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Effect of Graphite Content and Milling Time on Physical Properties of Copper - Graphite Composites Prepared by Powder Metallurgy Route

[1]Farouk M.Mahdi, [2]Raed N.Razooqi, [3]Saif S. Irhayyim

[1,2,3]Department of Mechanical Engineering, College of Engineering, Tikrit University, Iraq

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ABSTRACT

Copper – graphite composites found many applications such as sliding bearings and electric brushes due to their high wear resistance and excellent electrical and thermal conductivity.Current research aims to study the effect of graphite content and milling time on physical properties of copper – graphite composites made by powder metallurgy route. Copper and graphite powders with 0,5,10,15,20 and 25vol% graphite, were milled mechanically using ball mill for 1,3,5,7 and 9 hours. The milled mixture was cold pressed at 700 MPa for 30 second, followed by sintering at 900 ºC for one hour. In the present work it was found that increasing both milling time and graphite content results in a decrease in the bulk density, the apparent density and the thermal conductivity. It was found that the bulk density ranges between (8.47-5.25) g/cm^3, the apparent density ranges between (8.55-5.7) g/cm^3 and the thermal conductivity ranges between (226.5-89.5) W/m.k. On the other hand both total and apparent porosity were increased with increasing graphite content and milling time. It was also found that water absorption is directly proportional with the apparent porosity and is ranges between (0.1-1.5)%. Microscopic examinations were used to consolidate and interpret the results.

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Nomenclature		
Code	Meaning	Units
K	Coefficient of Thermal Conductivity	W / m . K
Q	The Amount of Heat	W
A	Heat Flow Section Area	m^2
	Thermal Gradient	K/m
	Density of the individual alloying elements	g/cm^3
	Mass fraction of the individual alloying elements present in the composite	/
	Density of Water	g/cm^3
W_d	Dry Weight of the Sample	g
W_s	Water Saturated Body weight	g
W_i	Hanged Body Weight	g
T.D.	Theoretical Density	g/cm^3
A.D.	Apparent Density	g/cm^3
B.D.	Bulk Density	g/cm^3
A.P.	Apparent porosity	%
T.P.	True Porosity	%
W.A	Water Absorption	%
X_1,X_3	Distance Between the Thermocouples on Either Side of the Sample	mm
X_2	Thickness of Sample	mm

INTRODUCTION

composite material is a duplex and multifunctional material composed of at least two elements working together to produce a structural material with mechanical and physical properties that are greatly enhanced compared to the properties of the separately working components. Composites are used not only for their structural properties, but also for electrical, thermal, tribological and environmental applications (François, 2008; Nikhilesh *et al*., 2006; Daniel *et al*., 2001;K. Rajkumar *et al*., 2009).

Corresponding Author: Farouk M. Mahdi, Department of Mechanical Engineering, College of Engineering, University of Tikrit, Iraq,
E-mail: farouk_engineering@yahoo.com.

Composites can be designed to be very strong and stiff and very light in weight too, giving them strengthtoweight and stiffnesstoweight ratios severaltimes greater than that of steel or aluminum. These propertiesare highly desirable in applications rangingfrom commercial aircraft to sportequipment (Daniel *et al*., 2001; S. Suresh *et al*., 1993; Mikell *et al*., 2010).The powder metallurgy technology is one of the most important methods that are used to produce metal matrix composites (MMC). The process of converting metallic powders into ingots or finished components via compaction and sinteringis called powder metallurgy (Azim *et al*., 2011;Brian, 2004).

Powder metallurgy is used wheneverporous parts are needed, whenever parts have intricate shapes, whenever the alloyor mixture of metals cannot be achieved in any other manner, or whenever the metalshave very high melting points (Brian, 2004; Hiroaki *et al*., 2006).

Powder metallurgy is widely used in the production of copper and its alloys which found important applications for their outstanding properties such as high thermal and electrical conductivity and excellent resistance to corrosion.Copper enters with many other materials for the production of compositesthat have superior properties suitable for a number of applications (Wenlin Ma *et al*., 2011; K. Rajkumar *et al*., 2010). Among the most important of these materials is graphite which is considered as a solid lubricant. Copper–graphitecomposites possess the properties of copper, i.e. excellent thermal and electrical conductivities, and properties of graphite, i.e. solid lubricant and small thermal expansion coefficient. Copper – graphitecomposites are widely used as brushes, and bearing materials in many applications (W. Ma *et al*., 2009;T. Futami *et al*., 2008;S.F. Moustafa *et al*., 2002).

Yongping Jin *et al.(2011)* have investigated the densification of Cu – graphite compound powders and found that the relative density decreases with extending milling time. The researchers concluded that pressing 700MPa for 30s produced copper – graphite composite with near full densification.Chandana P. (2012) has studied the effect of process parameters on the hardness and microstructure of copper – graphite composites prepared by powder metallurgy route. He found that the optimum pressure, sintering temperature and sintering time for conventional sintering are 700 MPa, 900 ^{o}C and 1h respectively. He also found that the hardness was increased with increasing milling time which resulteda very fine and uniformly distributed reinforcing particles throughout the matrix. C. Vincent *et al*. (2012) studied the effect of porosity on the thermal conductivity of copper processed by powder metallurgy.They showed that the thermal conductivity decreases as volume fraction of porosity increases.

MATERIALS AND METHODS

1- Fabrication of Samples:

Copper matrix composite was prepared from copper powder (≤ 63 µm in size with 99.7% purity) and graphite powder(≤ 63 µm in sizewith 99.8 % purity). The volume fraction of graphite was (0,5,10,15,20 and 25%).Graphite powder was dried at 200^{o}C for two hours to get rid of moisture and other volatile substances.The powders were milled in a high energy ball milling machine which was designed and produced for this purpose as shown in Figure (1). The milling time was (1,3,5,7,9) hours, weight ratio of balls and powders was (5:1), milling speed was (500 r.p.m) and ball size (diameter) was (10 mm).

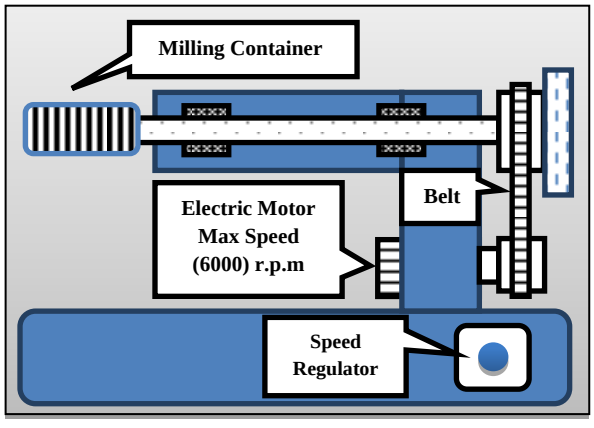

Fig. 1: High energy ball milling machine

The milled powder mixture was cold pressed through uniaxial pressing at 700 MPa for 30 seconds into a cylindrical mold to produce samples with 10mm in diameter and 5mm in height. Cold pressed samples were sintered at 900 ^{o}C for 1 hour followed by slow cooling inside the furnace. To prevent oxidation, the samples were placed in a ceramic container with multilayer graphite powder and cast iron chip and is sealed with fire

clay as shown in fig.(2). Type (K) thermocouple was placed inside the sintering box to monitor and control the sintering temperature. Figure (3) represents a flow diagram of the experimental procedure.

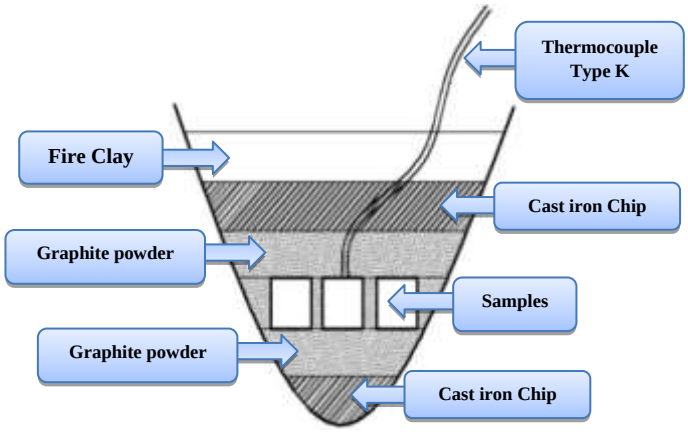

Fig. 2: The container used in the sintering process.

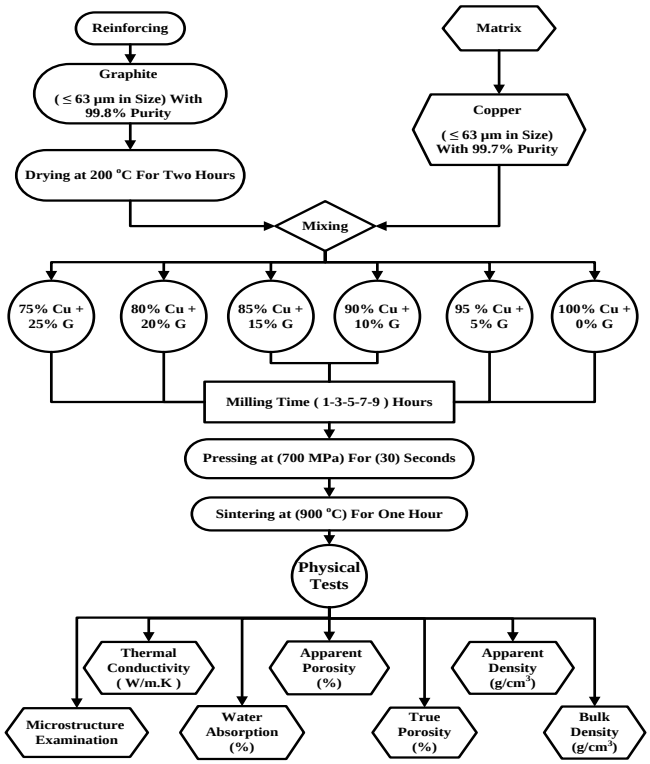

Fig. 3: Flow diagram for the experimental procedure.

2- Physical Properties:

2-1 *Thermal Conductivity:*

Thermal conductivityis the property of a material's ability to conduct heat. The thermal conductivity predicts the rate of energy loss (in watts, W) through a piece of material. It is evaluated primarily in terms of Fourier's Law for heat conduction. Heat transfer occurs at a higher rate across materials with high thermal conductivity than across materials of low thermal conductivity. Correspondingly materials of high thermal conductivity are widely used in heat sink applications and materials with low thermal conductivity are used as thermal insulators. Thermal conductivity is measured in watts per kelvin-meter W/K.m. (Terry M., 2004; Subhash *et al*., 2006, Mohammed S., 2008; Ali I.*et al*., 2011,Ali J., 2011). The Fourier's Law is given by:

$$-\mathbf{K} = {}^{\mathbf{Q}}/_{\mathbf{A} \times (\Delta \mathbf{T}/\Delta \mathbf{X})} \quad (1)$$

Theoretical Density:

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Can be defined as the ratio between the mass to the real size of a material free of pores and voids, theoretical density can be calculated through the following relationship (S. Lowell *et al*., 1984;Goutam D. *et al*., 2012; S. K. Abd – Al Hassan, 2010)*:*

$$\mathbf{T.D.} = \sum_{i=1}^{n} (\boldsymbol{\rho_i} \,.\, \mathbf{X_i}) \quad (2)$$

Apparent Density:

Represents the ratio between the mass and volume of a martial that includes closed pores only. Apparent density is calculated using the following relationship(S. Lowell *et al*., 1984; S. K. Abd – Al Hassan, 2010;M. B. Berger, 2010;ASTM C373 – 88, 2006):

$$\mathbf{A.D.} = \frac{\mathbf{W_d}}{\mathbf{W_d - W_i}} \times \boldsymbol{\rho_w} \quad (3)$$

Bulk Density:

Is defined as the ratio of the mass on the overall size, which includes the actual material and the opened and closed pores. Bulk density can be calculated using the following relationship(S. Lowell *et al*., 1984; S. K. Abd – Al Hassan, 2010; M. B. Berger, 2010;ASTM C373 – 88, 2006; Adnan S., 2013):

$$\mathbf{B.D.} = \frac{\mathbf{W_d}}{\mathbf{W_s - W_i}} \times \boldsymbol{\rho_w} \quad (4)$$

Apparent Porosity:

Is the percentage of opened pores to the overall size of the body. Apparent Porosity can be determined from the following relationship (S. Lowell *et al*., 1984; S. K. Abd – Al Hassan, 2010; M. B. Berger, 2010;ASTM C373 – 88, 2006;Adnan S., 2013):

$$\mathbf{A.P.} = \frac{\mathbf{W_s - W_d}}{\mathbf{W_s - W_i}} \times \mathbf{100}\,\% \quad (5)$$

True Porosity:

Represents the size of the opened and closed pores to the overall size of the body,and can be found from the following relationship (S. Lowell *et al*., 1984; Goutam D. *et al*., 2012; S. K. Abd – Al Hassan, 2010; M. B. Berger, 2010;ASTM C373 – 88, 2006):

$$\mathbf{T.P.} = \frac{\mathbf{T.D. - B.D.}}{\mathbf{T.D.}} \times \mathbf{100}\,\% \quad (6)$$

Water Absorption:

Is defined as the ratio of the volume of opened pores to the body volume, and can be calculated by using the following relationship (S.K. Abd – Al Hassan, 2010;ASTM C373 – 88, 2006;Adnan S., 2013):

$$\mathbf{W.A.} = \frac{\mathbf{W_s - W_d}}{\mathbf{W_d}} \times \mathbf{100}\,\% \quad (7)$$

3- Physical Tests:

Thermal Conductivity:

Thermal conductivity is measured using a heat conduction unit which is manufactured by PA Hilton Ltd company and is shown in Fig.(4). The sample is located between two brass blocks. One block is heated through heating coil while the other is cooled through circulating cooling water. Many thermocouples are used to measure the temperature gradient along the two brass blocks and the supported sample between them. Heating will continue till a steady state is reached between the two sample ends (T_h and T_c) as shown in Fig.(5).

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Fig. 4: Thermal conductivity device.

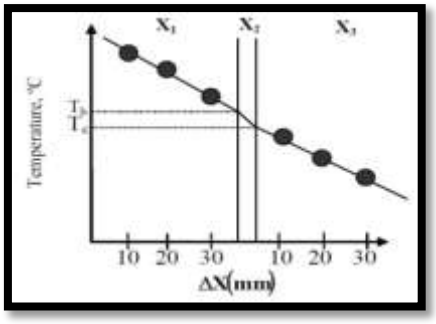

Fig. 5: Thermal gradient curve.

Bulk and Apparent Density, True and Apparent Porosityand Water Absorption:

According to ASTM C373 – 88, the following steps were performed to obtain the above physical properties (M. B. Berger, 2010;ASTM C373 – 88, 2006; Adnan S., 2013):

- Samples were dried for an hour using an electric oven at a temperature of (150 ^{o}C), followed by oven cooling. The dry weight (W_d) for the cooled sample is obtained.
- The dried samples were immersed in a boiling water for (5) hours. The samples are then immersed in a distilled water for (24) hours. The saturated weight (W_S) is found after removing the excess surface water only.
- The suspended weight (Wi) of the samples is determined using suspended sensitive balance (0.1 mg accuracy) while the sample is immersed in distilled water.

The above sample weights were used to calculate the true porosity (T.P.) and apparent Porosity (A.P.), Bulk density (B.D.) and apparent density (A.D.) and the proportion of water Absorption (W.A.) using the relations of (2) to (7).

RESULTS AND DISCUSSION

Effect of Milling Time on Shape and Size of Particles:

It is found as shown in fig.(6), that on increasing milling period, copper particles which have originally a spherical shape tends to flatten and refine into smaller size particles. It is also observed that some of these flattened particles were agglomerated as clusters. The refining behavior is attributed to cold working resulted from blowing effect of milling balls. Cold working is accompanied by work hardening and lack of ductility which leads to a brittle behavior and breaking down of powder particles. While agglomeration is attributed to forge welding of flattened particles under the same blowing effect of milling balls. A similar results were found by (*Chandana P., 2012*) and (H. Zuhailawati*et al*., 2009) in Cu – graphite and Cu – NbC composites respectively. It is also found that dynamic balance between refining by breaking down and agglomeration by cold welding of particles is satisfied on milling for a period of 5 hours or longer, a result which is observed by (KekeGan *et al*., 2008) in Cu – SiC Composite.

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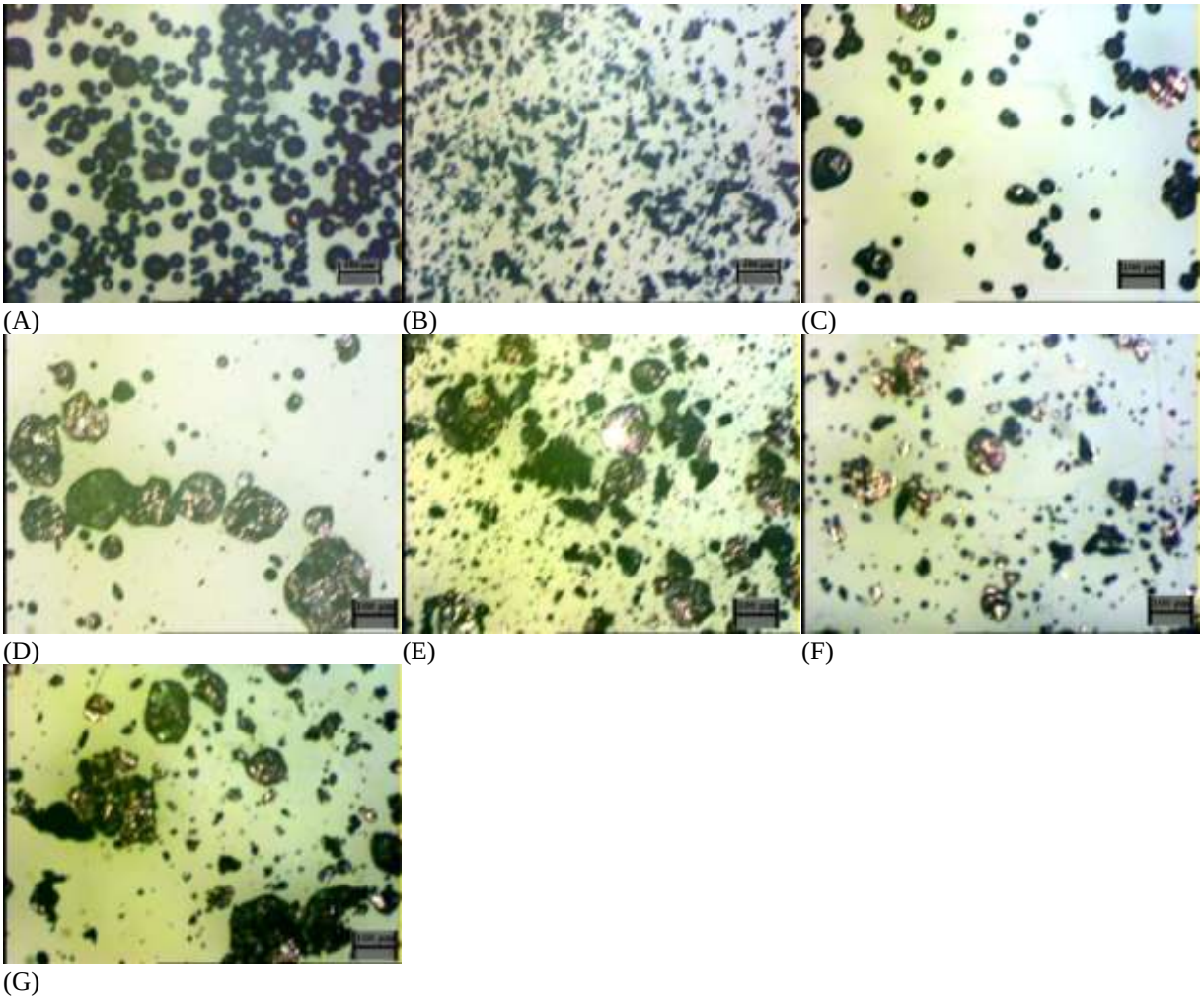

Fig. 6: Microstructure of copper and graphite powders
A- Copper powder before milling.
B- Graphite powder before milling.
C – G - Copper and graphite mixture after 1,3,5,7 and 9 h milling respectively.

Effect of Milling Time on physical properties of Cu – Graphite Composite :

Figures (7,8,9,10,11,12) demonstrates the effect of milling time on several physical properties of Cu – graphite composite. From these figures it can be observed that on increasing milling period, the bulk density, the apparent density and the thermal conductivity decreases while the true porosity, the apparent porosity and the water absorption increases. This behavior can be attributed to the effect of cold working suffered by the particles i.e the increase in hardness and flow stress with the decrease in ductility which reduces the ability of the powder to both compacting and plastic deformation which in turn will produce a composite with low density and high porosity content. Moreover the refining process will increase the number of pores within the compact and reduce its density. Similar results were found by (Yongping Jin *et al*., 2011; K. Rajkumar *et al*, 2009) and (KekeGan *et al*., 2008) in Cu – graphite and Cu – SiC composites respectively.The decrease in thermal conductivity with increasing the milling period can be attributed to the increases in porosity within the composite which acts as insulators and reduces the metal to metal paths which reduces thermal conductivity.

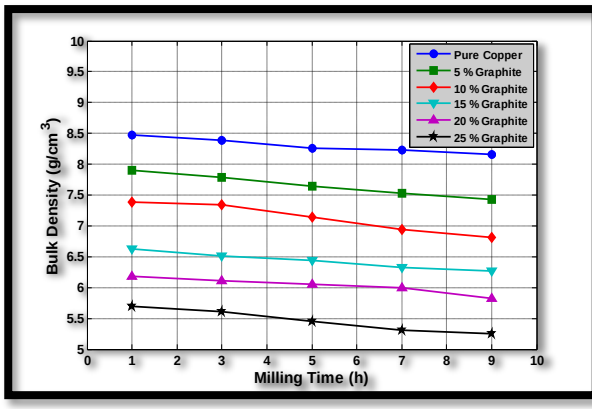

Fig. 7: Relationship between bulk density and milling time.

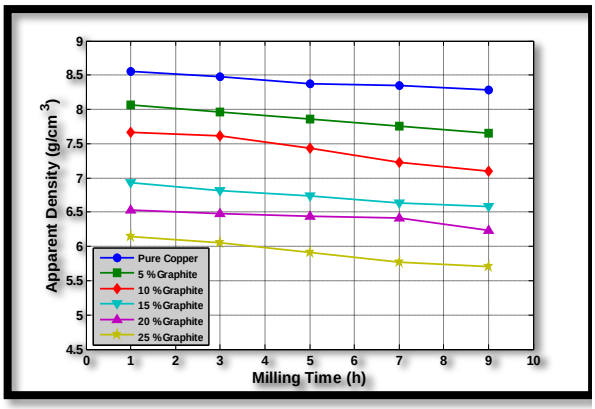

Fig. 8: Relationship between apparent density and milling time.

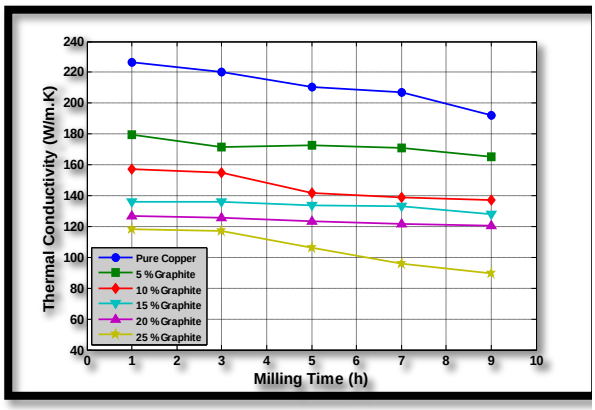

Fig. 9: Relationship between thermal conductivity and milling time.

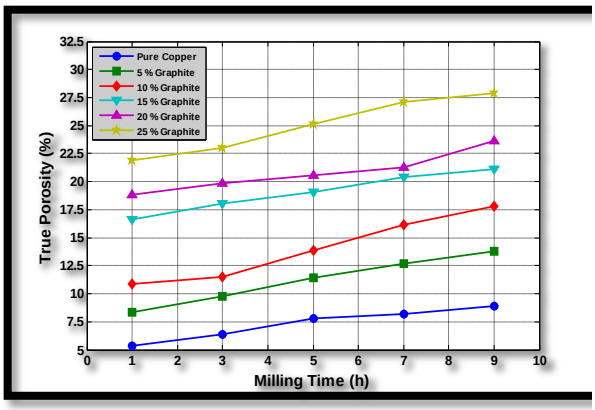

Fig. 10: Relationship between true porosity and milling time.

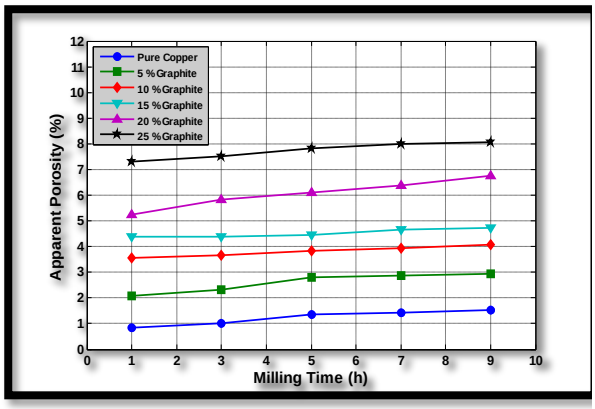

Fig. 11: Relationship between apparent porosity and milling time.

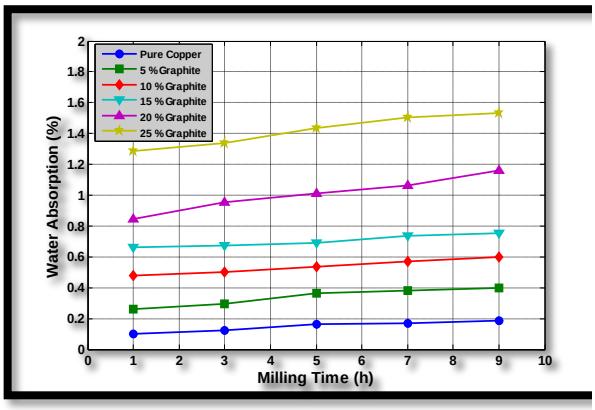

Fig. 12: Relationship between water absorption and milling time.

Effect of graphite content on physical properties of Cu – Graphite Composite:

Figures (13,14,15,16,17,18) shows the effect of graphite content on various physical properties of Cu – graphite composite. In these figures it is observed that increasing the volume fraction of graphite decreases the bulk density, the apparent density and thermal conductivity and increases the true porosity, the apparent porosity and water absorption of the composite. It is also observed that increasing both milling time and graphite content has the same effect on these properties. However the effect of graphite content is much greater than the effect of milling time and graphite volume fraction is the dominant parameter that control the physical properties of the composite rather than the milling time. The effect of graphite content is attributed to the low density (2.26 g/cm^3) of graphite compared with that of copper (8.96 g/cm^3), so any increase in graphite content will decrease both the bulk and apparent densities. Moreover coating of copper particles with graphite will prevent their complete consolidation and reduce both densification and volume shrinkage of the compact during sintering, so a great deal of closed porosity will remain in their positions during sintering. As these effects increases with

increasing the volume fraction of graphite, the bulk and apparent densities decreases and the true and apparent porosities increases which in turn increases the water absorption by increasing the vacant sites within the compact and the available paths through the apparent porosity. Similar results concerning the effect of graphite on density and porosities was observed by (K. Rajkumar *et al*.,2009).

Finally increasing graphite content decreases the thermal conductivity of the composite to great extent. This effect is due to the low thermal conductivity of graphite (23.9 W/m.K) compared to that of copper (398 W/m.K), and to the great increase in true porosity on increasing graphite volume fraction. This porosity acts as insulators within the compact and reduces its thermal conductivity. Moreover the coating effect of graphite on the copper particles reduces the copper to copper contact, a condition that results a lower thermal conductivity. Figures (19,20,21,22,23,24) represents the XRD results of the compact which shows the presence of copper oxide (Cu_2O). Cu_2O increases the insulating effect and reduces the thermal conductivity of the compact due to its low thermal conductivity (75 W/m.K). These results are in good agreement with (C. Vincent *et al*., 2012) and (XU Wei *et al*., 2012).

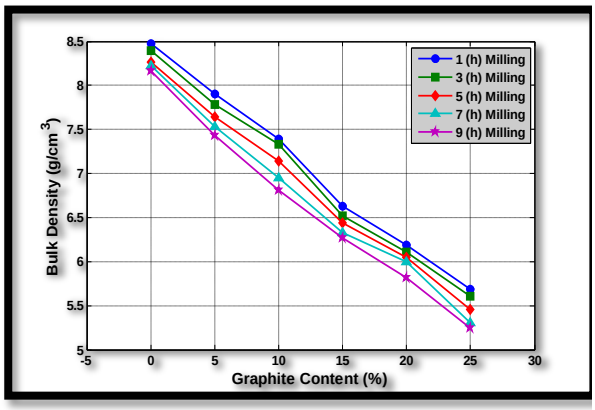

Fig. 13: Relationship between bulk density and graphite content.

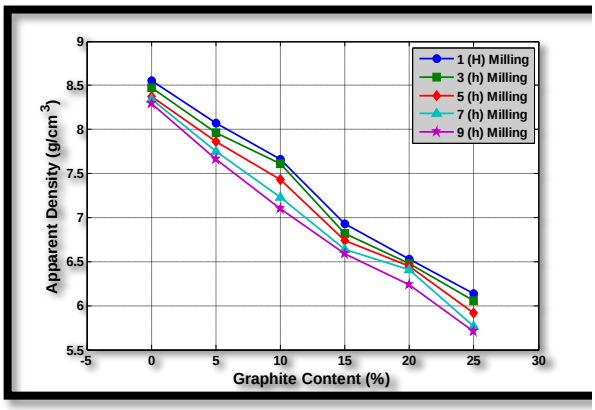

Fig. 14: Relationship between apparent density and graphite content.

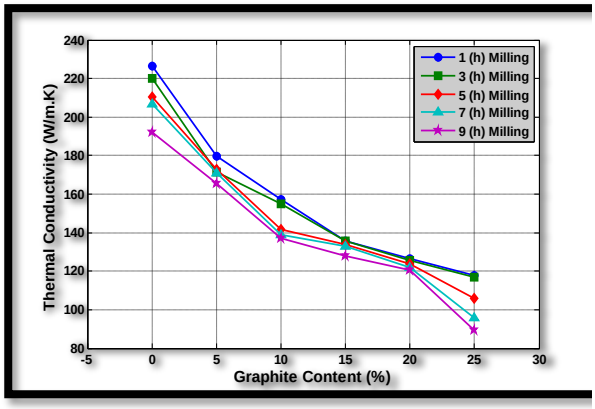

Fig. 15: Relationship between thermal conductivity and graphite content.

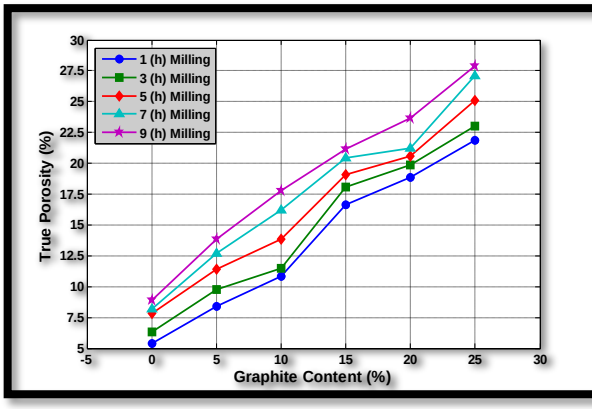

Fig.16: Relationship between true porosity and graphite content.

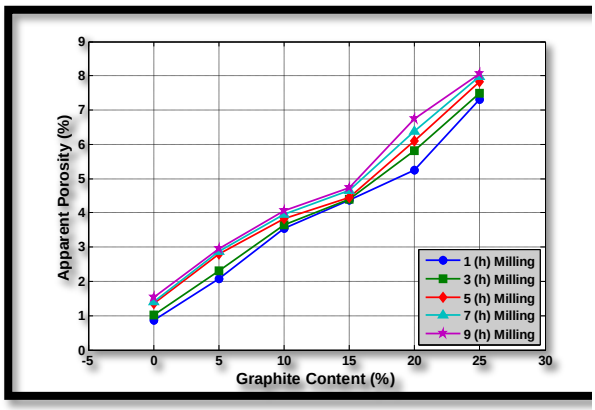

Fig. 17: Relationship between apparent porosity and

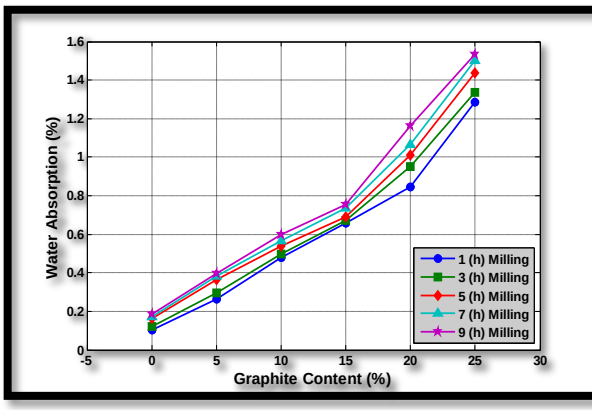

Fig. 18: Relationship between water absorption and

graphite content.

graphite content.

Conclusions:

1. Flattening, refining, and clustering of Cu particles occurs during milling.
2. The bulk density, the apparent density and the thermal conductivity decreases with increasing both milling time and graphite content.
3. The true porosity, the apparent porosity and water absorption increases with increasing both milling time and graphite content.
4. Graphite volume fraction is the dominant parameter that control the studied physical properties rather than milling time.

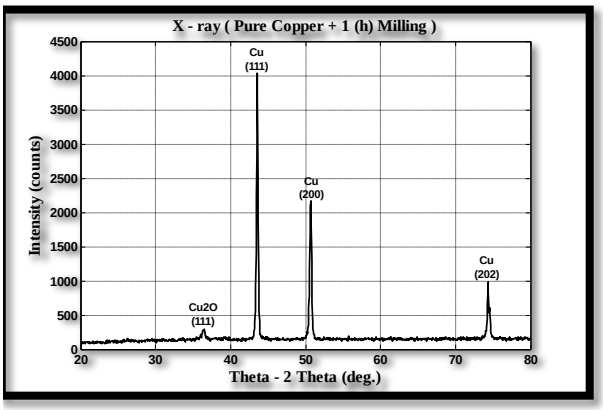

Fig. 19: XRD for pure copper after 1 h milling.

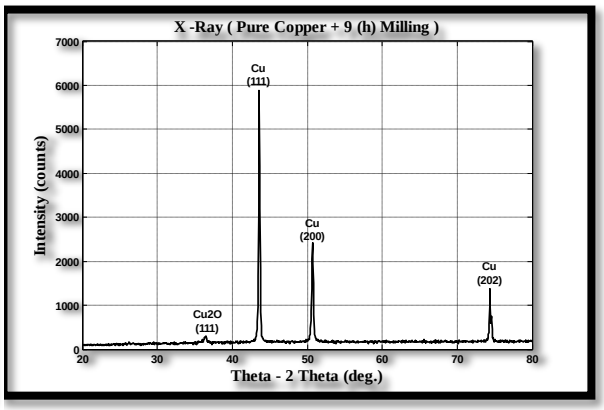

Fig. 20: XRD for pure copper after 9 h milling.

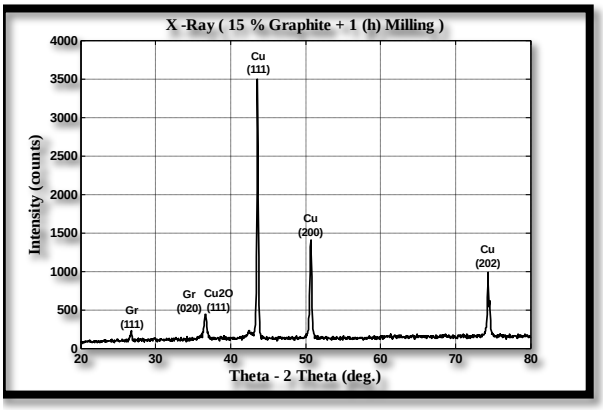

Fig. 21: XRD for copper – 15% graphite composite after 1 h milling.

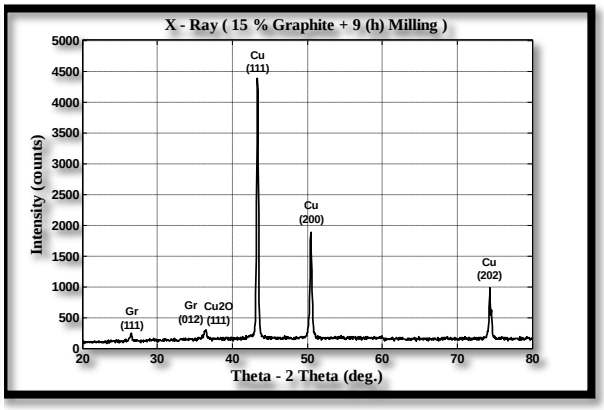

Fig. 22: XRD for copper – 15% graphite composite after 9 h milling.

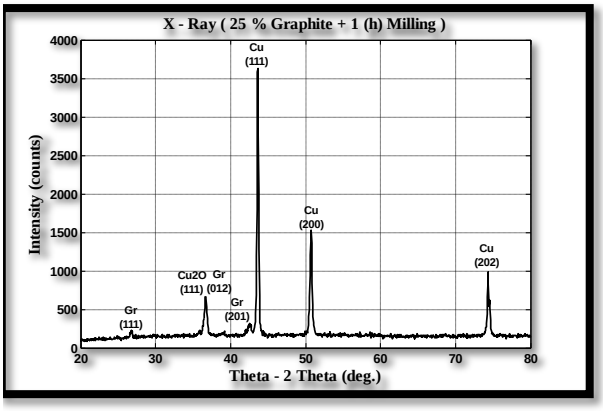

Fig. 23: XRD for copper – 25% graphite composite after 1 h milling.

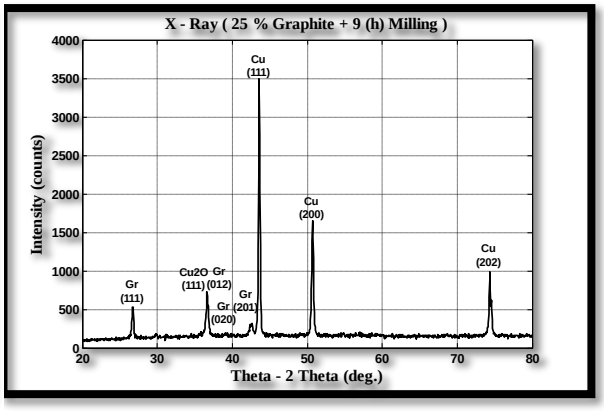

Fig. 24: XRD for copper – 25% graphite composite after 9 h milling.

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