متر. كذلك تبين ان لقطر الثقب الجانبي تأثير فعال على حجم حبيبات الألومينا حيث تناقص حجم حبيبات الألومينا من عدة مايكرو مترات عند قطر الثقب الجانبي(**µm**٩٠٠) الى حجم النانو ((**nm**١٠٠≤) عند قطر الثقب الجانبي (**µm**٥٠٠) باستخدام الخلط اليدوي. الزيادة بسرعة الخلط ادت الى النقصان بحجم حبيبات الألومينا حيث اصبح من الممكن الحصول على حبيبات الألومينا بحجم **nm**٥٠≥ باستخدام سرعة خلط ٢٢٨٠ دورة/ الدقيقة وقطر الثقب الجانبي **µm**٩٠٠. كذلك اظهرت النتائج، مثلما لزيادة سرعة الخلط ونقصان قطر الثقب الجانبي تأثير ايجابي بنقصان حجم حبيبات الألومينا من عدة مايكرو مترات الى حجم النانو كذلك لها تأثير سلبي على توزيع حبيبات الألومينا ضمن المعدن الأساس.

INTRODUCTION

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Metal matrix composites were synthesized by two main methods ex- situ and in-situ. In the first synthesis route the reinforcements were added to the metal matrix, while the in-situ processes refer to those methods leading to generation of reinforcements by reaction between components during the process [1]. In-situ method attracted researcher's interests because of low costs and less hazards because the fine particulate reinforcement phases are eliminated and there is no surface contamination of the reinforcements [1, 2].

In - situ method could be classified according to state of the compounds into solid – solid (powder technology), solid – liquid, gas –liquid and vapor-liquid-solid [3-8]. The reinforcement can be (oxide, nitride, and boride) with different forms (particles, whiskers or short fibers) [9]. Commercial application of gas-liquid in-situ processing technologies requires an understanding of several key processing steps. Nevertheless, common processing features of gas-liquid (oxidation) in- situ, including fabricating routes themselves, are not easily found out. Moreover, the mechanisms responsible for the gas-liquid in - situ formation of ceramic reinforcements in the metallic matrix are not well understood. This study is continuing to previous work where Aluminum matrix composite reinforced with Alumina particles prepared by gas- liquid in-situ method (oxidation)

Effects of process temperature, oxygen gas flow rate on alumina particles size and the percent of alumina in Aluminum matrix composites were studied. It was found that alumina percent will increase when process temperature increase for there more no significant effects of process temperature on alumina particle size where the obtained alumina average particle size was 3±1μm at process temperature (720,740,760 and 780^{0}C) respectively .also it found The minimum flow rate (50 ml/min) is not favorable to prepare metal matrix composite by gas- liquid In-situ method [10].

Current study is concentrating on using gas –liquid (i.e. Oxidation) in-situ method. This study aimed to enrich the knowledge about gas-liquid in-situ (i.e. Oxidation) methods by studying the effective parameters that affecting on alumina particles size and its distribution in aluminum matrix composites and how to control this effective parameters.

Another aim is to put controlled standard process of gas –liquid (Oxidation) in-situ method to prepare master aluminum matrix reinforced composites with alumina particles.

Experimental work

In this work Aluminum matrix composite reinforced with Alumina was prepared by the gas –liquid (Oxidation) in - situ method. Commercial aluminum wire was used as raw fabric with chemical composition Al- 1.4 wt**. %** Fe - 0.558 wt**. %**Si - 0.042 wt. % Mg.

250g Aluminum wires were melted in silicon carbide crucible using an electric resistance furnace (0 -1200 ±5^{0}C) under Argon gas atmosphere with flow rate (2 l/min). After furnace temperature reached gas –liquid In-situ process temperature which was740^{0}C and be ensured that Aluminum wires completely melted, then Oxygen gas was pumped into the molten Aluminum inside the furnace in rate of (150ml/ min) by using stainless steel tube with 12 millimeter diameter to generate alumina particles. One end of the stainless steel tube is closed; other end is connected to oxygen tank. Near this closed end there are 4 holes on the tube side. In-situ process temperature was 740^{0}C

and Oxygen gas was flowing time 2 minutes which represented in-situ process time. After 2 minutes from the beginning of in-situ process, Oxygen gas stopped with continues stirring for additional 2 minutes.

A 2.5g (1%) of Mg Alloy with chemical composition Mg -2.67 Wt. % Al - 0.679 Wt. % Zn -0.369 Wt. % Zr - 0.0233 Wt. % Fe, was appended to enhance the wettability between Al matrix and Alumina particles.

After sufficient time for Mg Alloy melting, the in-situ process product was restored then burred in tool steel mold which preheated to 250^0C.

- **Effecting Side hole diameter**

To study the effects of side hole diameter on Alumina particle size, the 4 holes which were on stainless steel tube side drilled with (900μm) diameter for each hole, and that had repeated with another three tubes but with a side hole diameter (800,600 and 500 μm) respectively.

- **Effecting of stirring method and stirring velocity.**

Effects of stirring method and stirring velocity on Alumina particle size were studied, where 4 stirring velocities were used (manual string, 1770, 1950 and 2280 rpm) at constant side hole diameter. Stirring method effect was examined by using three stirring methods at constant of side hole diameter and stirring velocity which are:

1. First was by manual movement of the stainless steel tube into the molten Aluminum along with process time which mentioned above.
2. The second was by immersing stainless steel tube into the molten Aluminum and it then a separated electric stirrer for stirring molten Aluminum was used.
3. The third stainless steel tube was used as stirrer its self by connecting directly to the stirring fan(*two blades ,60mm fan diameter ,The oblique angel of the fan is 45°*) on the closed end positioning it on electric stirrer as shown in Figure 1.

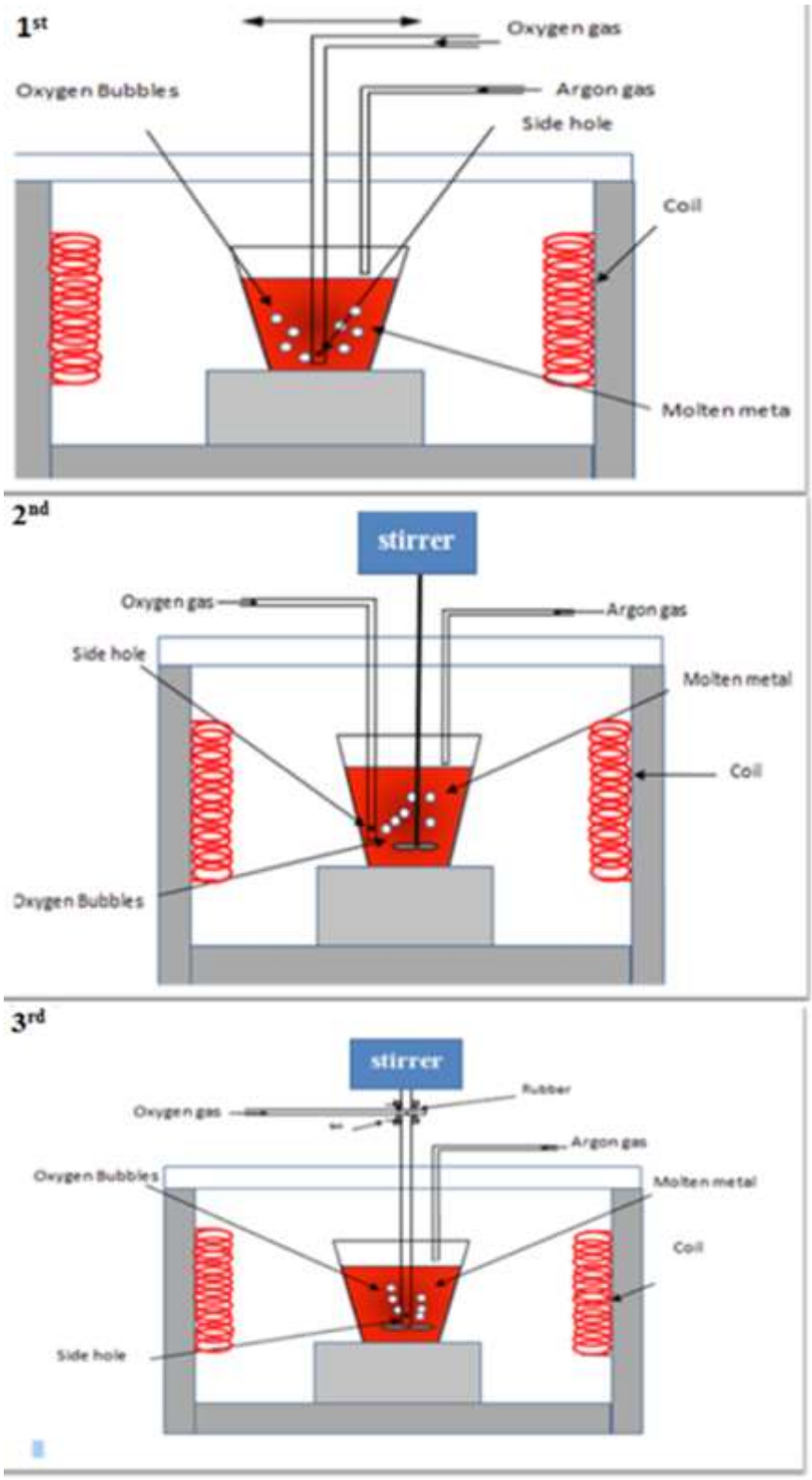


Figure. 1 illustrated 1st ,2nd and 3rd stirring methods

- **X-ray Diffraction (XRD) Test**

X-Ray Diffraction test was done by SHIMATZO 6000X - Japan for Aluminum Matrix Composites samples to ensure that Alumina particle was obtained with measuring condition as below. This test is done at Specialist Institute for Mechanical Industries, Baghdad, Iraq.
Target: Cu, Wave length= 1.5406 A, 2Theta range = 20 -70 deg.

- **Scanning Electron Microscopy (SEM) Test**

Metal Matrix Composite samples were characterized by HITACHI S-4160 Scanning Electron Microscope at the electrical engineering department, Tehran University- Iran.

Results and Discussion

From XRD test, Alumina was obtained in this employment by using In-situ method, as shown in Figure2.

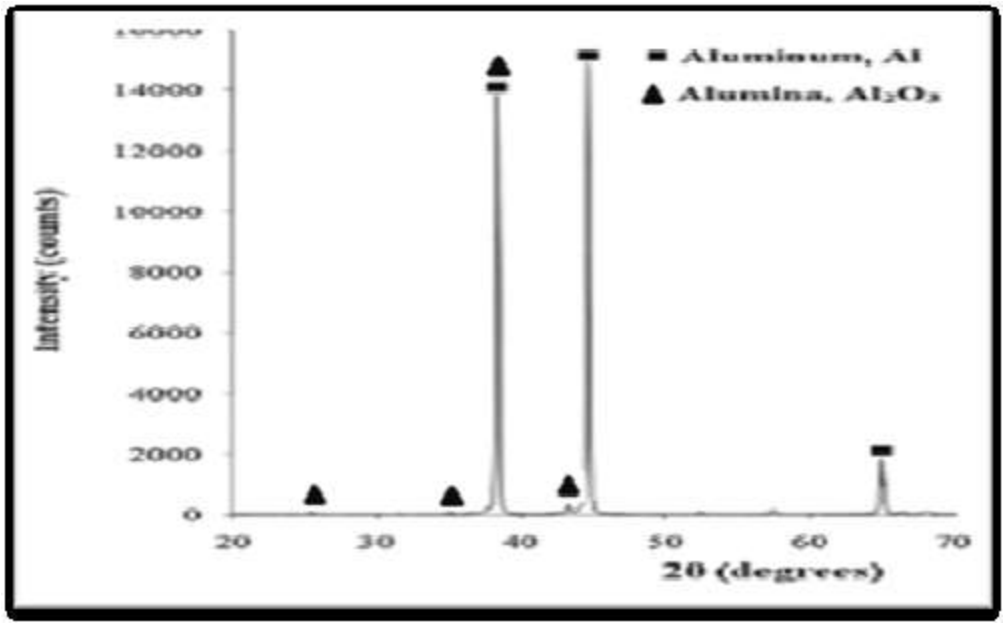


Figure 2. XRD pattern of samples

SEM images showed that Alumina particle size has been affected by three parameters Side hole diameter, Stirring velocity and Stirring method.

Effecting of side hole diameter.

Figure 3, showed the particle size is ≥1 µm at side hole 900µm and reaches Nano size (≥100nm) at side hole diameter 500 µm by first stirring method. It can be deduced that, the particle size of which has decreased when in time with side hole decreases, the reason might be the decrease of bubble size because the side hole diameter decreases.

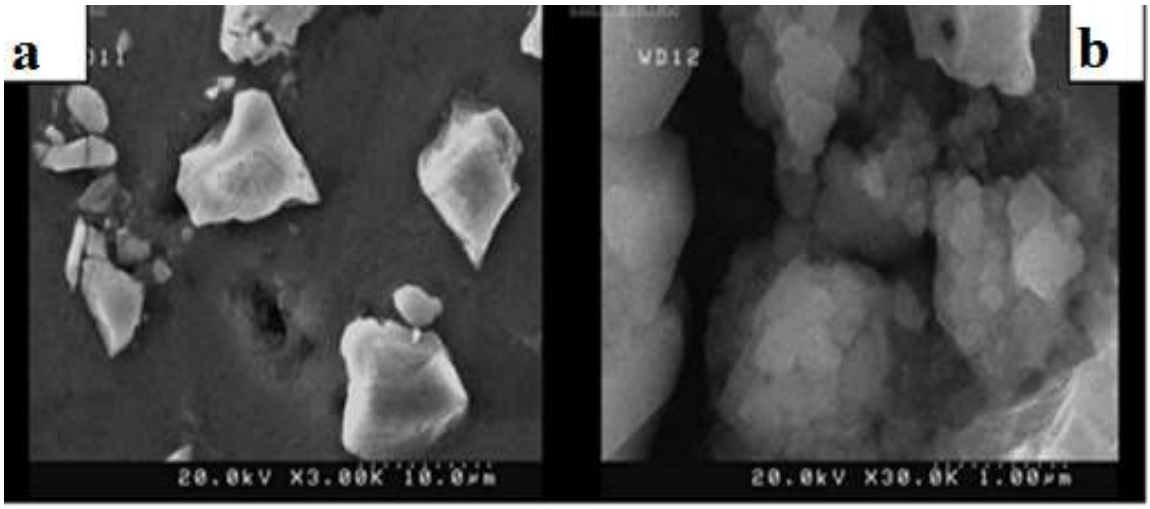


Figure 3 SEM images Illustrated effects of side hole dia. On alumina particles size a) at 900µm side hole dia. , b) at 500 µm side hole dia. by first stirring method

- **Effecting of stirring velocity.**

Figure 4 shows that at stirring velocity 1770 rpm , the Alumina particles size was between (100nm to 500nm) ,and at stirring velocity 2280 rpm alumina particles size was ≤ 50 nm with side hole 900µm by second stirring method. The decreasing of particles size with increasing stirring velocity was found in third stirring method too. Decreasing in particles size with increasing stirring velocity due to increase of the chopping oxygen gas bubbles of which would chop to small one, and this chopping would be continued with increasing in stirring velocity to smallest, and that will lead to decrease the alumina particles size.

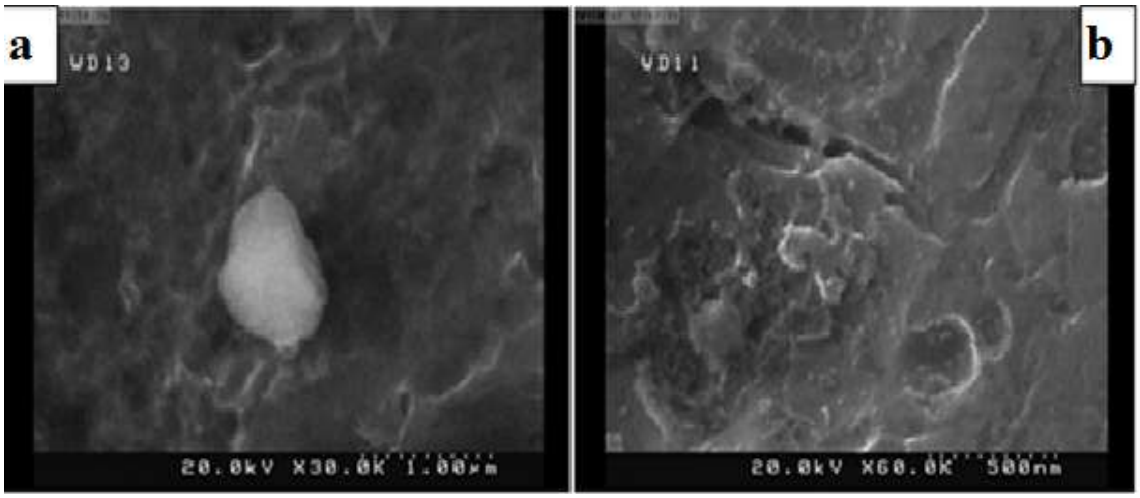


Figure 4 SEM images illustrated effects of stirring velocity on alumina particle size a) at 1770 rpm stirring velocity, b) at 2280 rpm stirring velocity with hole dia.900µm by second stirring method

- **Effect of stirring method.**

The effect of stirring methods on alumina particles size is clear by comparing between first stirring method from hand with second and third stirring methods from another hand where at second and third stirring methods will be producing smaller particles size than particles size which produced in first stirring method SEM images shows that in figures 3, 4 and5.

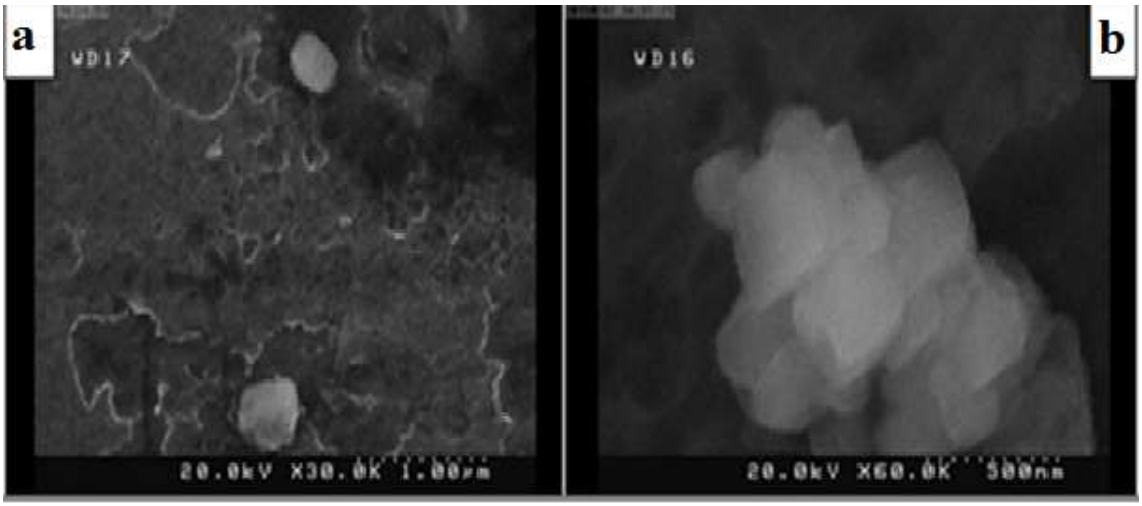


Figure 5 SEM images Illustrated alumina particle size a) at 1770 rpm stirring velocity, b) at 2280 rpm stirring velocity with hole dia.600µm by third stirring method

But comparison between second and third methods was difficult because it couldn't obtain a single alumina particle to measure its size but just clusters, in general in both methods at side hole 500µm and stirring velocity 2280 rpm alumina particles size were ≤70nm.

The addition of 1% Mg did not improve the wettability between alumina particles and aluminum matrix as shown in Figure 6. During grinding and polishing of the prepared composites show alumina particles pull out from the matrix and leaving interstices behind it as show in figure 6. The decreasing in alumina particles size to ward Nano size means increasing in surface area and the addition of 1% Mg is not sufficient to capture all alumina particles to form interface between alumina particles and aluminum matrix. The addition of fresh Mg is more effective as compared to pre-alloyed Mg[11].

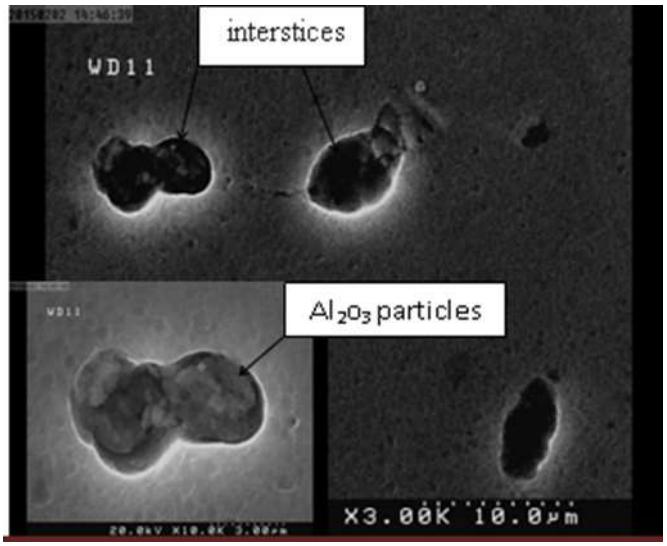


Figure 6 illustrated interstices in Al matrix composites

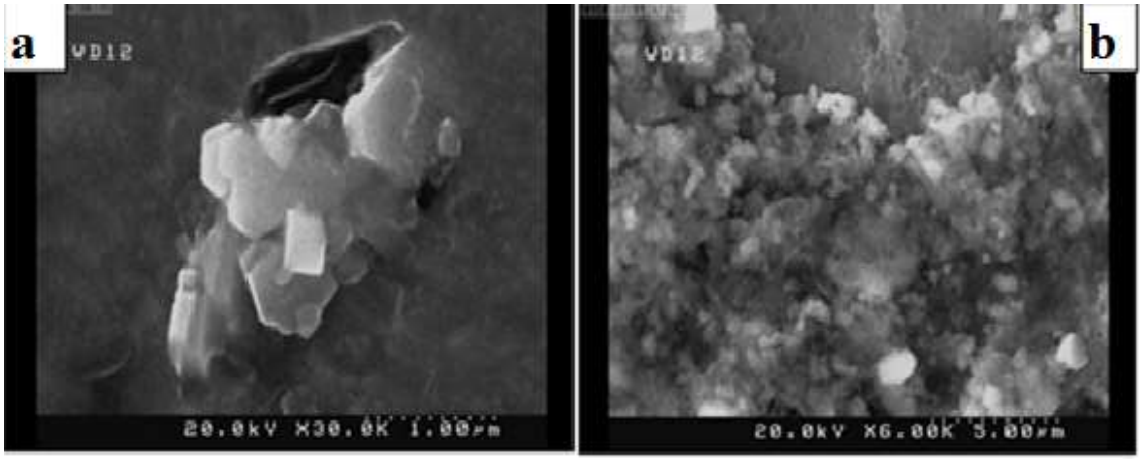
- **The distribution of alumina in aluminum matrix.**

From figures 7 it is noted that alumina particles at two sizes (micron and Nano) were agglomerated in two forms (particle – particle) form to constrict alumina particle groups in condition and clusters in other condition will discussed below, and (cluster – cluster) form to constrict chain-like alumina clusters [12].

In general, Alumina particles agglomeration occurs when there was a convergence between particles in molten aluminum to form groups or cluster. In this work Convergence between particles are resulted by convergence between oxygen gas babbles when it go out from500µm side hole diameter at first stirring method , in order to form clusters or chain-like alumina clusters, at the second and third stirring methods the convergence between particles or clusters are resulted from stirring power also, when stirring power increase convergence between particles or clusters will increase this lead collisions and sticking probability will increase too[13]. In this work, cluster and chain-like alumina clusters were formed at high stirring power (1770, 1950and 2280 rpm) in second and third stirring methods as show in figures 7(a and b).

The convergence between particles or clusters will lead to sticking between particles or clusters themselves based on Van der Waals force and capillary force applied by the melt surface tension which made particles have an ability to stick together causing alumina particles agglomeration[14].

The decreasing in side hole diameter and increasing in stirring velocity lead to decrease in alumina particles size from several microns toward Nano size, but it has a negative influence on alumina particles distribution.



Figures 7 illustrated a) particle-particle agglomeration and b) cluster-cluster agglomeration

In this work there were some practical results with it the preparation process will dune successfully which is:

-Stainless steel tube, stirring fan and skimming tool should be pre heated in melting furnace before immersing it in the molten aluminum to avoid any chilling for molten aluminum that will lead to close side holes or decrease in molten temperature (process temperature).

- It noted that when using 500µm side hole diameter the hole was closed by chilling aluminum more than other side hole (600,800 and 900 µm), therefor Stainless steel tube with hole diameter 500µm should be heated for longer time.

- After each run Stainless steel tube should be cleaned from sticking aluminum which Cause close side holes and the stirrer should be cleaned too.

CONCLUSIONS

According to the above results and findings, the following can be concluded:

1- Aluminum matrix composite which reinforced by alumina particles was obtained successfully through gas-liquid (oxidation) method in micron and nano sizes via control side hole diameter and stirring velocity with the same low cost .

2- Aluminum matrix nano composites were obtained ,where particle size decreasing from several microns (≥1 µm) at side hole 900µm and reaches Nano size (≥100nm) at side hole diameter 500 µm by first stirring method.

3- Aluminum matrix Nano composites also obtained when stirring velocity increased from manual stirring velocity into 2280 r.p.m, where alumina particles size decreasing from several microns to less than 50nm at constant side hole diameter.

4- Addition of 1% of Mg pre- alloyed wasn't sufficient to enhance the wettability between aluminum matrix and alumina particles when alumina at nano size.

5- The decreasing in side hole diameter and increasing in stirring velocity lead to decreasing in alumina particles size from several microns to ward nano size but it have a negative influence on alumina particles distribution.

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