

# The Effect of Mixing Time and Method on Some Properties of Aluminum Alloy (Al2024) Reinforced With Carbon Nanotubes (MWCNTs)

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## Abstract

This research includes the use of powder metallurgy (PM) in preparing composites based on alloy (Al2024) with adding of (1.5Vol%) of carbon nanotubes (MWCNTs) as a reinforcement material to the form of the two composites (B,C). Mixing powders in amixing times of (0.5,1,1.5,2) hours with using two mixing methods, the first method is mechanical mixing with steel balls, and the second method is ultrasonic mixing method, and it was cold pressed with a pressing pressure of (600MPa) in one direction for the purpose of forming.

The effect of reinforcement, mixing time and method on some physical and mechanical properties of the composites was studied after sintering at a temperature of (550°C) for two hours using an electric furnace and at a heating rate of (10°C/min). The microscopic images and examination of the(EDX) illustrates the activities of strengthening the mechanical bonds and the homogeneity of the elements distribution between the components of the composites by increasing the mixing time. Also the results showed the inverse relationship between the mixing time and each of the experimental density, hardness and compressive strength of the alloy (A) when using the mechanical mixing method, while the relationship is direct in the case of the two composites (B,C) at the same time and mixing method, but with lower percentages and this is similar behavior when using the ultrasonic mixing method.

**Keywords:** Alloy (Al 2024), Mixing, (MWCNTs), Powder Metallurgy ,Ultrasonic.

## Introduction

Many engineering applications require optimum properties such as specific strength, toughness, and rigidity and the ability to form. And these properties are not available in engineering materials such as alloys and plastics, so the need for composite materials appeared which is a mixture of two or more materials with different phases to form a composite that has the required properties and the components may be organic or mineral in the form of particles, fibers or sheets as they give a mixture of properties cannot be obtained from the original materials single one, so they are used in aircraft, ships, cars and other applications. Thus, it appears to us that the term “composite materials” is old, as it appeared from historical information that the inhabitants of Mesopotamia were the first to know the composites and use them in arming the building layers with reed fibers when building ziggurats, in order to stabilize the huge building layers, as for the Babylonians they used reeds and papyrus with bitumen in building boats that are still used in the marsh areas, which considered the first generation of boats, as for the ancient Egyptians they added straw to the mud in manufacturing of bricks to prevent it from cracking during Drying process .In addition the modern techniques and various composite materials were not achieved until the twentieth century [1,2].

Powder metallurgy is one of the most prominent methods of preparation used in composite materials, as it is characterized by the possibility of producing a variety of microstructure and materials that have a combination of the properties that cannot be obtained from metals and alloys formed or hot cast, as well as the possibility of manufacturing products from almost all metals and preparing alloys that cannot be prepared by traditional methods absolutely, it is also possible to control the particle size and the relative homogeneity of the microstructure, and this method is economical and highly productive [3,4].

The researchers were interested in studying the aluminum alloy-based composite materials, as the researcher **R.Peroz - Bustamante et al.** in **2011** studied the effect of mixing time on the microstructure of the aluminum alloy (AL2024) and its composites resulting from adding ratios from carbon nanotubes, as the increase in the mixing time using the mechanical mixing method led to an increase in the size of the base alloy particles, while the increase in the mixing time when adding a specific percentage of carbon nanotubes leads to a decrease in the size of the particles in addition to strengthening the mechanical bonds between the (Al2024 -MWCNTs) [5], the researcher **M. Jafar et al.** in **2012** were interstede in study the mechanical properties of the nanocomposite resulting from adding variable rations of carbon nanotubes (MWCNTs) to the base alloy (Al2024) prepared using powder metallurgy, as the mixing process was done using the mechanical mixing method with Steel balls at intervals of (2,4) hours, the results showed that the best mixing time for preparing nanocomposites is (4) hours [6].

**Farouk M. Mahdi et al.** in **2013** were interested in studying the effect of graphite content and mixing time on the properties of copper-graphite composite prepared using powder metallurgy.The powders were mixed using the mechanical mixing method with time intervals of (1,3,5,7,9) hour, the results illustrated that increasing the mixing time leads to an improvement in the mechanical

and physical properties of the prepared composites and for all graphite ratios [7]. In 2015, researcher **Farhad Ostovan et al.** studied the effect of mixing time on the mechanical behavior of composites (AL-MWCNTs) when using the mechanical mixing method with intervals of (0.5-12) hours, the results showed that increasing the mixing time plays an important role in the distribution of (MWCNTs) inside the aluminum alloy and consequently its effect on the mechanical properties [8]. The researcher **Sonia Simoes et al.** in 2016 studied the Microstructure of the nanocomposites (Al-MWCNTs) using two different mixing methods, the first was done by ultrasonic mixing and the second was done by mechanical mixing with steel balls, the results showed that both of them give the same mechanical properties at the same percentage of (MWCNTs) [9].

The research aims to using powder metallurgy to prepare composites based on aluminum alloy (Al2024) reinforced with a fixed ratio of multi-wall carbon nanotubes in addition to changing the mixing time and method and studying some of their physical and mechanical properties in comparison with the properties of the base alloy.

## Experimental Part

### Powders Used

Table (1) shows the powders used in preparing the research samples.

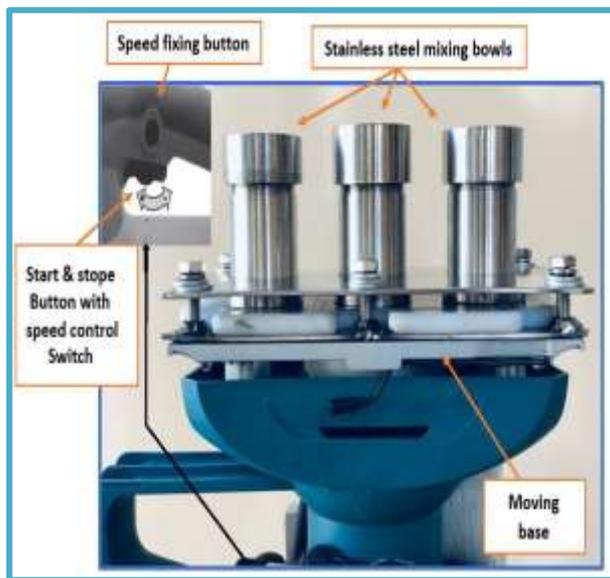
**Table (1): Elemental powders used.**

Metal powder	Purity%	Density g/cm <sup>3</sup>	Particle Shap	Origin
Aluminum	99.8	2.71	Dendritic	Chinese
Copper	99.5	8.94	Irregular	Chinese
Magnesium	98.8	1.73	Dendritic	Chinese
Manganese	99.0	7.44	Dendritic	Chinese
Carbon Nanotubes	95.0	2.10	Multi wall tubes	American

The weights of the components of each model were prepared according to the volumetric ratios and as shown in Table (2). The mixing process of the powders was carried out in two stages. The first included mixing the basic alloy elements with a percentage of (1.5Vol%) of carbon nanotubes (MWCNTs) using a mechanical mixing device that works on a high vibration level and contains the steel balls, as shown in Figure (1) according to [10], with a mixing time of (0.5,1,1.5,2) hours, and mixing the components of the alloy (A) (without addition) at the same time and method of mixing. As for the second stage, it included mixing the elements of The base alloy with the same percentage of (MWCNTs) and time, but using an ultrasonic mixing device (ULTRASONIC HOMOGENIZER \ MODEL 300 V/T) shown in Figure (2).

**Table (2): The volume fraction of the constituent elements of the composites and the method of mixing.**

Code	Al%	Cu%	Mg%	Mn%	MWCNTs%	Mixing time/h	Mixing Method	
A	A1	94.7	3.8	1.2	0.3	-	0.5	Ball Millig
	A2	94.7	3.8	1.2	0.3	-	1.0	Ball Millig
	A3	94.7	3.8	1.2	0.3	-	1.5	Ball Millig
	A4	94.7	3.8	1.2	0.3	-	2.0	Ball Millig
B	B1	93.2	3.8	1.2	0.3	1.5	0.5	Ball Millig
	B2	93.2	3.8	1.2	0.3	1.5	1.0	Ball Millig
	B3	93.2	3.8	1.2	0.3	1.5	1.5	Ball Millig
	B4	93.2	3.8	1.2	0.3	1.5	2.0	Ball Millig
C	C1	93.2	3.8	1.2	0.3	1.5	0.5	Ultrasonic Milling
	C2	93.2	3.8	1.2	0.3	1.5	1.0	Ultrasonic Milling
	C3	93.2	3.8	1.2	0.3	1.5	1.5	Ultrasonic Milling
	C4	93.2	3.8	1.2	0.3	1.5	2.0	Ultrasonic Milling



**Figure (1): Mechanical Mixing Device [10].**

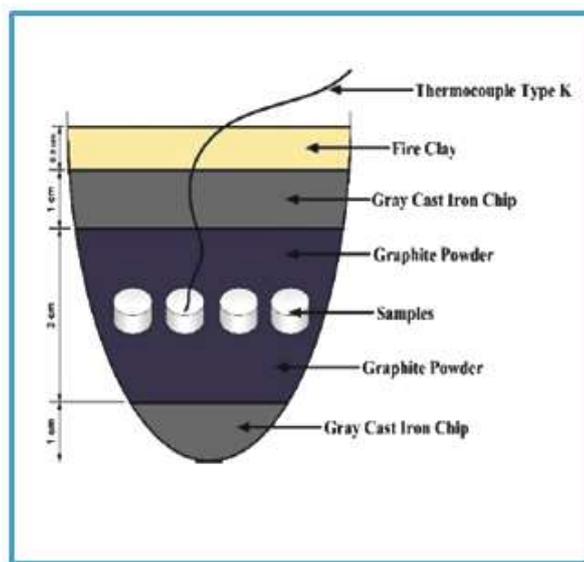


**Figure (2): Ultrasound Mixing Device.**

The green samples were formed through a Uniaxial cold pressing process by using a hardened steel press mold at a pressing pressure of (600MPa) and retention period of (60Sec) to prevent a flexible return to prepare samples with dimensions (10Ø×6) mm using the universal testing machine Figure (3), the sintering process was carried out by putting the samples inside a ceramic vessel containing cast iron sculpor and graphite to avoid oxidation of the samples as shown in Figure (4) according to [7]. The ceramic vessels were placed inside an electric furnace and its temperature was gradually raised at a rate of (10°C/min) until it reached (550°C), this temperature was kept for a period of (2h) and then cooled slowly inside the oven at room temperature.



**Figure (3): The University Test Machine.**



**Figure (4): The Mechanism of putting samples Inside the ceramic vessel container [7].**

The elements were analyzed by using a Japanese-origin device (X-MET8000 Expert Handheld X-Ray Fluorescence Analyzer XRF), which is shown in Figure (5), and using (EDX) system attached to a scanning electron microscope (SEM), while Figures as (6to8) show a photograph of the device screen in includes the results of the element analysis and its distribution map for the sample (B4), respectively.



Alloy 10s. 1:04pm

POSSIBLE MATCH (1/21)

ELEMENT ↓	%	+/-	LIMIT
Cu	5.21	0.032	3.30 - 4.70
Al	91.26	0.610	89.00 - 95.00
Fe	0.20	0.180	0.10 - 0.70
Mg	2.24	0.053	1.00 - 2.75
Mn	0.60	0.021	0.00 - 0.35
P	0.00	0.000	
S	0.10	0.012	
Si	0.39	0.018	0.20 - 0.60

Figure (5): Elemental Analyzer Device.

Figure (6): The results of the analysis of the sample elements (B4) on the (XRF) screen.

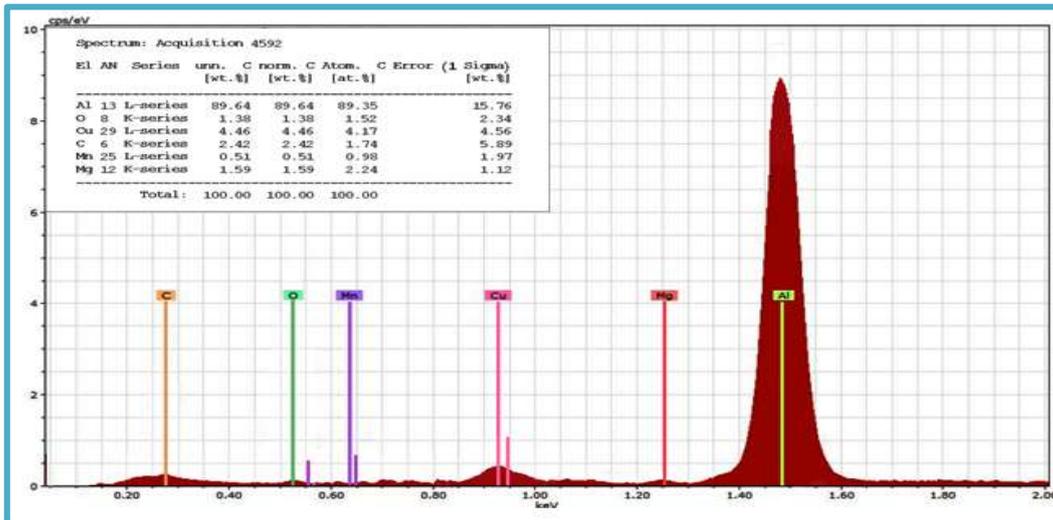


Figure (7): Elemental analysis model for the sample (B4) with the (EDX) system.

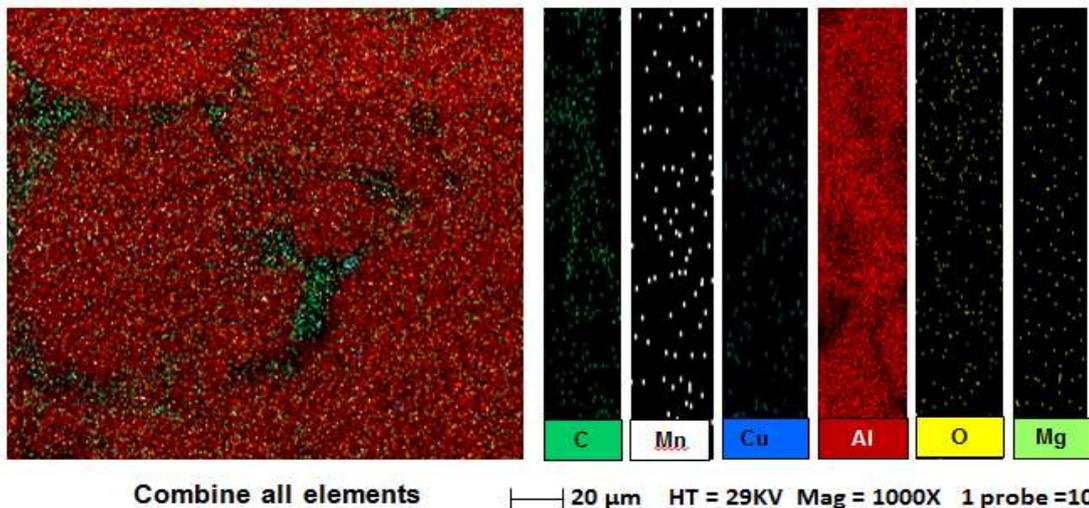


Figure (8): The mapping of the distribution elements in the sample (B4).

## Physical Tests

### Sample preparation

The samples were prepared for the purpose of procedure microscopy test by using smoothing paper of silicon carbide (SiC) with a gradient granular size (400,600,800,1000,1500,2000,2500) in the presence of a continuous water current, after polishing with diamond paste in multiple stages, the solution was used (1ml HF,1.5 ml HCL, 2.5 ml HNO<sub>3</sub>, 95 ml H<sub>2</sub>O) to show the microstructure according to [11], the samples were washed with water, then dried and imaging was performed by using an optical microscope (OLYMPUS) type Japanese origin provided by camera (Amscope-FMA050\9.0Mp) type in addition to scanning electron microscopy (SEM).

### Experimental and theoretical density and real porosity

The experimental, theoretical and true porosity of the prepared samples were calculated by using Archimedes' theory according to the international standard (ASTM B962-08) using equations (1to3) [12].

Since:- 
$$TD = \sum (TD_i \times X_i) \dots \dots \dots (1)$$

TD: Theoretical density (g/cm<sup>3</sup>).

X<sub>i</sub> : The proportion of the element in the sample.

TD<sub>i</sub>: Theoretical density of the sample components (g/cm<sup>3</sup>).

Since: 
$$ED = \frac{Ma}{Ma-Mi} \times Dw \dots \dots \dots (2)$$

ED : Experimental density (g / cm<sup>3</sup>).

Ma: Weight of the dry sample (g).

Mi : Weight of the sample while it is in suspension (g).

Dw: The density of water (g/cm<sup>3</sup>).

Since:- 
$$TP = \left[ 1 - \left( \frac{ED}{TD} \right) \right] \times 100\% \dots (3)$$

TP: True porosity

### X-Ray diffraction

An X-ray diffraction device (SHIMADZU XRD - 6000) was used in the scanning process with a range of (10°- 120°) using a copper target (CuK $\alpha$ ) With a wavelength (1.541874 Å), a voltage of (40 kV) and a current (300 mA).

### Mechanical Tests

#### Hardness test

The indents method was adopted using the micro-hardness device (Mekton-THV-501E), as a load of (300 g) was applied to the sample surface by the indenter tool. An average of five readings was taken and measured in randomly selected areas so that they covered most of the surface area, the average was calculated The five readings, which represents the hardness of the sample.

#### Compressive strength test

The universal testing machine (Fig.3) was used to test the compressive strength of the samples, as its value represents the load applied to the sample divided by the sample area at the moment of failure, and It is calculated from equation (4) [13].

$$\sigma = \frac{2F}{\pi hD} \dots \dots \dots (4)$$

$\sigma$ : Compressive strength (M

F : The force applied to the sample (N).

h : Height (mm).

D: Diameter (mm).

## Results and discussion

### Measurement of experimental density and true porosity

The Figure (9) shows the relationship between the mixing time and the experimental density of the alloy (A) and its composites (B,C), as the density of the base alloy decreased by (4.8%) when the mixing time was increased from (0.5-2) hours as a result of the increase in particle size during the mechanical alloying process (MA) accompanying the mechanical mixing process, while the

experimental density of the two composites (B,C) prepared by the methods of mechanical mixing and ultrasound increased by (3.4%) and (3.9%), respectively at the same mixing time as a result of the presence of (MWCNTs) on the granular boundaries of the composite, which acts as a lubricant that enhances compaction and compressibility and thus increases the experimental density. In the mixing time of (2) hours, the experimental density of the two composites (B,C) decreased by (6.7%) and (8%) comparison to the base alloy and this is consistent with what the researcher found [5] of the similar behavior for each of the base alloy and its composites, the Figure shows (10) X-ray diffraction examination results for each of the base alloy and its composites prepared by the two mixing methods under study and for a fixed mixing time of (2) hours. The results of the examination showed the formation of metallic composites (AL-Mg, AL-Cu, AL-Mn, AL-Cu-Mg) Which have peaks of different intensities that rely on the properties of the components of the base alloy and their composites, and the homogeneity of the distribution of the elements Figures (6to8).

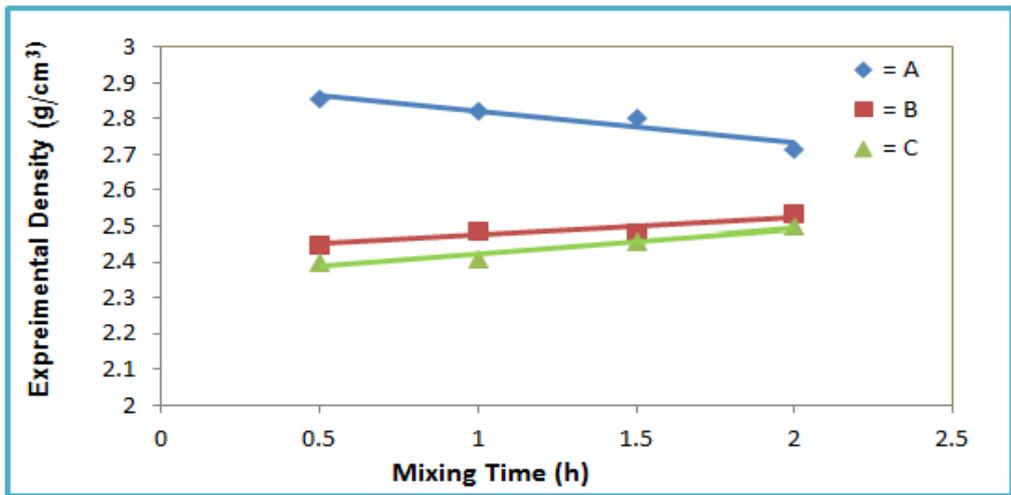


Figure (9): The relationship between the mixing time and the experimental density of samples (A,B,C).

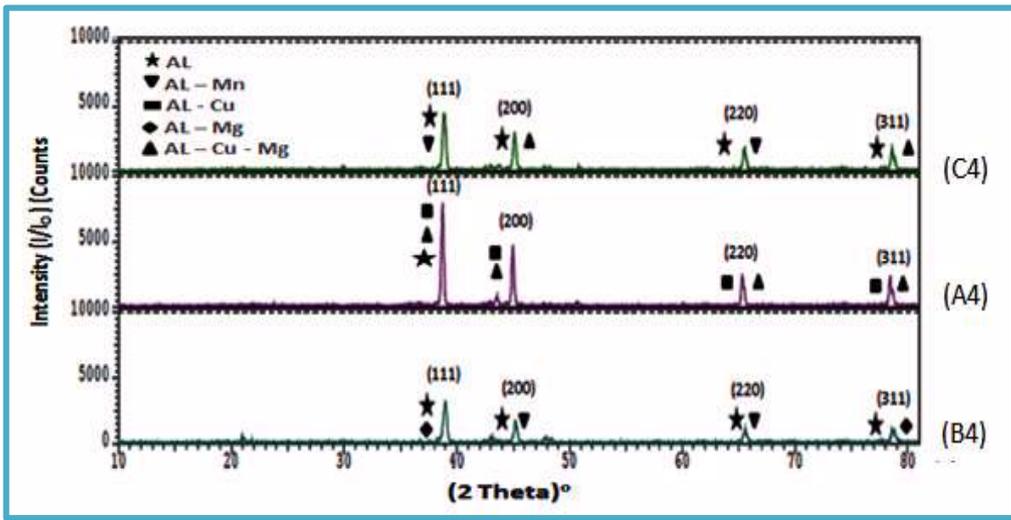
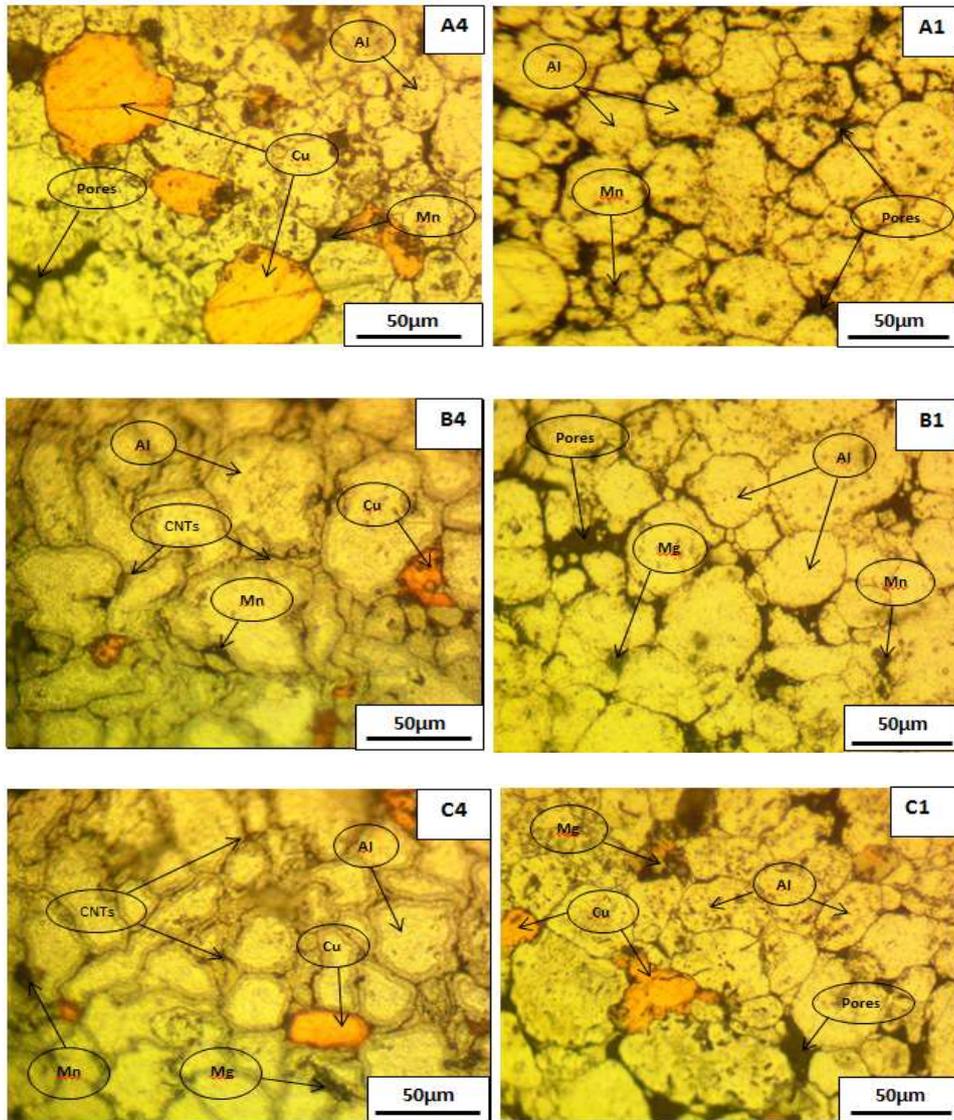


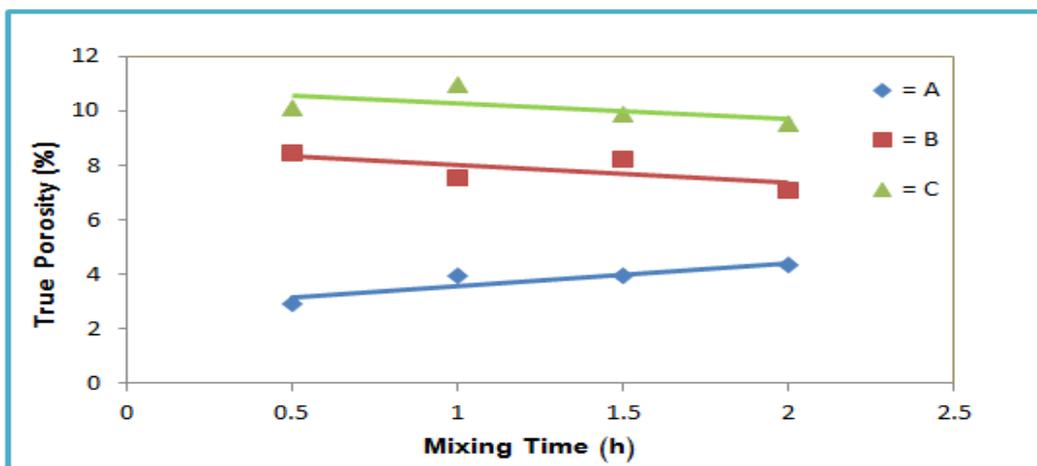
Figure (10): X-Ray diffraction of samples (A4, B4, C4) at constant mixing time (2) hours.

Figure (11) shows the pictures of the microstructure of some samples prepared with different variables in terms of mixing method and time, which show their effectiveness in smoothing the microstructure.



**Figure (11): Optical microscopy images of samples at a mixing time of (0.5, 2) hours using the mechanical mixing method and the ultrasonic method.**

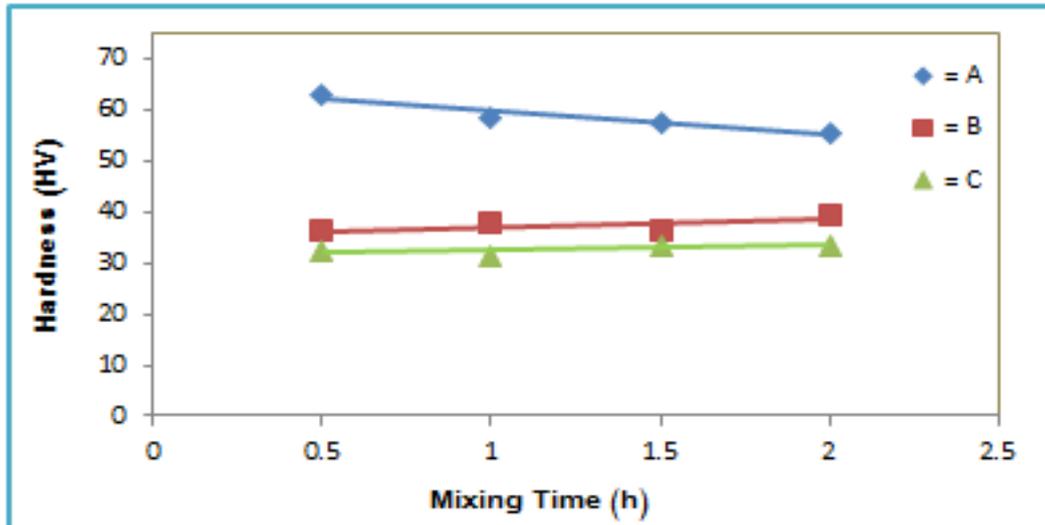
The mixing time increasing led to an increase in the real porosity of alloy (A) by (43%), while it decreased by (14%) and (15%) respectively for the two composites (B,C) when the mixing time was increased from (0.5-2) hours (Fig. 12), but at a mixing time of (2) hours, the real porosity of the composites (B,C) increased by (25%) and (51%), respectively comparison with the base alloy and this is consistent with the decrease in the experimental density of the base alloy and the increase in the experimental density for the two composites (B,C) with an increasing in the mixing time Figure (9).



**Figure (12): The relationship between the mixing time and the true porosity of the samples (A,B,C).**

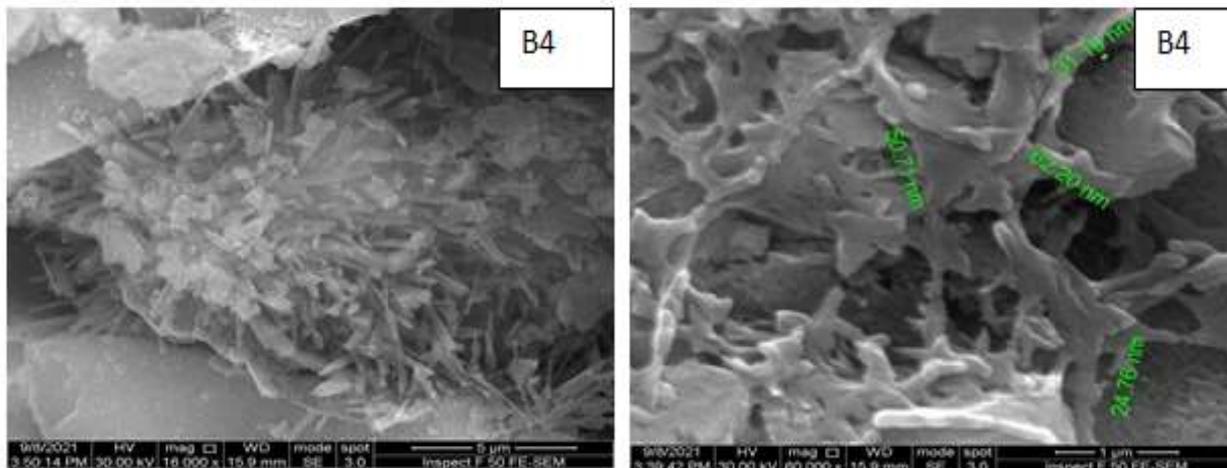
## Hardness

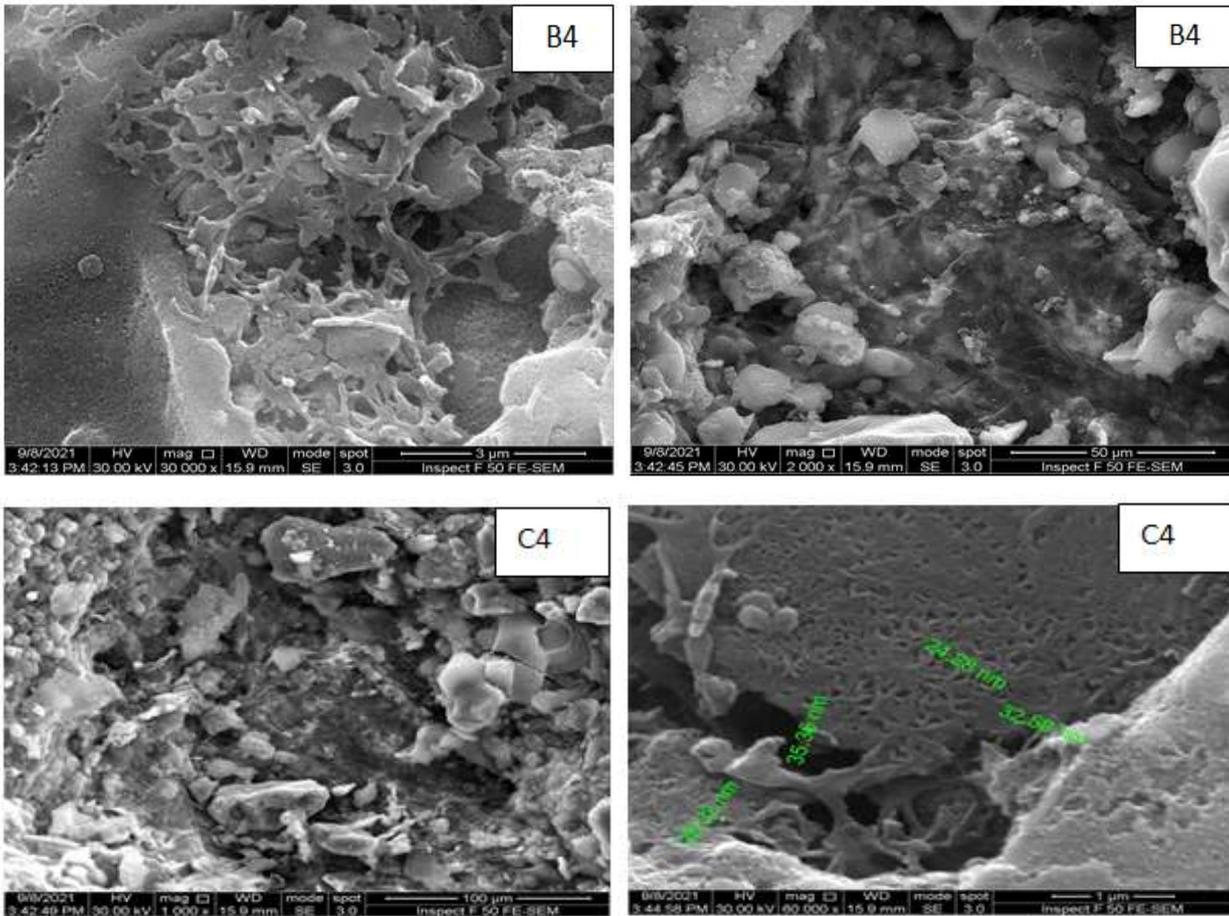
Figure (13) shows the behavior of the relationship between the mixing time and the Vickers hardness for each of the alloy (A) and its composites. A decrease in the hardness of (12%) was observed for the base alloy (A) when the mixing time was increased from (0.5-2) hours, and at the same time the hardness increased for each of the compounds(B,C) in the proportions (9%) and (3.5%), respectively, while the hardness decreased at a mixing time of (2) hours for each of the compounds(B,C) in the proportions (28%) and (39%) respectively from what it is in alloy (A) and this is due to the difference in the ratios of experimental density and real porosity Figures (9) and (12), and in accordance with what the researcher found [5] of similar behavior for each of the base alloy and its composites.



**Figure (13): The relationship between the mixing time and the hardness of the samples (A,B,C).**

Figure (14) shows the SEM images of two composites prepared using the mechanical mixing method and the ultrasonic mixing method at a fixed mixing time (2) hours.

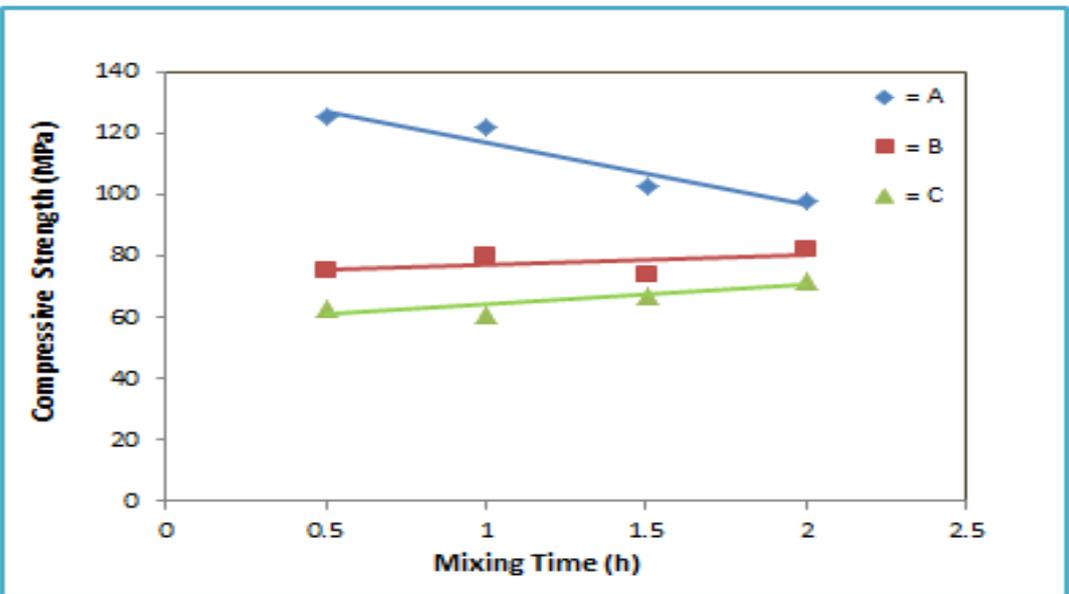




**Figure (14): Scanning electron microscope (SEM) images of two composites prepared by mechanical mixing method and ultrasonic mixing method at a mixing time of (2) hours.**

**Compressive strength**

The effect of increasing the mixing time on the compressive strength is shown in Figure (15), as the compressive strength of alloy (A) decreased by (21%) when the mixing time was increased from (0.5-2) hours, and at the same time the compressive strength of the two composites (B,C) increased in proportions (9.3%) and (14.3%), respectively, but in the case of mixing time (2) hours, the compressive strength of the composites (B,C) decreased by (16%) and (26%), respectively, and this is due to the difference in the values of hardness figure (13).



**Figure 15: The relationship between the mixing time and the compressive strength of the samples (A,B,C).**

## Conclusions

Increasing the mixing time using the mechanical mixing method of the base alloy leads to an increase in the particle size and consequently a decrease in the experimental density, hardness and compressive strength in percentages (4.8%) (12%) (21%) respectively, in addition to an increase in the real porosity for (43%), while When using the same method and the mixing time of the composite resulting from adding a percentage of (1.5Vol%MWCNTS) to the base alloy, the increase in the mixing time led to an increase in the experimental density, hardness and compressive strength in proportions (3.4%) (9%) (9.3%) respectively. The real porosity was reduced by (14%), but when using the ultrasonic mixing method for the same composite and mixing time, the experimental density, hardness and compressive strength had similar behavior than when using the mechanical mixing method, but with some what lower values, as the increase in percentages (3.9%), (3.5%) and (14.3%) respectively, in addition to the low real porosity of (15%).

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