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# The Influence of the Graphite Content and Milling Time on the Hardness, Compressive Strength and Wear Volume of Copper - Graphite Composites Prepared Via Powder Metallurgy

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mechanical properties
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## A B S T R A C T

Copper – graphite composites are widely used in sliding bearings and brushes due to their excellent thermal and electrical conductivities and high wear resistance. The aim of this research is to study the Influence of the graphite content and milling time on hardness, compressive strength, wear volume and friction coefficient of copper - graphite composites that prepared via powder metallurgy. A powder mixture containing 0,5,10,15,20 and 25 vol% graphite was milled 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 ℃ for one hour. It was found through this work that increasing milling time results an appreciate increase in hardness and radial compressive strength. Slight reduction in wear volume and slight increase in the coefficient of friction for all compositions except that for pure copper in which a considerable increase in wear volume and decrease in the coefficient of friction was observed. On the other hand, increasing the graphite volume fraction increases the composite hardness, to reach an optimum value, and decreases the radial compressive strength. A great decrease in both wear volume and coefficient of friction was observed due to increasing the graphite content up to 25 vol%. Finally, a graphite, cast iron chips and fireclay sintering configuration was found to be an effective procedure which minimizes oxidation to levels comparative with those observed previously by sintering in argon or hydrogen atmospheres.



## A R T I C L E  I N F O

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## تأثير محتوى الكرافيت وزمن الطحن على الصلادة، مقاومة الانضغاط، حجم البلى لمتراكبات النحاس – كرافيت المحضرة بواسطة ميتالورجيا المساحيق

### الخلاصة

تستخدم متراكبات النحاس – كرافيت بصورة واسعة في المحامل الانزلاقية والفرش الكهربائية بسبب موصليتها الحرارية والكهربائية الممتازة ومقاومتها العالية للبلى. يهدف البحث إلى دراسة تأثير محتوى الكرافيت وزمن الطحن على الصلادة، مقاومة الانضغاط، حجم البلى، معامل الاحتكاك لمتراكبات النحاس – كرافيت المحضرة بواسطة ميتالورجيا المساحيق. تم طحن خليط المساحيق الذي يحتوي على (0، 5، 10، 15، 20، 25) % نسبة حجمية من الكرافيت لمدة (1، 3، 5، 7، 9) ساعة. بعدها كُبس الخليط المطحون على البارد بضغط 700 ميكاباسكال لمدة 30 ثانية ومن ثم لُبد بدرجة حرارة 900 درجة مئوية لمدة ساعة واحدة. لقد وجد من خلال هذا العمل بأن زيادة زمن الطحن يؤدي إلى زيادة ملحوظة في الصلادة ومقاومة الانضغاط القطرية وكذلك يؤدي إلى انخفاض طفيف في حجم البلى وارتفاع طفيف في معامل الاحتكاك لجميع النسب ما عدا النحاس النقي إذ يزداد حجم البلى وينخفض معامل الاحتكاك. ومن جانب أخر فأن زيادة الكسر الحجمي للكرافيت يسبب زيادة صلادة المتراكبات حتى تصل للقيمة المثلى، بينما تنخفض مقاومة الانضغاط القطرية. وقد لوحظ انخفاض كبير في كل من حجم البلى ومعامل الاحتكاك بزيادة محتوى الكرافيت حتى يصل إلى 25%. أخيرا فأن الكرافيت، نحاتة حديد الزهر والطين الحراري المستخدمة في عملية التلبيد قد أعطت فعالية في خفض الأكسدة إلى مستويات مقاربة لعمليات التلبيد في جو من الآركون أو الهيدروجين.

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## 1. INTRODUCTION

Powder metallurgy is a manufacturing process which is used to produce a metal parts with a uniform and fine microstructure. The conventional process includes compacting the powder mixture into the desired shape and then sintering the produced compact at elevated temperatures to cause bonding of the particles into a hard, rigid mass. The method makes it easy to combine different types of materials together in order to get a unique property combination. Powder metallurgy techniques have several advantages over wrought or cast techniques in producing composite materials, porous materials, refractory metals, special high duty alloys and the most important benefit is lower cost. The purity of the elements can be controlled very accurately, so material with any required chemical composition can be achieved [1-4].

Powder metallurgy technology is widely used in the production of copper based composites which are found widespread applications, such as electronic packaging, electrical contacts and of the electrodes resistance welding due to their high electrical and thermal conductivity, good corrosion resistance, good tribological properties and high melting point. Graphite is one of the most important additives to the copper matrix composites due to its excellent lubrication effect. Consequently, the addition of graphite is very effective in decreasing the friction coefficient and wear rate of these composites by forming a graphite-rich transfer layer on the counterpart surface due to its lamellar structure. However, solubility of graphite in copper does not exceed 0.02% even at very high temperatures. Therefore, graphite wettability by copper is a real problem. Moreover, graphite has low density as compared with copper. These two reasons excluded casting and made powder metallurgy the most suitable technique used to produce such composites [5-12].

Copper–graphite composites are characterized by their high thermal and electrical conductivity and excellent solid lubrication properties for solid contacts and sliding conditions which arise from a synergetic composite effect between copper and graphite. These composites are widely used in manufacturing sliding components, such as electrical brushes and bearings. For electrical applications copper-graphite composites with low graphite content are used for slip rings, switches, relays, connectors, plugs and low voltage d.c. machines with very high current densities. While those with high graphite content are used with lower current densities and better cooling conditions. It has been reported that the addition of solid lubricant particles into a metal matrix improves not only the anti-friction properties, but also wear and friction properties [13-20].

Moustafa et al. [14], studied the friction and wear behavior of copper–graphite composites with uncoated and Cu-coated graphite powder. They found that coating graphite powder with copper has little effect on wear rate of the produced composites. However, the transition loads of Cu-coated graphite composites are much higher than those of the uncoated graphite composites, for the same graphite content. They also found that coated graphite composites have the lowest friction coefficient values, followed by uncoated graphite composites and pure copper compacts respectively.

Kovacik et al. [6] have studied the effect of graphite content on friction coefficient and wear rate of Cu–graphite composites. They found that both coefficient of friction and wear rate decreases with increasing graphite content till a critical value after which the coefficient of friction becomes independent on the percentage of graphite.

Samal [19], has investigated the effect of compacting pressure, conventional sintering parameters and milling time on microstructure and hardness of copper– graphite composites that prepared using powder metallurgy route. He found that the optimum pressure, the optimum sintering temperature and time are 700 MPa, 900 ℃, and 1h respectively. On the other hand, he found that milling of initial composite powder mixture results in a very fine and homogeneously distributed reinforcement particles throughout the matrix. He also found that increasing of milling time up to 2 hours results in a corresponding increase in hardness of the sintered composite.

The aim of this research is to investigate the influence of the graphite content and milling time on hardness, compressive strength and wear volume of copper- graphite composites that are fabricated via powder metallurgy technique.

## 2. EXPERIMENTAL PROCEDURE

### *2.1. Sample Preparation*

Copper – graphite composites were prepared from copper powder with particle size ≤ 63 µm and 99.7% purity and graphite powder with particle size ≤ 63 µm and 99.8% purity. The copper powder has spherical shape particles as shown in Fig. 1 while graphite particles are of irregular shape as shown in Fig. 2.

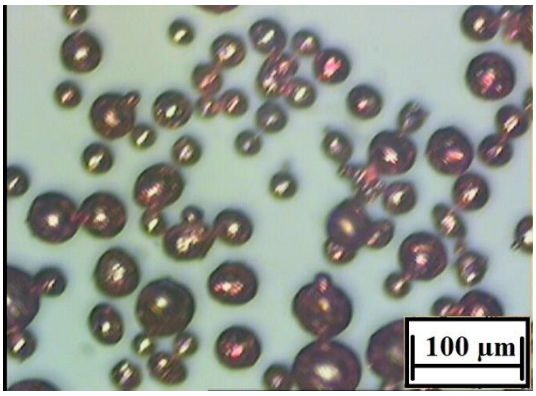


**Fig. 1**. Optical micrograph of spherical copper particles.

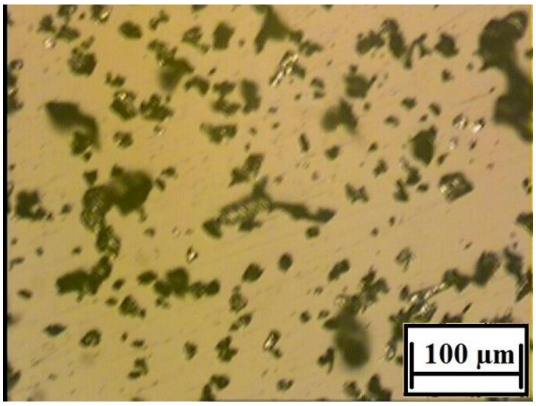


**Fig. 2**. Optical micrograph of irregular shaped graphite particles.

The volume fraction of graphite was 0,5,10,15,20 and 25%. Graphite was dried at 200°C for two hours to get rid of moisture and other volatile substances. The powders were milled in a high energy ball milling machine. Ball size, balls to powder weight ratio, milling speed and milling time were 10 mm, 5:1, 500rpm and (1,3,5,7,9) hours respectively. 10mm in diameter and 5 mm in height specimens were prepared from the milled powder mixture by uniaxial cold pressing process using universal testing machine type (HOYTOM), which is Chinese origin. Pressing was continued for 30 seconds at 700M Pa. Cold pressed samples were sintered at 900 ℃ for 1 hour followed by furnace cooling. To prevent oxidation, the samples were placed in a ceramic container with a multilayer graphite powder and gray cast iron chip with fire clay top layer as shown in Fig. 3.

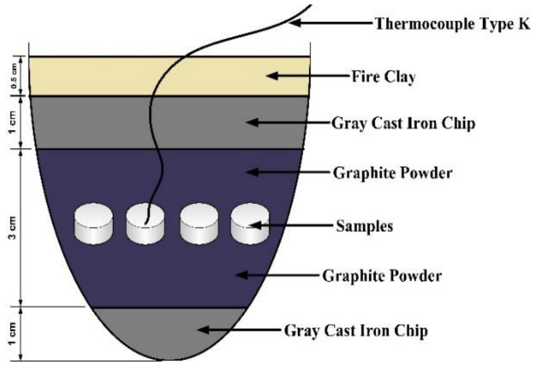


**Fig. 3**. Arrangement of samples into the ceramic sintering container.

Type K thermocouple was placed inside the sintering container just adjacent to the specimens to monitor and control the sintering temperature. This configuration is effective in preventing oxidation as shown in XRD pattern (Fig. 4) in which very low levels of oxidation was observed; where similar $Cu_2O$ contents are observed even with sintering in argon and hydrogen atmospheres in the previous studies [14,19].

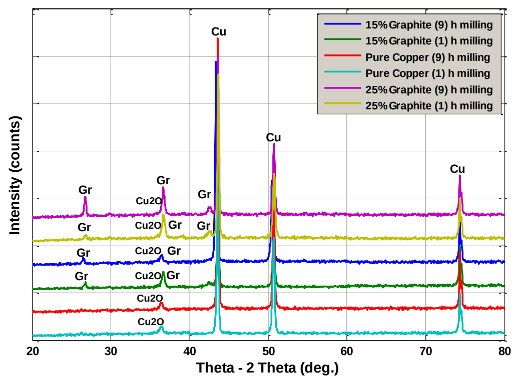


**Fig. 4**. XRD pattern for specimens at different milling time and graphite content.

### *2.2. Friction and Wear Tests*

Friction and wear tests were performed using a pin-on-disc tribometer type DUCOM's Wear and Friction Monitor ED – 201. Copper – graphite composite specimens with 10 mm diameter and 5 mm height represents the cylindrical pins that are rubbed against a hardened steel disc (SAE 1045) with 100 mm diameter, 8mm thickness and 62 HRC surface hardness. The specimens were ground with 1000 grid size SiC emery paper. The ground samples were cleaned with acetone prior and after the wear test. The load, the sliding time and the disc rotational speed were kept constants at 20 N, 30 min and 470 rpm respectively. All tests were performed at laboratory temperature of 20±2 °C. Wear depth and friction force were continuously monitored by the linear variable differential transformer (LVDT) probes that were incorporated in the wear tester. The value of friction coefficient was calculated using the following formula [21,22]:

$$\mu = F / N \qquad (1)$$

where μ is the friction coefficient, *F* is the friction force (N) and *N* is the applied normal load (N).

### *2.3. Hardness Test*

Brinell hardness number was measured using Proceq Equotip 2 machine. Each recorded value represents the five measurements at different regions of the test specimen surface.

### *2.4. Compressive Strength Test*

The radial compressive strength was conducted as shown in Fig. 5 using the universal testing machine type (HOYTOM), which Chinese origin. Compressive strength was calculated from the following equation:

$$\sigma = 2F / \pi dh \qquad (2)$$

where $\sigma$ is the compressive strength (MPa), *F* is the applied force (N), d is the diameter of specimen (mm) and *h* is the thickness of the specimen (mm).

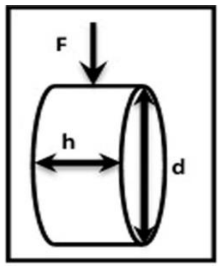


**Fig. 5.** Radial compressive strength specimen.

## 3. RESULTS AND DISCUSSION

### *3.1. Effect of Milling Time on Hardness and Compressive Strength of Cu – Graphite Composites*

Figs. 6 and 7 show the effect of milling period on both hardness and radial compressive strength. These two figures reveal that increasing milling time results in an appreciate increase in both hardness and compressive strength. This effect can be attributed to the following factors:

1- Particle refining during milling which increases the surface area by producing new surfaces of both copper and graphite particles. So, the contact area between the particles increases giving rise to the solid state diffusion which results in better consolidation on sintering.

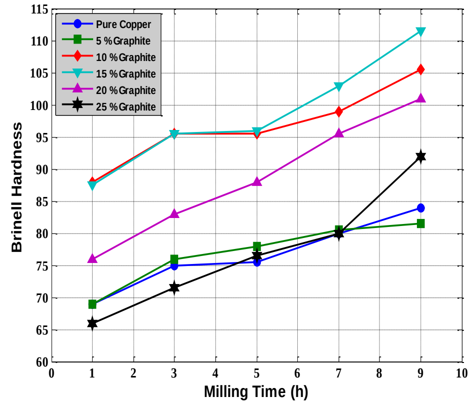


**Fig. 6.** Relationship between Brinell Hardness and milling time.

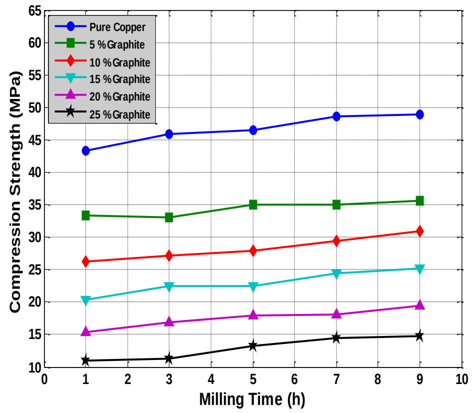


**Fig. 7.** Relationship between compressive strength and milling time.

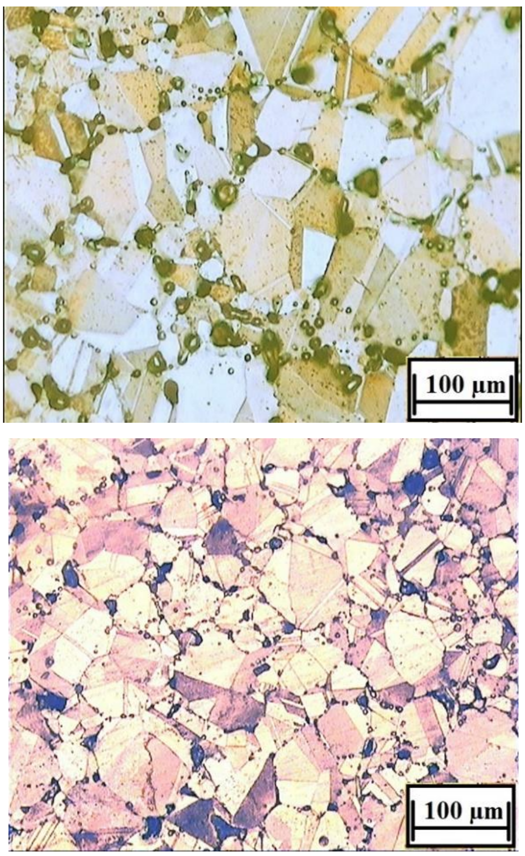


**Fig. 8.** Microstructure of the sintering sample of pure copper at (1) and (9) hours milling time.

2- Increasing milling time results in better graphite distribution which gives better bond between copper and graphite particles. This result is in good agreement with Ref. [23].

3- The longer of the milling time is, the greater of the cold working degree of copper particles and the finer are the recrystallized grain size on sintering as shown in Fig. 8 which reveals the finer recrystallized structure on milling time for 9h as compared with milling for 1h. This fine grained structure contributes in hardness and compressive strength increase through increasing the grain boundary area.

### *3.2. The Effect of Graphite Volume Fraction on Hardness and Compressive Strength of Cu – Graphite Composites*

Figs. 9 and 10 represent the relationship between volume fraction of graphite and both brinell hardness and compressive strength. Fig 9 shows that adding graphite up to 5% has a little effect on the hardness, while increasing graphite volume fraction between (5 to 10%) results in a rapid increase in the hardness for all the milling periods. On increasing graphite content to 15%, the hardness goes to a higher values for milling periods of 7 and 9 hours and remains constant for periods 1, 3 and 5 hours. Any further increase in the graphite content beyond 15% results in rapid decrease in the composite hardness for all the milling periods. Similar results were found previously by Samal [19], Rajkumar et al. [24] and Wei et al. [25] who found that the maximum hardness was achieved with (10 vol%) graphite. This abnormal behavior i.e., increasing the composite hardness with increasing the graphite content up to 10% in the previous studies and up to 15% in the present study needs more investigation since it is well known and well accepted that increasing the content of the soft graphite particles should result in a decrease of the copper–graphite composite hardness as is found by Moustafa et al. [14], Dewidar et al. [26] and Mutter [27].

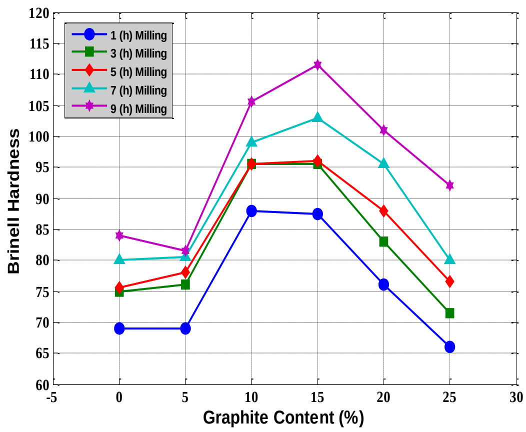


**Fig. 9.** Relationship between Brinell hardness and graphite content.

Fig. 10 shows that the compressive strength decreasing drastically with increasing the volume fraction of graphite.

This behavior can be attributed to:

1- The coating effect of the graphite particles which prevent a perfect consolidation between adjacent copper particles on sintering.

2- The decrease in density and increase in true porosity of copper – graphite composite as was found by Mahdi et al. [28], Samal [19] and Samal et al. [23].

The increase of the composite brittleness with increasing the graphite content.

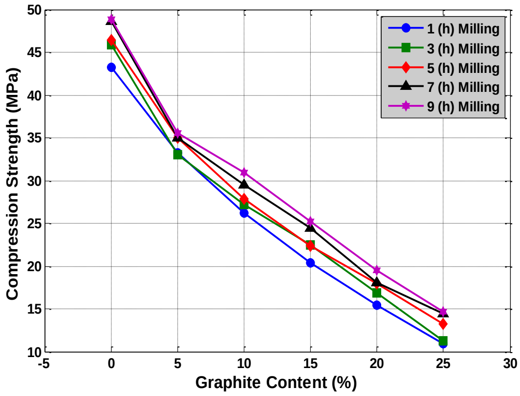


**Fig. 10.** Relationship between the compressive strength and the graphite content.

### *3.3. The Effect of milling Time on Wear Volume and Coefficient of Friction*

Figs. 11 and 12 reveal the effect of milling period on wear volume and coefficient of friction of copper–graphite composites. It is obvious from Fig. 11 that wear volume decreases continuously with the increase of the milling time except for pure copper in which the wear volume increases with increasing the milling time.

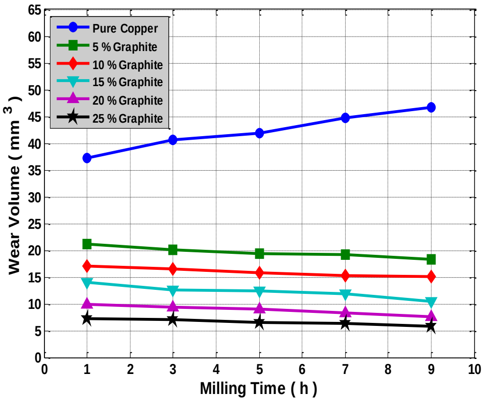


**Fig. 11.** Relationship between the wear volume and the milling time.

On the other hand, Fig. 12 shows that the milling time has a little effect on the coefficient of friction where a slight increase was observed in the friction coefficient for copper – graphite composites and a slight decrease was observed for pure copper.

The behavior of the pure copper can be attributed to a considerable decrease in the bulk density and the great increase in the true porosity with increasing of the milling period which weakens the bond between the adjacent copper particles and make it easy to strip them out on their sliding motion against the steel disc. Moreover, the absence of the lubricant film increases the opportunity of stripping the particles to stick on to the steel disc and acts as stress concentrators, a condition which accelerates the continuous removal of copper particles. While the decrease in wear volume of the copper – graphite composites with increasing of the milling time can be attributed to a corresponding increase in the hardness with increase of the milling period as was shown in Fig. 6. This attribution is assisted by Moustafa et al. [14] and Yang et al. [8] who formulated the relationship between the hardness and wear volume as follows:

$$W = K\left(N.S - C.H\right) \quad (3)$$

where $W$ is wear volume loss, $K$ is the wear constant, $N$ is the applied normal load, $S$ is the sliding distance, $C$ is geometrical factor that depends on the microstructure and $H$ is hardness.

Fig. 12 shows that increasing of the milling time produces a slight increase in the friction coefficient due to the increase in the bonding force between the copper and the graphite particles. Which in turns prevents squeezing out graphite and reduces the thickness of the graphite lubrication film between the composite specimen and the disc or breaks. These effects lead to an increase in the coefficient of friction as was found by a previous works [8,11].

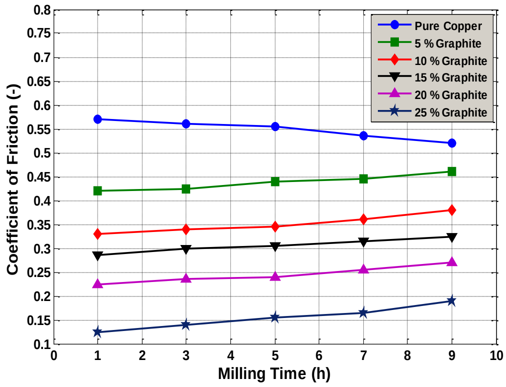


**Fig. 12.** Relationship between the coefficient of friction and the milling time.

### *3.4. The Effect of Graphite Volume Fraction on Wear Volume and Coefficient of Friction*

Figs. 13 and 14 show the effect of graphite content on the wear volume and coefficient of friction of copper – graphite composites. It can be observed that the wear volume decreases drastically due to increasing the graphite content up to 5% for all milling periods. Afterward wear volume continues decreasing but at a lower rate. This behavior is attributed to the formation of a graphite film between the sliding surfaces of the composite pin and the steel disc. This graphite film acts as a solid lubricant and changes the nature of contact from metal to metal contact to a contact condition in which the two surfaces are separated from each other by the graphite film. The presence of the graphite film reduces the shear stress and improves the tribological properties of the sliding surfaces which lead to a great reduction in wear volume. Increasing the graphite volume fraction in the composite produces a dense, thick and continuous graphite film due to the weak bond between copper and graphite within the composite which is limited to a mechanical bond since the solubility of carbon in copper does not exceed 0.02atm%, a condition which on sliding squeezes the graphite out to the surface [6]. Returning to Figs. 11 and 13, it is obviously seen that the dominant factor in determining the wear volume is the graphite content rather than the milling time. The changes in the graphite film that accompanied the increase in the graphite content i.e., dense, thick and continuous film leads to a respective decrease in the coefficient of friction as shown in Fig. 14.

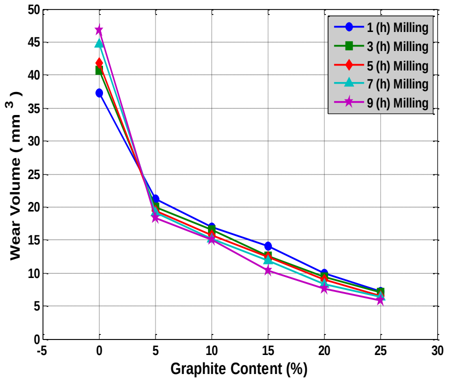


**Fig. 13.** Relationship between the wear volume and the graphite content.

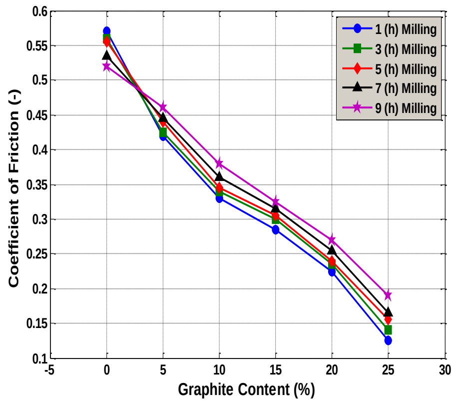


**Fig. 14.** Relationship between the coefficient of friction and the graphite content

## 4. CONCLUSIONS

Throughout this work the following conclusions are reached:

1. Graphite, gray cast iron chips and fireclay sintering configuration minimizes oxidation effectively.
2. Increasing the milling period is accompanied by appreciate increase in the hardness and radial compressive strength.
3. A slight reduction in the wear volume and a slight increase in the coefficient of friction was observed due to increasing the milling period.
4. A great decrease in the wear volume, coefficient of friction and compressive strength were resulted due to increasing the graphite content. On the other hand, optimum graphite content, depending on the milling period, which was found to it gives the maximum hardness.
5. The graphite content is the dominant factor rather than the milling time which controls copper – graphite composite properties.

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