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Effect of Graphene Nano-Sheets Additions on the Microstructure and Wear Behavior of Copper Matrix Nano-Composite

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Abstract

Six samples of copper matrix composites contain different weight percentages of graphene that are 0, 0.2, 0.4, 0.6, 0.8, and 1 wt. % GNs were fabricated using the electro-less copper deposition followed by the powder metallurgy technique. The sintering process has proceeded in a hydrogen atmosphere furnace. The morphology and microstructure of the as-received powders, as well as the sintered samples, are examined using the scanning electron microscope. The chemical composition of the fabricated composites was evaluated by the EDAX analysis. The effect of GNs content on the hardness, wear rate The microstructure of the composites reveals a good distribution of the (GNs and MoS_2) in the copper matrix. Also, good adhesion between GNs and the (Cu-10MoS_2) matrix was achieved. The 0.4 wt. % GNs composite exhibits the highest hardness and the lowest wear rate.

Keywords

Solid self-lubricant materials;
Hardeness;
Microstructure;
Wear;
Cooper matrix.

1. Introduction

This work is part of a set of themes that aim at developing materials of low friction coefficient and high mechanical resistance in order to open the application fields for selflubricating materials. Dry self-lubricating bearings have been used for decades in household equipments and in office slight equipments (printers, electric shavers, drills, blenders, among others). Usually these materials have a high content of solid lubricant (15 to 35 vol. %) and this results in a high degree of discontinuity of the metallic matrix leading to poor mechanical strength of composite. Thus, these materials cannot be used for many typical mechanical applications where it is necessary higher mechanical and wear resistance of the self-lubricating sintered material. In this direction, the development of dry bulk self-lubricating materials combines a low friction coefficient with high mechanical strength, tuned for each particular application. A solid lubricant can be applied to components of a tribological pair in the form of films/layers deposited or generated on its surface and incorporated into the volume of the material in dispersed particle form in the matrix, generating self-lubricating composite materials. Lubricating composites are used to increase the service life of machinery and equipment where oil and grease cannot be used. For example, metal matrix composites which contain solid lubricants for high temperature applications and vacuum conditions are produced by powder metallurgy.

To obtain a composite which can conciliate a low friction coefficient of and high mechanical strength it is necessary that the composite matrix has a high degree of continuity and the additive (solid lubricant) is not soluble and do not react with the matrix.
Graphite (C) and molybdenum disulfide (MoS2) are the most commonly used solid lubricants. Molybdenum disulfide is attractive due to its extremely low friction coefficient in the presence of different materials, operating rates, temperatures and pressures. However, its performance decreases in the presence of humidity and that is the reason it has been used as solid lubricant to vacuum work and space applications. Graphite has better properties as solid lubricant when in the presence of humidity. Hexagonal boron nitride (h-BN) is a soft, white, lubricant powder with unique features which makes it attractive in place of graphite, molybdenum disulfide and other inorganic solid lubricants. With its superior adhesion property and thermal stability, BN presents an opportunity for the formulation of new solid lubricants for

applications where conventional solid lubricants are inefficient or do not exhibit the expected performance.

Graphene is a typically two-dimensional (2D) layered material. It has many strikingly properties, which are high transparency (97.7% transmittance in the visible spectrum), high thermal conductivity at room temperature ($3x10^3$ W/m K), high electrical conductivity ($\sim10^4$ Ω^{-1} cm^{-1}), high Young's modulus (1.1 TPa) and high specific surface area (2630 m^2/g). All these extraordinary properties made graphene used in various applications, including transparent electrodes, energy storage, solar cells, wearable devices and catalysis [1-5].

Powder metallurgy method has become increasingly interesting for engineering parts manufacturers due to its advantages, which include high productivity, minimum consumption of raw materials, and energy, efficient use of the initial metals, near net shape character and unique capability of porous material production [6].

The main aim of the present research is to fabricate a new self-lubricating materials and studying their tribology properties.

2. Materials and Methods

In this research, molybdenum disulfide powder (MoS_2) and graphene nano-sheets (GNs) are used as reinforcement materials for the copper matrix. The MoS_2 has 1- 0.5µm particle size is supplied by the DOP ORGANİK KİMYA SAN.VE TİC. LTD ŞTİ, company, while the graphene nano-sheets has 2-10 nm thickness is supplied from ACS Material, LLC (Advanced Chemical Supplier). Copper metal matrix is precipitated on the surface of the MoS2 and GNs powders by the electro-less deposition process, from a bath contains 70g/l of copper (II) sulfate pentahydrate as a source of copper metal, 170g/l of potassium sodium tartrate as a complex agent of copper ions, 50g/l of sodium hydroxide, and 200 ml/l of formaldehyde as a reducing agent of copper ions [6, 7]. In order to facilitate the copper deposition on MoS_2, and GNs particles; its surfaces are metalized with 3 wt.% Ag, from a bath, contains 472ml water, 0.944g silver nitrate of 99.97% purity, ammonia 33% (Ph=11), and formaldehyde of 38% concentration (30ml/100ml water).

The produced Cu-MoS_2-GNs nan-composites powders are heated in a tube furnace at 500 ^oC for 1 hr in a hydrogen atmosphere to reduce the copper oxide that formed during the electro-less deposition, into Cu. Also, that leads to an increase in the contact area between the reinforcements and copper matrix, where the insulated oxides have removed. The composites are cold pressed by applying a load of 900 MPa in a die of 12 mm diameter, 10mm sample height.

To consolidate the compacted composites; it is sintered in a reducing atmosphere of pure hydrogen at 1000 ºC for 120 min as shown in figure 1.

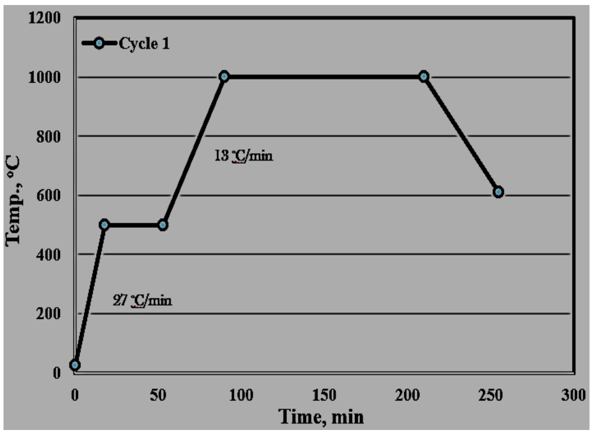

Fig. 1 Heating cycle of the fabricated composites

In order to characterize the microstructure of the fabricated composites, their surfaces are prepared by the grinding, and polishing processes.

To study morphology, and distribution of the different elements of all the consolidated composites as well as the used powders, the scanning electron microscopy (SEM) of the model QUANTA FEG250-EDAX is used. Also, the EDAX analysis and mapping are performed for the consolidated composites. The X-ray diffractometer of the model D8 kristalloflex (Ni-filtered Cu Ka) is used to characterize the Phase compositions and crystal structure of the powder and sintered composites.

The composites macro-hardness is measured along the cross-section surface of the specimens using the Vickers hardness tester of the model 5030 SKV England, at 5 kg_f load and 15 sec holding time. The mean of five readings is calculated and used as the hardness value of each fabricated composite.

A dry adhesive wear test of the composites is carried out by using a pin-on-disc test machine to investigate the tribological properties coefficient of friction and wear rate. The disc was made of steel with a hardness of 65 HRC. 0.7m/s speed, (10, 20 and 30N) load and 5min time were the test conditions.

3. Results

Figure 3 (a and b) shows the morphology of the GNs, and MoS_2 utilized powder materials at high magnification. In addition, images (c and d) for the coated GNs, and MoS_2 with copper by the electro-less coating process. It is obvious that the graphene layers take the shape of the nano-thikness sheets, and the molybdenum has irregular morphology in the form of platelets. It is obvious that the graphene layers have a large surface area and a thin thickness.

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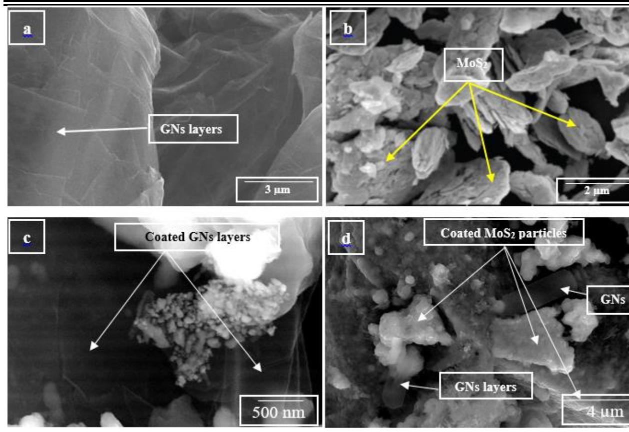

Fig. 2 SEM Micrograph of (a) Graphene nano-sheets (GNs), (b) Molybdenum disulfide (MoS_2), and (c and d) Cu Coated GNs and MoS_2

3.1 Microstructure of Fabricated Composites

The morphologies and microstructures of the sintered samples that contain 0, 0.2, 0.4, 0.6, 0.8, and 1 wt. % GNs respectively at the low heating rate, are shown in figure 3. The composites are showed good sinter-ability. Three phases are observed from the microstructure, which are gray Cu matrix, white gray MoS_2 reinforcement, and the changeable black GNs reinforcement. The microstructures of all the samples that contain GNs and MoS_2 show that the GNs and the MoS_2 particles of the lamellar crystal structure have a plate-like shape. All the composites show high relative densities so that no porosities are detected. It is obvious that both MoS2 and GNs are distributed homogeneously in the Cu matrix. Also, good adhesion between all the constituents of the composites is clear. The good adhesion may be due to the coating process, which increases the contact between the matrix and reinforcement. Also, the heating process of the coating powders, which preceded the compaction process to eliminate the oxides from a deposited layer of copper has a great effect.

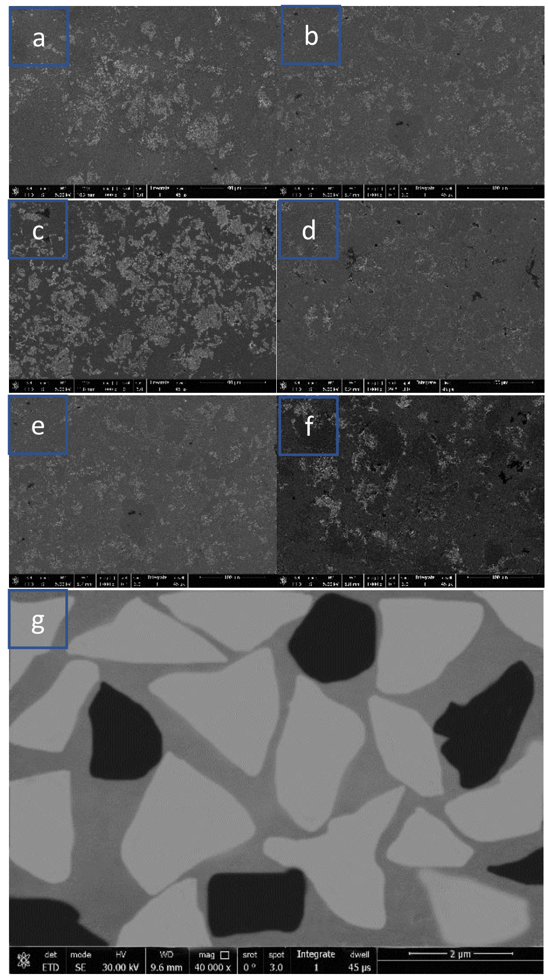

Fig. 3 SEM Micrograph of the Cu/MoS_2 matrix (a), reinforced (b) 0.2 wt. %GNs, (c) 0.4 wt. %GNs, (d) 0.6 wt. %GNs, (e) 0.8 wt. %GNs and (f) 1 wt. %GNs; (g a) of the Cu/10 wt. % MoS_2 matrix and Cu/10 wt. % MoS_2 /1 wt.%GNs, respectively for particle size

Figure 4 shows the chemical composition of the EDAX analysis of the sample, which contains 1 wt. %GNs. It exhibits that the region that analyzed contains 34.7 wt. % of copper, 22 wt. % of molybdenum, 40.68 wt. % of sulfur, and 2.62 wt. % of graphene that appears in the form of carbon.

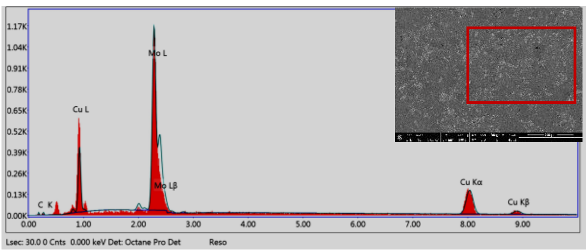

Fig. 4 EDAX analysis Cu/MoS_2 matrix reinforced 1 wt. %GNs

3.2 Hardness Measurement

Figure 5 shows the impact effect of the GNs weight percentages on the hardness of the Cu/MoS_2 composite. It is clear that the hardness increases gradually with increasing GNs content up to 0.4 wt. % then decreased. The increase in hardness with the addition of 0.4 wt.% GNs to the copper matrix is believed to be caused by the good distribution, and good adhesion of the graphene layer with the matrix that holds the particles of copper and MoS_2 together, and prevents the indenter of the macro-hardness to penetrate in the composite. Decreasing the hardness of the Cu/MoS_2 composite at percentages over than 0.4 wt. %GNs may be related to producing some stuck layers of GNs, and may also be attributed to change of the ordination of GNs in the matrix as a result of increasing its percentages.

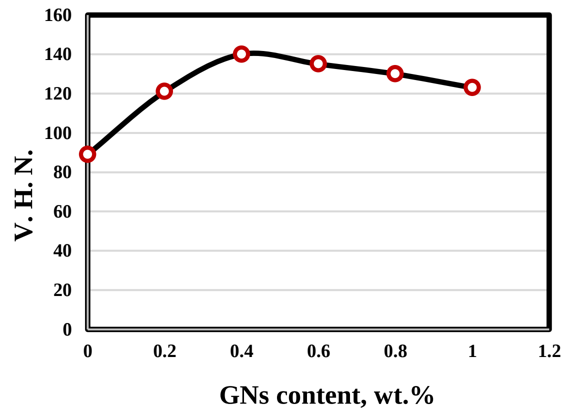

Fig.5 Micro-hardness of the fabricated nano-compoistes

3.3 Composites Wear Rate

Copper/graphene composite is one of the few studied metal matrix composites. Introducing graphene into a copper matrix is hoping to further improve in mechanical, electrical and thermal performance. Graphene is considered as a novel solid lubricant material as its excellent performance in previous studies [8-11]. Figure 6 shows the influence of the graphene contents on the wear rate of the Cu/MoS_2 composite at different loads. Two phenomena are observed from the figure; the first is that the wear rate of the Cu/MoS_2/GNs composites increases with increasing the load, and the second is that the wear rate decreases with increasing the graphene percentages up to 0.4GNs. The results show that the wear rate of the Cu/MoS_2 composite decreases from 0.02013g/min to 0.00559g/min as a result of reinforcing it with

0.4GNs wt. %. Reinforcing of Cu/10MoS_2 composite with GNs leads to decrease the percentage of the MoS_2, and this reduces the number of sliding MoS_2 layers and consequently minimizing the wear rate. Graphene is produced as a result of graphite decomposition [12]. Graphene as a two dimensions layer lost its lubricant ability, and its presence in the matrix in a homogenous distribution manner leads to increase the strength of the copper matrix as the hardness results showed [6].

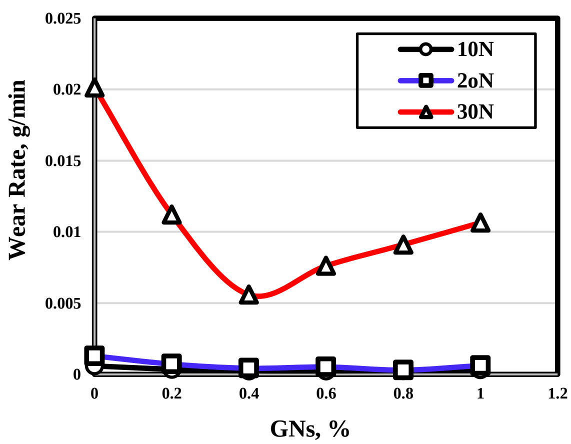

Fig. 6 wear rate of the fabricated composites

Conclusions

According to the results and their discusion, the conclusion can be summarized in the following points.

- The microstructure refers to the good distribution of the MoS2 and GNPs in the copper matrix and good adhesion.
- The EDAX analysis emphasized the presence of the used elements and no strange elements detected.
- The hardness was improved by reinforcing the matrix with the GNs up to 0.4 wt. %.
- The wear rate and the coefficient of friction were enhanced by increasing the GNs up to 0.4 wt. %.

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Conflicts of interest

The authors have no conflict of interest related to this work.

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References

1. Bae S, Kim H, Lee Y, Xu X, Park JS, Zhang Y, et al. Roll-to-roll production of 30-inch graphene films for transparent electrodes. Nat Nanotechnol 2010;5:574e8.
2. Zang X, Chen Q, Li P, He Y, Li X, Zhu M, et al. Highly flexible and adaptable, all-solid-state supercapacitors based on graphene wovenfabric film electrodes. Small 2014;10:2583e8.

3. Li X, Zang X, Li X, Zhu M, Chen Q, Wang K, et al. Hybrid heterojunction and solid-state photoelectrochemical solar cells. Adv Energy Mater 2014;4:1400224.
4. Li X, Xie D, Park H, Zeng Helen T, Wang K, Wei J, et al. Anomalous behaviors of graphene transparent conductors in graphene-silicon heterojunction solar cells. Adv Energy Mater 2014;3:1029e34.
5. Wang Y, Wang L, Yang T, Li X, Zang X, Zhu M, et al. Wearable and highly sensitive graphene strain sensors for human motion monitoring. Adv Funct Mater 2014;24:4666e70.
6. H. M. Yehia, Microstructure, physical and mechanical properties of the Cu/ (WC-TiC-Co) nano-composites by the electro-less coating and powder metallurgy technique, Journal of Composite Materials, (2018).
7. H. M. Yehia, F. Nouh, O. El-Kady, Effect of graphene nano-sheets content and sintering time on the microstructure, coefficient of thermal expansion, and mechanical properties of (Cu /WC –TiC-Co) nano-composites, Journal of Alloys and Compounds, 764 (2018) 36-43.
8. C.F. Gutierrez-Gonzalez, A. Smirnov, A. Centeno, A. Fernandez, B. Alonso, V.G. Rocha, et al., Wear behavior of graphene/alumina composite, Ceram. Int. 41 (6) (2015) 7434e7438.
9. W. Zhai, X. Shi, M. Wang, Z. Xu, J. Yao, S. Song, et al., Grain refinement: a mechanism for graphene nanoplatelets to reduce friction and wear of Ni3Al matrix self-lubricating composites, Wear 310 (1e2) (2014) 33e40.
10. D. Berman, A. Erdemir, A.V. Sumant, Graphene: a new emerging lubricant, Mater. Today 17 (1) (2014) 31e42.
11. J. Lin, L. Wang, G. Chen, Modification of graphene platelets and their tribological properties as a lubricant additive, Tribol. Lett. 41 (1) (2011) 209e215.
12. Ming Zhou and et al, Production of Graphene by Liquid-Phase Exfoliation of Intercalated Graphite, *Int. J. Electrochem. Sci.*, 9 (2014) 810 – 820.