Preparation of Aluminum Matrix Composite Reinforced with Alumina Particles by In-Situ Method

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ABSTRACT

In this work an Aluminum matrix composite reinforced with alumina prepared by in-situ (liquid-gas) method were investigated. Gas was represented by an oxygen with flow rate 50, 100, and 150 Sm/ min (Standard mile Liter/minute) at different temperature (720, 740, 760 and 780 °C). The effects of reaction temperature on alumina particle size and alumina volume fraction were studied extensively. Resulted alumina was identified by using XRD and SEM techniques. The resulted composites were characterized using density and hardness tests. It is found generally, that the alumina particle size was (3 ± 1 µm), the density and hardness are decreases as the alumina particles weight percent increases. The reason behind such behavior was the increment in porosity around alumina particles and also to the weakness in wettability between alumina and metal matrix. Furthermore, it is noted that, there are many problems need to be resolved like segregation, agglomeration and weak of wettability are occurred between the particles surface and matrix.

Keyword: Aluminum Matrix Composite, In-Situ Technique.

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In-Situ Technique.
INTRODUCTION

Aluminum alloys are used in advanced applications because their combination of high strength, low density, durability, machinability, availability and cost is very attractive compared to competing materials. However, the scope of these properties can be extended by using Aluminum matrix composite materials. Composite materials technologies offer a unique opportunity to tailor the properties of aluminum. This could include increased strength, decreased weight, higher service temperature, improved wear resistance, higher elastic modulus, controlled coefficient of thermal expansion, improved fatigue properties [1].

There is a multitude of fabrication techniques of metal matrix composites depending on whether they are aimed at continuously (Continuous fiber composites) such as Liquid state techniques and Solid state techniques or discontinuously reinforced composites such as Solid state routes, Liquid state route and Spray methods reinforced MMC production. The techniques can further be subdivided, according to whether they are primarily based on treating the metal matrix in a liquid or a solid form [2, 3].

A number of problems associated with these conventional processing techniques in fabrication of MMCs tend to limit the widespread use of these materials in many applications. These problems include high production costs, poor mechanical properties caused by weak interfacial strength between reinforcements and matrix materials, inadequate performance reliability and limitations on the sizes and shapes of the parts that can be produced. It is, therefore, necessary to develop new processing techniques for MMCs in order to overcome these problems [1, 4].

In recent years, various new processing techniques have been developed for the production of composite materials utilizing the in-situ synthesis methods which characterizes by fine reinforcements, clean interface between matrix and reinforcement and good mechanical characterization [4, 5, and 6].

Zhang Xiuqinga et. al. were Prepared Magnesium matrix composites reinforced with TiC particulates by New In-situ Synthesis Method. Remelting and dilution (RD) technique is one of the in-situ synthesis methods. The RD technique contains two steps. Prefabricated block that consists of reinforcements is prepared firstly, and then prefabricated block is diluted into metal matrix melt to synthesize composites. The results showed that mechanical properties of the composites increase much. But the reaction of Al-Ti-C perform in molten magnesium is precarious and uncontrollable [7].

Trojanova et al. was prepared Mg-1%Al₂O₃ materials using powder metallurgy technique. Only the damping capacity of the materials increased due to the improvement of dislocation density [8].

The aim of this work is study effects of In-Situ method parameters (temperature and oxygen gas flow rate) on Alumina particle size additional to study physical and mechanical properties for Aluminum matrix composites.

EXPERIMENTAL WORK
In this work Aluminum matrix composites reinforced with Alumina were prepared by in-situ method. Aluminum wire used as raw material, its chemical composition illustrated in Table (1).

<table>
<thead>
<tr>
<th>Elements</th>
<th>Al</th>
<th>Fe</th>
<th>Si</th>
<th>Mg</th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
<td>98</td>
<td>1.4</td>
<td>0.558</td>
<td>0.042</td>
</tr>
</tbody>
</table>

125g Aluminum wire put in silicon carbide crucible then put in electric resistance furnace, the furnace turn on to melt Aluminum wire, Argon gas with flow rate 1SL/Min was charged from the beginning to prevent the oxidation. When Aluminum wire melt, Oxygen gas was charged by using stainless steel tube with 12 mm diameter, one of tube ends was closed, near this closed end there was 4 holes on the tube side with 2 mm diameter for each, this closed end entered into molten metal with manual movement, as shown in Figure 1. different temperature(720,740,760,780⁰C) and different Oxygen gas flow rates (50,100,150 ml/Min) were used to study the effect of temperature and Oxygen gas flow rate on particle size and percent of Alumina, Oxygen gas flowing time was 2 minutes, when Oxygen gas flowing time finished, stainless steel tube pulled up. At the same time the final product left to solidify in the furnace under Argon gas.

Figure (1) Illustrated in-situ process system.

1. X-Ray Diffraction (XRD) Test
After samples preparation by grinding and polishing, X-Ray Diffraction test was done by SHIMATZO 6000X for Aluminum Matrix Composites samples to ensure that
Alumina particle was obtained with measuring condition as below. This test done at Specialist Institute for Mechanical Industry, Baghdad, Republic of Iraq.

Target: Cu, Wave length= 1.5406 A, 2Theta range= 0-70 deg.

2. Scanning Electron Microscopy Test

Metal Matrix Composites samples were characterized by HITACHI S-4160 Scanning Electron Microscope (SEM) at electrical engineering department, Tehran University, Islamic republic of Iran.

3. Average Particle Size Measurements

From Scanning Electron Microscopy images and by using AutoCad 2010 Program Alumina average particle size was measured. Relationship between temperature and average particle size at different Oxygen gas flow rates was studied.

4. Alumina Percent Measurements

Scanning Electron Microscopy (SEM) images was divided in to equal areas by using photo shop program then Alumina volume fraction was measured by Arial fraction method according to equation 1 [9], as shown in Figure 2. Relationship between temperature and Alumina volume fraction at different Oxygen gas flow rates was studied.

\[ A_A = A_α / A \] ... (1)

Where:
- \( A_α \): summation of alumina sections area exits in total area.
- \( A_A \): percent of alumina.
- \( A \): total area.

![Figure 2](image.jpg)

Figure (2) Illustrated Areal fraction method pattern used to measuring alumina volume fraction.

5. Density Test

ASTM D 792 standard was used in all density measurements, which are base on Archimedes principle. The specific gravity of material is given by equation 2. This test repeated for 3 samples from each Alumina percent, finally the average was tacked as result. Relationship between temperature and density at different Oxygen gas flow rates was studied.
Sp.Gr of material = \[ \frac{W_1}{W_1 - W_2} \] * Sp.Gr. of acetone

\[ \text{Where} \]
Sp.Gr. = Specific gravity of material
W_1 = weight of material in air
W_2 = weight of material suspended in acetone.

6. Hardness Test
Brinell hardness was measured by using steel ball with diameter 10 mm with applied load 5 KN for 10 seconds to measure metal matrix composites samples hardness, to study the relationship between hardness and process temperature.

RESULTS AND DISCUSSIONS
From (XRD) test, Alumina was obtained in this work by using In-situ method, as shown in Figure (3) this can be explained from thermodynamic hand, according to Ellingham diagram for Aluminum metal oxidation reaction at elevated temperature which explained the relationship between Gibbs free energy and reaction temperature as shown in Figure(4). It can be noted that Aluminum oxidation react is favorable at elevated temperature, this led to form Alumina as steady phase, equation 3 explained that [10].

\[ \Delta G(T) = 1117992.69333 + (11.10154T \log T) + (244.45717333T) \ldots (3) \]

Figure (3) XRD pattern for samples.
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From SEM images for Alumina particle as shown in Figure (5), it can noted that Alumina particle size was increases with reaction temperature increases at Oxygen gas flow rates (100,150 ml/min), this could be due to the free energy which accelerated reaction with reaction temperature increases then led to produce large particles.

According to particles size at flow rate (50 ml/min), the relationship between Oxygen gas flow rate and reaction temperature wasn’t clear, it could be due to the flow rate was too low this led to the Oxygen gas was go out from the holes by batches, this led to the Oxygen gas bubbles have variable size this was the cause of random particle size, as shown in Figure (6). In general there was no significant changing in particle size (3±1µm) this could be due to the weak effect of reaction temperature and there were other factors (stirring velocity, holes diameter, Oxygen gas jet mechanism) had effect on Alumina particle size, it didn’t studied it in this work.
Figure (5) SEM images for samples A, B, C at oxygen gas flow rate 50, 100, 150 ml/min respectively and 1, 2, 3 and 4 at reaction temperatures 780, 760, 740, 720°C respectively.
For Alumina percent test, we noted that Alumina percent increased with reaction temperature increases at any Oxygen gas flow rate, by other way it can obtains any Alumina percent at any Oxygen gas flow rate at the same process time (2 minutes) but reaction temperature will be variable. It was difficult to obtain exactly Alumina percent with these Oxygen gas flow rats (50,100,150 ml/Min), but it could obtain range of Alumina percent, as shown in Figure (7).

Figure (6) Relationship between alumina average practical size (µm) and reaction temperature (°C).

Figure (7) The relationship between alumina percent and reaction temperature °C.
Figure (8) explain the relationship between density and process temperature, it can be noted that density decreases with process temperature increases.

![Figure 8 Relationship between density and reaction temperature °C.](image)

From Figure (9) Scanning Electron Microscopy (SEM) image we saw there wasn’t any wetability between Alumina and metal matrix this insured by Brinell hardness results as shown in Figure (10), this caused by Capillary forces hinder wetting of the ceramic reinforcement by molten metal and viscous drag through perform interstices, this led to reduces density and Brinell hardness with process temperature increases ( porosity increase with Alumina percent increases led to Metal Matrix Composites density decrease) Evans et al. [2]

![Figure 9 SEM image illustrated interstices around alumina particles.](image)
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Figure (10) Illustrated samples hardness with process temperature.

It can be noted there was alumina particle segregation and agglomeration at grain boundaries this is could be due to the free solidification of the produced metal matrix composite SEM image illustrated that in Figure (11).

Figure (11) SEM images illustrated alumina particles (A) segregation and (B) agglomeration at grain boundaries.
CONCLUSIONS
1. In-situ (liquid-gas) method was used successfully in obtained alumina particles as a reinforcement phase of MMC.
2. In this method it is difficult to control a uniform alumina volume fraction.
3. Poor wettability between alumina particles and aluminum are obtained, so that further investigation should be awarded for improvement.
4. A slight effect of reaction temperature on alumina particle size.
5. Minimum flow rate (50 ml/min) is not favorable to prepare metals matrix composite by In-situ (liquid-gas) method.

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