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IMPACT OF PROCESS CONTROLLING AGENT ON THE MICROSTRUCTURE, AND WEAR RESISTANCE OF COPPER /GRAPHENE NANOCOMPOSITE

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ABSTRACT

Copper-graphene nano composite is prepared with 0.25,0.50,0.75,1.00,1.25 and 1.50 wt.% graphene nano sheets. Powder metallurgy technique is used for the preparation process. In which copper powder is mechanically milled with nano graphene sheet by 10: 1 ball to powder ratio, and 400 rpm for 12 hr. milling time. The mixtures are compacted by a uniaxial press under 700 Mpa pressure. The compacted samples are sintered under controlled atmosphere at 950 oC for 1.5 hrs. A comparison between methanol & hexane as a process controlling agent is established. In which Cu-GNSs are mixed with methanol or hexane by 10% to study their effects on the tribological properties of Cu-GNSs composite. Their effects on the microstructure & tribological properties of the prepared Cu/Graphene nanocomposites were studied. All results indicated that hexane samples have the more homogeneous microstructure, low porosity, higher wear resistance & coefficient of friction than those of methanol samples. Also, the density is decreased by increasing graphene percent for both groups. For methanol group 0.25wt. recorded the lowest wear rate. and good microstructure while for hexane group 1.00 wt. percentage graphene is the best one.

KEYWORDS: Copper composites, Graphene nano sheets, Powder metallurgy, Mechanical alloying, Process Controlling agent, Microstructure, Wear Resistance.

1. INTRODUCTION

Copper and its alloys and composites are widely used as a structural material in engineering applications owing to their excellent thermal and electrical conductivities and chemical stability. However, they exhibit relatively poor mechanical properties, especially at elevated temperature and high coefficient of thermal expansion (1) that greatly limits their uses. Since the rapid developments in machinery, electronic, transport and other industries highly demand for Cu and Cu alloys with both excellent conductivity and good mechanical properties, the enhancement of their mechanical performance is increasingly required. The most effective strategy to achieve superior strength is the introduction of secondary phases in Cu and its alloys to fabricate Cu matrix composites.[2] But unfortunately Cu has a high coefficient of the expansion (CTE) and low strength. So, manufacturing Cu composite reinforced with a low (CTE) material with high strength, produces a material suitable for either mechanical or electronic application.

Graphene, has attracted a significant attention as a nanofiller due to its exceptional electrical 105×10^4 cm²/Vs, thermal (5×10^3 W/mK), and mechanical (1 TPa Young's modulus and 130 GPa tensile strength) properties. It is a single layer of covalently bonded sp²-hybridised carbon atoms, arranged in a two-dimensional, hexagonal lattice. Graphene surface can be easily contaminated by airborne hydrocarbons, as it is exposed to ambient air, masking prevents its wettability with any metal mixed with it (R) Given the challenge to completely suppress contamination, many researches have explored ways to remove contamination from the graphene surface. In order to improve the wettability between GNSs and any metallic surface which decreases the aggregation and the formation of internal voids that have a negative effect of the properties of the produced composite.

The technological properties in processing graphene-reinforced MMCs are more pronounced than in case of polymer-matrix composites. In particular, driven mainly by the strong van der Waals forces between aromatic rings, graphene is difficult to disperse uniformly into a metal matrix since it tends to form agglomerates in order to reduce its surface energy during manufacturing process. In addition, obtaining an effective interfacial bonding is difficult due to the poor affinity of graphene to metals. In which, copper (Cu) does not wet graphene and covalent bonding is not possible as no reactions take place between Cu and graphene, which just leaves weak mechanical adhesion and van der Waals interactions enhancing the mechanical interlocking between the graphene and Cu, which in turn leads to a better load transfer. A final challenge is that graphene can easily become damaged

during the fabrication process. Thus, a key challenge in producing good graphene MMCs is their fabrication, by powder metallurgy technique.

It is difficult to mix GNSs with the metallic copper by casting technique as it has very low density (2.2 g/cc) makes it floats on its surface, so the process control agent of powders and controls the particle size instead of cold welding between the hergubring particles reduces the heat energy produced during the milling process and prevents the sticking of powders with the milling containers. many of processing techniques have been developed to optimize the structure and properties of the manufactured Cu/graphene nanocomposites. Powder metallurgy is a very versatile process for manufacturing of composites with graphene due to its simplicity, flexibility and near-shape capability [2]. Mechanical alloying technique can produce composites with fine microstructures and a better distribution of graphene in the Cu matrix. The composite powders can be prepared by simple mixing techniques including mechanical or magnetic sonication and vortex mixing. However, high-energy ball milling (BM) or mechanical alloying (MA) are also employed.[4] In which the total milling energy can be tailored by varying the ratio of balls milling. to the powder, ball mill design, atmosphere some time, speed and temperature. In certain cases, a process control agent (PCA), such as stearic acid or petroleum ether, is added to the powder mixtures to prevent excessive sticking and agglomeration of Cu powders during the milling process [4]. Ethanol, or Acetone, hinders the agglomerations of graphene into clusters. The organic solvents must be evaporated to obtain dry composite powders before consolidation.[5] Also, these solvents must be expelled from the compacted samples during the sintering process by a low heating rate to give the internal gases to get out slowly without breaking down of the consolidated samples or cases any cracking in the sample. (R)

A few known techniques to clean the surface of graphene include thermal annealing, UV–O₃ exposure, solvent cleaning, and dry cleaning. Thermal annealing is a convenient practice that was already employed to remove poly-methyl methacrylate used in transferring CVD-grown graphene and thermal annealing at 550 °C removes air-borne hydrocarbons from the graphene surface. The water contact angle was reduced to 55° from which it returned back to around 80° upon exposure in air for about one hour. The use of UV/O₃ results in the same effect of removing the hydrocarbons from the graphene, reducing the water contact angle. This technique cannot be employed for a long amount of time since UV radiation is known to damage the graphene surface causing defects that can also lower the contact angle of the graphene and may account for the recently reported UV-induced wetting transition in graphene. plasma was used to remove contamination however, significant damage to the surface was reported. Recently reported a dry-cleaning method which is capable of removing 95% of the contaminations without damaging the graphene surface. In their approach,

single layer graphene was surrounded with activated carbon and thermal annealed at 210 °C. Through high-resolution transmission electron microscopy and Auger electron spectroscopy they were able to show that the graphene was atom-cally clean and retained its structural integrity.[6] The previous studies focused on the modification of properties of the milled powders with the PCA additions. There is no significant study that discusses the effect of PCAs on the precipitation characterization and microstructure- property relation of Cu composites. So, this work aims at studying the effect of different PCAs (Methanol and Hexane) not only on the microstructure but also, on the wear behavior of Cu- GNSs. So, the work aims at preparing a high qualified Cu-graphene composite by a good PCA for different mechanical applications.

2.EXPERMINTAL WORK

Cu with 75 μm ,99.90 % purity supplied from (International Co. for Scientific & Medical) is reinforced with 0.25,.0.50,0.75,1.00,1.25 and 1.50 wt. % graphene nano sheets that has 50 nm particle size 99.95 purity supplied from (Fiber Max Composites company, Greece) High energy ball mill is used in mixing Cu with nano graphene by 400 rpm mixer/mill with a ball-to-powder ratio of 10:1. The milling time was 12 hours for a homogeneous mixing between copper and graphene powders. The mechanical alloying technique is processed using two types of process controlling agents (PCA) which are hexane & methanol. So, 10 % from each one is added separately to the milled mixture to study their effects on the mechanical & microstructure of the produced Cu-graphene nano composites. Also, paraffin wax as a lubricant material is added by 0.5wt % during the compaction process to decrease the friction with the die. The mixing process was performed using a stainless-steel container then, the mixture was dried in an oven for one hour at about 100 oC that allows the wax and to melt Then, the mixture was compressed in a cylinder die made from Cr-Mo alloy steel (DINW302). with 8 mm diameter with 12 mm height. under 700 MPa to attain the compacted specimens. The sintering process was performed in a vacuum furnace at 950 oC for 1.5 hour. by a heating rate adjusted by 3 oC/min up to 250 oC where the temperature was holded for 15 min. in a dewaxing step. Then the heating rate was increased to 950 oC by 4oC /min. and holding for 90 min. then the furnace was cooled. For microstructure examination, the specimens were grinding with 0,220,400,600,800,1000,1200,2000, and 3000 grit SiC paper and then polished with 6-micron diamond paste. Microstructure features digital camera type cannon PC1049 fitted with ZIESS lenses was used. The microstructure of the polished samples was investigated by field emission scanning electron microscope model (FESEM; QUANTA-FEG250), Holland.

The actual density of the sintered composites was calculated according to Archimedes rule, using water as a floating liquid. The sintered specimens were weighed in air and in distilled water and the actual density ($\rho_{\text{(act.)}}$) were determined according to the full owing equation: -

$$\rho_{act.} = W_a / (W_a - W_w)$$

Where W_a and W_w are the weight of the sample in air and water, respectively. The theoretical density ($\rho_{th.}$) for the investigated composite was determined according to the following equation: -

$$\rho_{th.} = (V_M * \rho_M) + (V_R * \rho_R)$$

Theoretical density Where V_M and R_M are the volume fraction and density of the matrix while V_R and ρ_R are those for the reinforcement sample. [7-8]

$$\text{Relative density} = \rho_{act.} / \rho_{th.}$$

The abrasive wear is carried out using no-ring technique under normal loads of 10, 20 and 30 N, at sliding speed of 1.5 m/s and under 150, 300, 450 rpm. during sliding process the abrasive wear of the pin was determined as the weight loss per unit sliding distance. A sensitive electronic balance was used to measure the weight loss.

3. RESULTS AND DISCUSSION

Tow group of samples are prepared, one of them is the Cu-GNSs composites with hexane as a process controlling agent and the other with methanol. This section illustrates and discusses the physical and mechanical properties of the sintered composites.

Microstructure Examination

Fig.1. shows SEM of Graphene nano sheets (a) and Copper particles (b) The Figure shows that graphene has flake sheets with 50 nm particle size while Cu particles are irregular particles with less than 100 nm size.

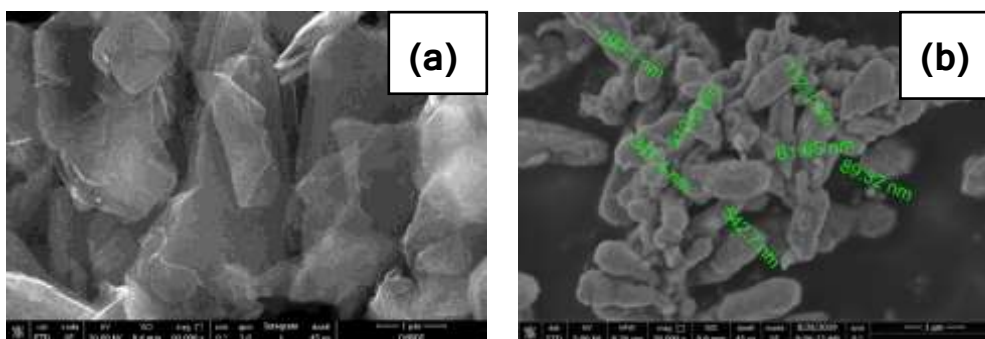


Fig.2 shows the microstructure of Cu-GNSs composites (a, b, c and d) represents the prepared composites by methanol as a process controlling agent, while (e, f, g and h) are those for the hexane ones. Generally, comparing the two groups, one can notice that samples prepared using hexane have a good microstructure and good homogeneity between GNSs and Cu matrix. For all samples there, are three areas, white grey, grey and black. The white grey area represents the Cu matrix while, the grey area represents the GNSs and the black one belongs to the pores. It is clear that in case of hexane samples no pores are detected and GNSs are well distributed all over the Cu matrix with no any aggregations. While the methanol group samples have some porosity. This may be attributed to the hexane nature in which hexane has six carbon atoms with no oxygen, so it is an inert organic solvent with low evaporation temperature. Hexane dissolves all the organic contaminants on the graphene nanosheets surface which decreases the surface energy between Cu and GNSs, so creation of a well bonded van-der walls forces is established. that facilitate the dispersion of it in the Cu matrix with good wettability and no agglomerations. Also, hexane acts as a lubricant that helps in slipping of the particles, so aggregations were decreased While methanol contains oxygen in its structure with short carbon chain, in which some researcher concluded that by increasing the c-chain of the organic solvent. Its efficiency becomes more better [9] Another observation from the microstructure is 1 wt. % Cu-graphene sample for the hexane group has the most homogeneous microstructure and lowest pore percent, while 0.25 wt.% graphene sample for the methanol group is the best one.

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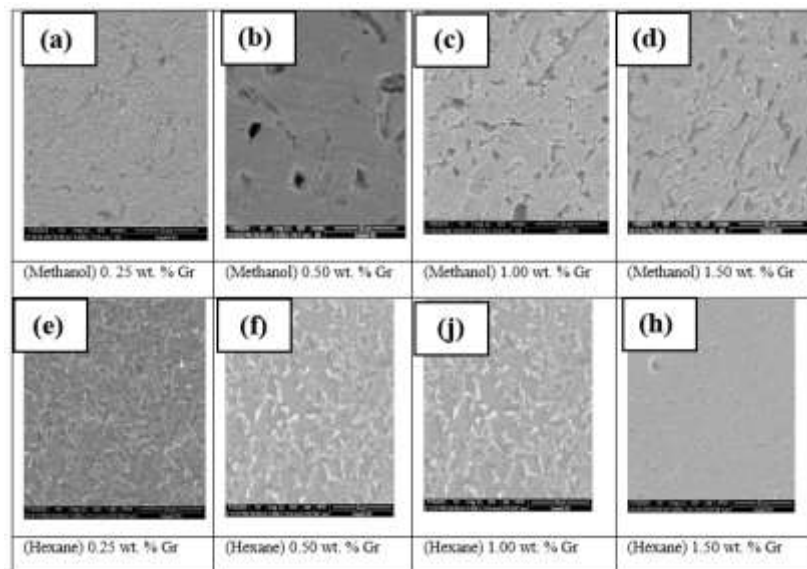


Fig.2. SEM of sintered Cu-GNSs composite (a,b,c and d) methanol group,(e, f, g and h)hexane group.

Fig. 3 (a, b) shows the EDX analysis of Cu 1 wt. % GNSs. methanol and hexane respectively. It is clear that the specimens have perfect homogenous dispersion with a smaller number of Gr agglomerations due to the good mixing process between Cu and GNSs Also using hexane (PCA)in sample (b) reverse sample (a) used methanol (PCA).

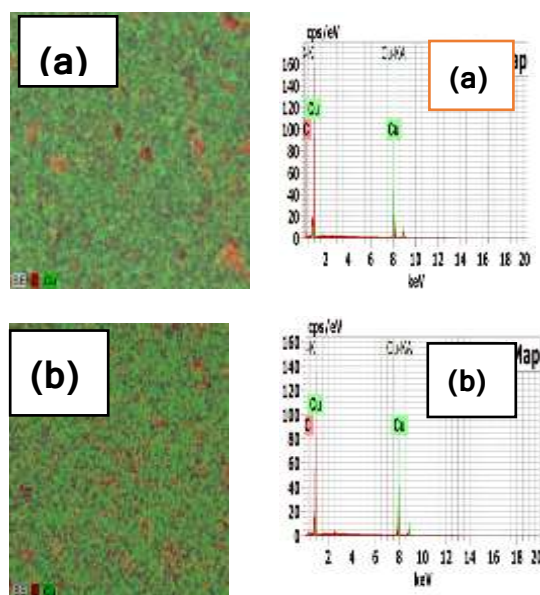


Fig.3. EDX of sintered (a) Cu /1 wt. % GNSs (M), (b) Cu/1wt. % GNSs (H).

Table1.Relative Density measured value.

Sample GNSs %	Hexane group %	Methanol group %
Cu pure	92.39	90.88
Cu +0.25	89.96	85.53
Cu +0.50	88.35	81.23
Cu +0.75	87.88	78.21
Cu +1.00	86.35	76.32
Cu +1.25	84.69	75.83
Cu +1.50	83.20	72.19

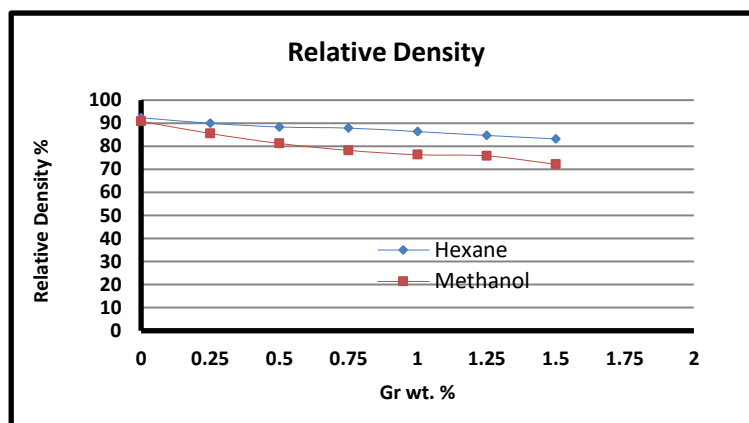


Fig. 4. Relative density versus graphene wt. %for Cu-GNSs (hexane), Cu-GNSs (methanol) composites

Table 1., Fig. 4 shows the effect of GNSs on the relative density of Cu-GNSs nanocomposites prepared by methanol and hexane as a process control agent PCA. The Figure shows two phenomena the first is the decreasing of the density value by increasing the graphene percent for both groups. This is may be attributed to the lower density value of graphene (2.2 g/c.c) than that of Cu (8.96 g/c.c). [9]

The second phenomena is increasing the density value of hexane group samples than those of methanol one. This is due to the hexane nature, in which it facilitates the separation and slipping of graphene sheets from each other's, consequently good dispersion of it in the Cu matrix without a aggregation takes place.[8] so smaller porosity was observed that increases the densification

Wear behavior

The tribological properties are a major function of the service environment, in which the parameters manipulating the wear will differ according to the environment. so, It is very important to study the wear mechanism and wear behavior. The wear rate of all prepared samples is examined under 10,20,30 N applied loads and 150,300,450 rpm (rotation per minute). The weight of samples was measured before and after the test to determine the wear rate.

The prepared samples are classified into three groups the first group is used to examine the wear rate under 10,20,0 by 150 rpm.

Tables (2-3) and Figures (4-5) represent the effect of graphene percent and applied load on the wear rate of Cu-graphene nanosheets at 150 rpm. it is clear that the wear rate is decreased by the addition of 0.25 wt.% graphene from the methanol group samples. This can be explained by the high strength and good distribution of graphene on the Cu matrix. Also, graphene has a low density so, it may be floating on the Cu surface and forms a tribo layer that causes the sliding of the indenter so, the wear rate is decreasing (10). By increasing the graphene percent, The wear rate is increased For higher graphene percentage than 0.25 wt.%, some agglomeration takes place in case of methanol group samples. But for hexane samples the lower wear rate was recorded for 1 wt.% graphene. This may be attributed to the aggregations of the graphene due to the non-wettability problem with Cu (11) but for 1.00 wt. % graphene from group hexane sample for all wear loads, it exhibited the lowest wear rate, which is attributed to the best microstructure and low porosity of this sample. For 1.25-1.50 wt. % graphene samples. The graphene volume fraction is increased and the surface energy with the Cu particles is increased, so the collection of graphene takes place in the Cu matrix. Forming pores and voids which facilitate the elimination of particles from the sample's surface by the sliding load.

Table 2. wear rate at 150 RPM and load (10,20,30) N group Hexane

Composite	PCA (Hexane)		
	10 N g/s 10 ⁻⁶	20 N g/s 10 ⁻⁶	30 N g/s 10 ⁻⁶
Cu pure	4.11	7.10	10.00
Cu +0.25 %Gr	2.88	5.25	8.10
Cu +0.50% Gr	3.00	6.20	8.96
Cu +0.75 %Gr	3.88	6.42	9.65
Cu +1.00 %Gr	2.35	4.62	7.90
Cu +1.25 %Gr	4.90	6.85	9.45
Cu +1.50 %Gr	7.77	9.40	8.51

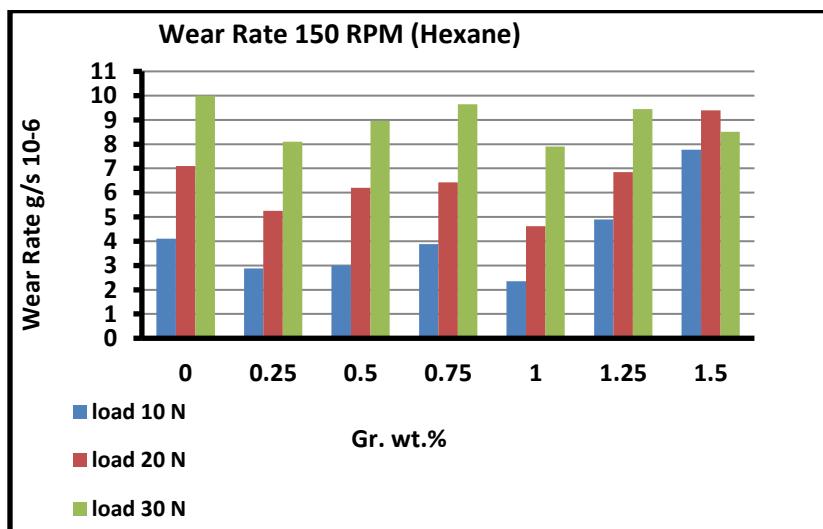


Fig.4. wear rate at 150 rpm and load 10,20,30 N from Cu-GNSs (Hexane)

Table 3. Wear rate at 150 RPM and load (10,20,30) N group Methanol.

Composite	PCA (Methanol)		
	10 N g/s 10 ⁻⁵	20 N g/s 10 ⁻⁵	30 N g/s 10 ⁻⁵
Cu pure	3.32	5.32	7.10
Cu +0.25 %Gr	2.21	4.86	6.13
Cu +0.50% Gr	3.00	5.76	6.96
Cu +0.75 %Gr	3.86	6.11	8.63
Cu +1.00 %Gr	4.21	6.46	7.90
Cu +1.25 %Gr	4.90	7.12	8.45
Cu +1.50 %Gr	6.31	8.40	10.02

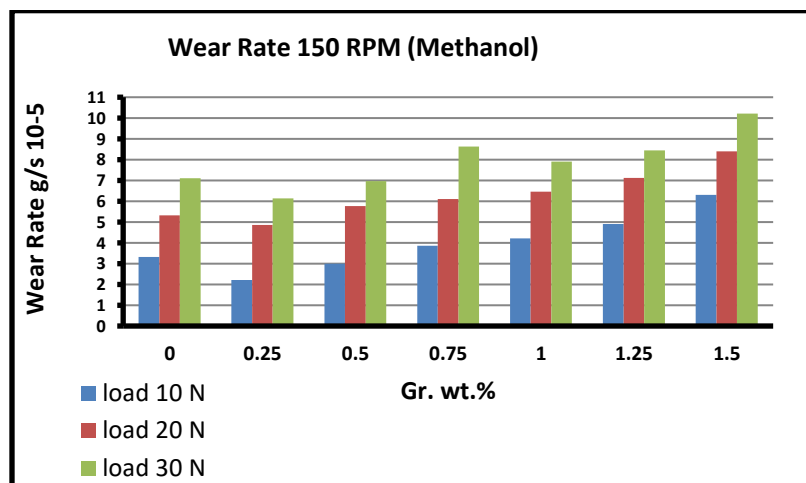


Fig. 5. wear rate at 150 rpm and load 10,20,30 N from Cu-GNSs (methanol)

[2] Wear rate under 300 rpm and 10,20,30 N.

Tables (4-5) and Figures (6-7) show the effect of graphene percent and applied wear load on the wear rate of all the prepared samples under 300 rpm rotation speed. There are phenomenon, the first is the decreasing of the wear rate for hexane group samples than those of methanol one. This is as stated previously due to

the good effect of hexane as a PCA on the reduction of the particle size the exfoliation of the graphene layers more than methanol. This improves the wear resistance. The second phenomenon is the same behavior under 150 rpm, in which 0.25 wt.% sample has the lowest wear rate for methanol group, while 1 wt.% is the best for hexane group sample. The third is the increasing of the wear rate by increasing the applied load, which is a natural phenomenon. By increasing the applied loads from 10 up to 30 N the press effect of the pin increased that gives it a more chance for penetrating the sample surface and wear the sample.

Table 4. wear rate at 300 RPM and load (10,20,30) N group Hexane

Composite	PCA (Hexane)		
	10 N g/s 10 ⁻⁵	20 N g/s 10 ⁻⁵	30 N g/s 10 ⁻⁵
Cu pure	6.3	5.10	7.00
Cu +0.25 %Gr	4.9	4.95	6.10
Cu +0.50% Gr	4.50	4.52	5.96
Cu +0.75 %Gr	4.1	4.39	7.35
Cu +1.00 %Gr	3.9	3.22	5.64
Cu +1.25 %Gr	5.20	5.84	8.45
Cu +1.50 %Gr	7.5	7.41	9.51

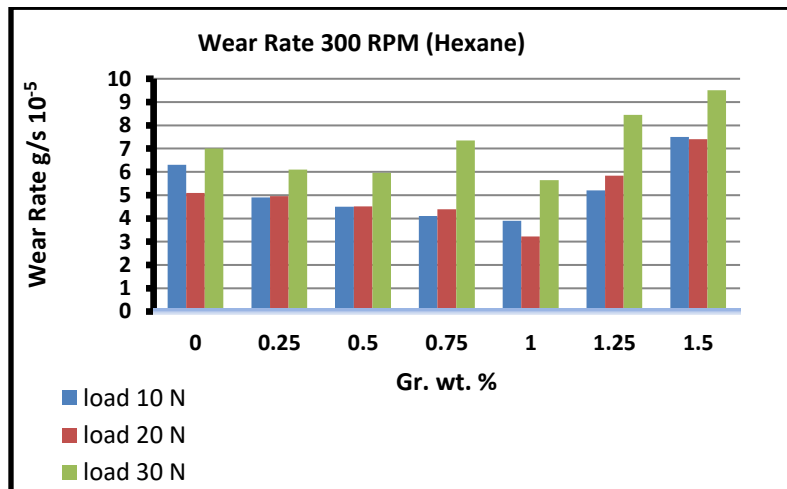


Fig.6.wear rate at 300 rpm and load 10,20,30 N from Cu-GNSs (Hexane)

Table 5. Wear rate at 300 RPM and load (10,20,30) N group Methanol.

Composite	PCA (Methanol)		
	10 N g/s 10 ⁻⁴	20 N g/s 10 ⁻⁴	30 N g/s 10 ⁻⁴
Cu pure	2.85	3.89	4.19
Cu +0.25 %Gr	2.12	3.01	3.71
Cu +0.50% Gr	3.11	4.17	4.98
Cu +0.75 %Gr	4.1	5.85	6.81
Cu +1.00 %Gr	5.92	7.21	7.15
Cu +1.25 %Gr	7.21	8.12	7.45
Cu +1.50 %Gr	6.51	7.41	8.02

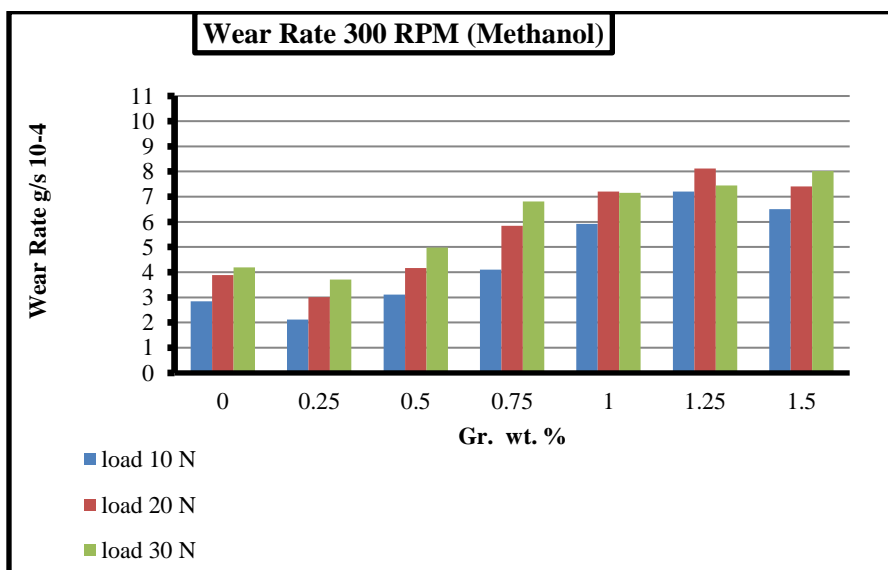


Fig.7. Wear rate at 300 rpm and load 10,20,30 N from Cu-GNSs (Methanol)

[3] Wear rate under 450 rpm and 10,20,30 N.

Tables (6-7) and Figures (8-9) show the effects of graphene and the applied load on the wear rate of the sample under 450 rpm. For the methanol group sample, it is clear that for all applied loads, the wear rate increases by the addition of wt.% graphene then increases gradually by increasing graphene content. but

group hexane lowest wear rate. because some agglomeration takes place that causes pore formation, which decreases the wear resistance.

Table 6. Wear rate at 450 RPM and load (10,20,30) N group Hexane

Composite	PCA (Hexane)		
	10 N g/s 10 ⁻⁴	20 N g/s 10 ⁻⁴	30 N g/s 10 ⁻⁴
Cu pure	3.42	4.1	5.32
Cu +0.25 %Gr	3.00	3.9	4.21
Cu +0.50% Gr	3.37	3.50	4.01
Cu +0.75 %Gr	4.22	4.1	5.31
Cu +1.00 %Gr	2.37	3.3	3.9
Cu +1.25 %Gr	4.90	5.20	5.41
Cu +1.50 %Gr	4.9	7.5	6.97

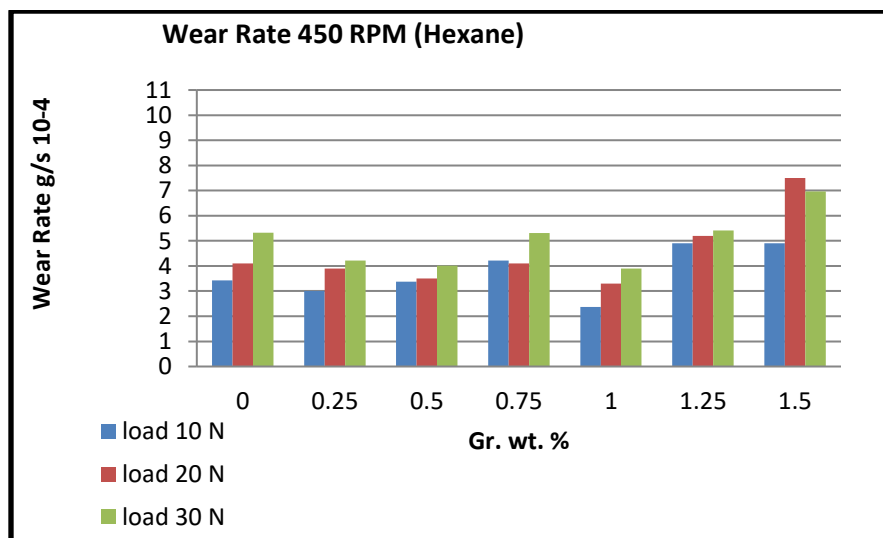


Fig. 8. Wear rate at 450 rpm and load 0,20,30 N from Cu-GNSs (Hexane)

Table7. wear rate at 450 RPM and load (10,20,30) N group Methanol.

Composite	PCA (Methanol)		
	10 N g/s 10^{-4}	20 N g/s 10^{-4}	30 N g/s 10^{-4}
Cu pure	4.41	5.88	6.29
Cu +0.25 %Gr	3.50	3.97	4.91
Cu +0.50% Gr	3.52	4.01	5.32
Cu +0.75 %Gr	4.56	5.12	6.3
Cu +1.00 %Gr	5.59	6.01	6.99
Cu +1.25 %Gr	6.90	7.84	8.64
Cu +1.50 %Gr	8.01	9.11	10.42

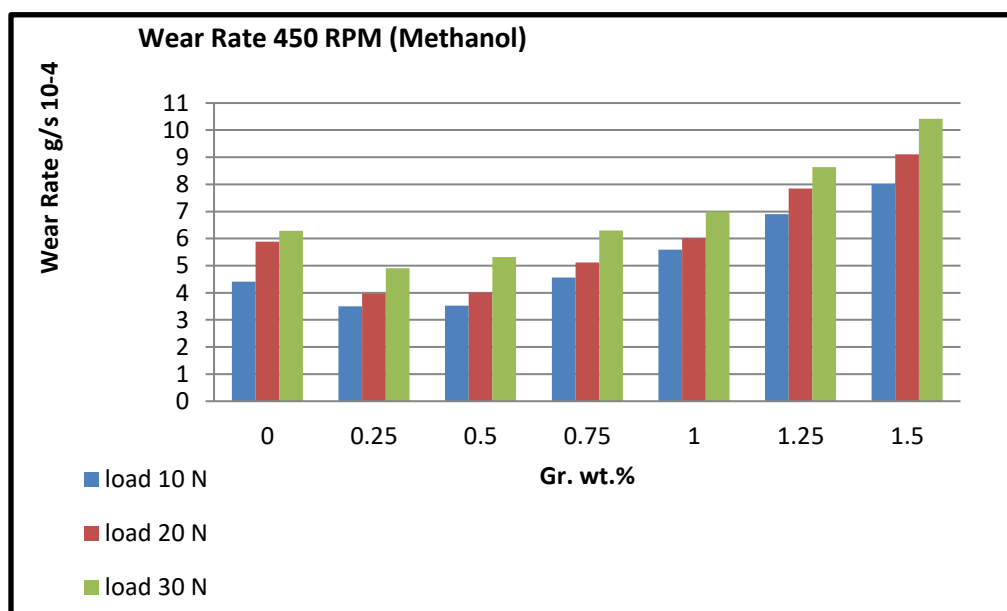


Fig.9.wear rate at 450 rpm and load 10,20,30 N from Cu-GNSs (Methanol)

Tables (2-7) and figures (4-9) show the effect of graphene percent and (PCA) type and wear load on the wear rate of the prepared samples .it is clear that the wear rate increases by increasing the applied load and rpm for all sample, in which the effect of load on the surface of the sample is similar to the indentation, is which as the applied load increases, the depth of the penetration of the indent or increases. The second is the increase of the wear rate methanol sample's group while decreasing that of the hexane one. this may be attributed to the good distribution of graphene nanosheets all over the Cu matrix with no agglomeration

and low pores. In which long-chain hexane compound has the ability to spread between graphene nanosheets

4. CONCLUSIONS

- 1- Cu- GNSs composite were success fully prepared by (PM) technique by 0.25, 0.50, 0.75, 1.00, 1.25, 1.50 GNSs percent.
- 2-Two groups of Sample are prepared depending on the addition of hexane or methanol as a process controlling agent.
- 3- The results showed that hexane group sample have the most homogeneous microstructure than those of methanol.
- 4- The density of hexane group sample are higher than that of methanol one.

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