Engineering Science and Technology, an International Journal ■■ (2015) ■■–■■

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Engineering Science and Technology, an International Journal

journal homepage: http://www.elsevier.com/locate/jestch

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Full Length Article

Tribological performance of self-lubricating aluminum matrix nanocomposites: Role of graphene nanoplatelets

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ARTICLE INFO

Article history:
Received 29 June 2015
Received in revised form
19 August 2015
Accepted 11 September 2015
Available online

Keywords:
Graphene nanoplatelets (GNP)
Metal Matrix NanoComposites (MMNCs)
Wear
Powder metallurgy

ABSTRACT

In the present investigation, aluminum matrix nanocomposites reinforced by graphene nanoplatelets were synthesized by powder metallurgy method. The microstructure of the Al-Graphene nanoplatelets sample was investigated by TEM. The hardness measurements of these samples were investigated using a Rockwell hardness tester. To investigate the tribological behavior of aluminum matrix composites reinforced by graphene nanoplatelets and pure aluminum, pin-on-disk experiments were conducted on the prepared samples. In the experiments, the influence of reinforcement, volume fraction, normal load, and sliding velocity on the tribological performance was investigated. Results showed that the wear rate of Al-1wt.% GNP is increased with increasing normal loads. However, the coefficient of friction (COF) of the Al-1wt.% GNP decreased with increasing normal loads. Formation of graphene film on the worn surface of Al-1wt.% GNP sample and morphology of the worn surfaces of aluminum and composite samples were analyzed by Optical Microscope (OM) and Scanning Electron Microscope (SEM). It was found that the graphene nanoplatelets reinforced nano-composites showed superior tribological properties and demonstrated the ability of the self-lubricating nature of the composite during tribological conditions.

1. Introduction

Aluminum and its alloys are one of the most widely used materials in different industries, such as aerospace, automotive and defense. This is due to their superior physical and mechanical properties, such as light weight, high specific strength, high specific modulus, and low thermal expansion coefficient [1,2]. However, the tribological behavior of aluminum and aluminum alloys is not superior and few methods have been proposed in the past to improve the tribological behavior of aluminum alloys. One method to reduce friction and consequently deterioration of material under wear condition is applying liquid or solid lubricants. However, in some cases, such as high vacuum environment, high-speed, high applied loads, and extremely low or high temperature conditions, liquid and grease type lubricants are unfeasible [3]. Another approach to enhance the tribological performance of aluminum and aluminum alloys is by replacing the liquid and grease type lubricants with solid lubricant coatings. The coatings are applied on the surface of materials by depositing via chemical or physical vapor deposition techniques to form a solid lubricant layer [4,5]. The disadvantages of solid lubricant coatings are limited lifetime, difficulty in replenishment, oxidation and aging-related degradation, and poor adhesion. Therefore, to avoid the drawbacks of both the liquid and grease type lubricants and the solid lubricant coatings, a new method has been proposed by embedding carbonous materials in the metal matrices [6]. More specifically, the addition of carbon allotropes, such as Carbon Nano Tubes (CNTs) and graphene as reinforcement in metal matrices to improve the performance of materials has been a topic of interest over the last few years [7]. Embedding nano carbonous materials into polymer matrices improved the tribological properties of polymer matrix composites [8,9]. By adding these nano carbonous materials into aluminum matrices one can synthesize self-lubricating aluminum matrix composites due to superior lubricant nature of CNTs and graphene materials [10]. As a result, the focus of the current research is to synthesize the self-lubricating aluminum metal matrix composites reinforced with graphene and study their tribological behavior.

Graphene is an allotrope of carbon atoms which has drawn attention of researchers recently due to its superior properties, such as high elastic modulus, good electrical conductivity, good thermal conductivity, and self-lubricating behavior [3,11]. Researchers have been adopting solid state processing or powder metallurgy techniques to synthesize metal matrix nanocomposites reinforced by graphene [12,13]. Graphene has been used as reinforcement in metal

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http://dx.doi.org/10.1016/j.jestch.2015.09.005
2215-0986/

matrices, such as aluminum [14], magnesium [15] and copper [16] to improve the properties of metal matrix nanocomposites (MMNCs). However, only a few attempts have been made to understand the tribological behavior of these metal matrix nanocomposites reinforced by graphene. Recent studies have shown that graphene as reinforcement in the matrix can act as a self-lubricating material leading to improvement in the tribological behavior of the MMNCs [14–16]. However, there are a limited number of investigations about aluminum/graphene nanocomposites to study the effect of different material and test parameters, such as normal load, sliding speed, and weight fraction of graphene nanoplatelets on tribological behavior of the metal matrix nanocomposites reinforced by graphene nanoplatelets. Ghazaly et al. [17] have synthesized the aluminum/graphene composites at different weight percentage (0.5, 3 and 5 wt.%) by employing powder metallurgy technique. A combination of cold compaction and hot extrusion at ~0.45T_m (305 °C) were employed to synthesize aluminum/graphene self-lubricating nanocomposites. In their study, the effect of weight percentage of graphene on wear rate of self-lubricating nanocomposites was investigated. The results showed that self-lubricating composite reinforced by 3 wt.% graphene has the best tribological properties under dry wear test conditions when compared to unreinforced and other compositions of graphene reinforced composites. Scanning Electron Micrographs of worn surfaces of unreinforced AA2124 and AA2124/graphene nanocomposites revealed longitudinal grooves in all samples. The results showed that the scratches, craters, delamination of AA2124/3wt.% graphene composite was significantly less than that of unreinforced AA2124. Thus, the authors concluded that the variations in the tribological properties of unreinforced alloy and AA2124/3wt.% graphene composite are due to their variations in wear regimes where unreinforced alloy exhibited severe wear regime and AA2124/3wt.% graphene composite exhibited mild wear regime. Rajkumar and Aravindan [16] have synthesized the copper matrix composites reinforced by micron- and nano-size graphite using powder metallurgy method to investigate the tribological behavior of these composites. The results showed that the nano graphite reinforced copper matrix composites have higher wear resistance and lower COF compared to the copper matrix composites reinforced by micron-graphite.

The friction and wear properties are system dependent [18]. Different researchers used different operating parameters and testing conditions in order to evaluate the friction and wear properties of composites. It is rather difficult experimentally to study the wide range of operating parameters on friction and wear properties as a large number of tests is needed. Further, the friction and wear properties will change within the same order of magnitude. Hence, in the present investigation, aluminum matrix nanocomposite reinforced by graphene nanoplatelets is synthesized at room temperature by powder metallurgy technique and the effect of tribological parameters, such as normal load and sliding speed and materials factor, such as weight fraction of graphene nanoplatelets on wear and friction behavior of the MMNCs were investigated. In addition, the wear mechanisms of these samples at different conditions were investigated.

2. Materials and methods

The primary materials used in this investigation were 99% pure aluminum powder (Acros Organics, Waltham, MA) with an average particle size of 75 µm and graphene nanoplatelets (GNP) M5 (XG Sciences, Lansing, MI) with an average thickness of approximately 6 nm and an average diameter of 5 µm. Fig. 1 shows the SEM micrographs of as-received pure Al powder and GNPs at magnifications of 400× and 20,000×, respectively. To produce nanocrystalline (NC) MMNCs, the reinforcements (GNPs with 0.1 and 1 wt.%) were dispersed in 99.5% anhydrous ethanol by ultrasonication. The aluminum

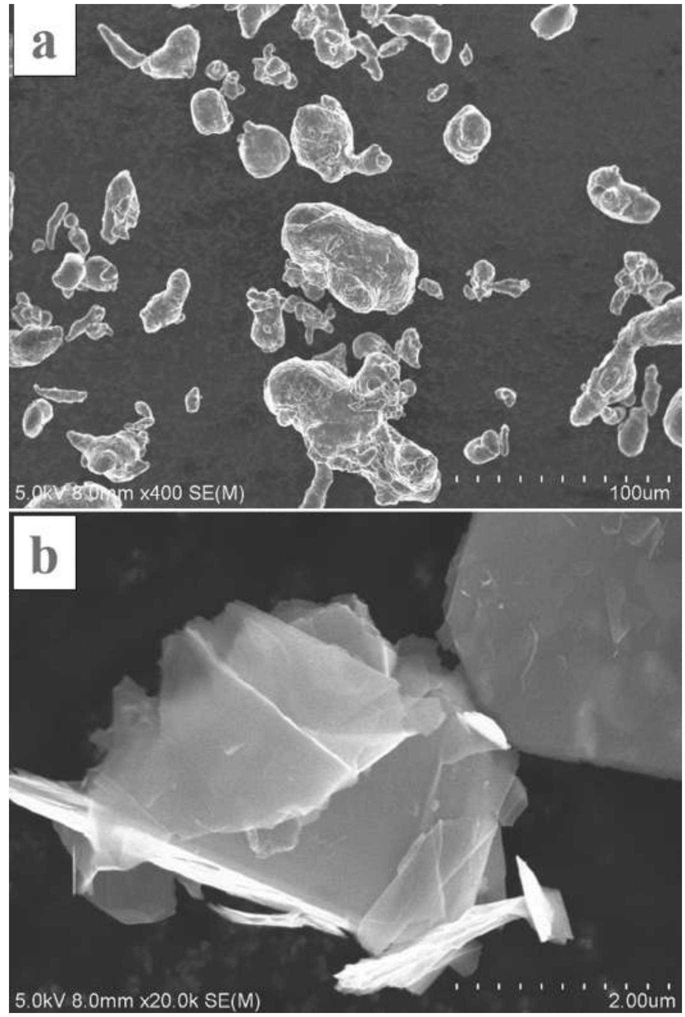

Fig. 1. SEM micrographs of (a) as-received pure aluminum powder at 400× magnification and (b) GNPs at 20,000× magnification.

powder and the reinforcement slurry were added to a Szegvari attritor mill equipped with an alumina reservoir and then milled for 6 hours at 500 rpm using a Ball to Powder Ratio (BPR) of 15:1 (5 mm diameter zirconia balls). The milled composite powders were dried at 135°C for 1 hour to remove the ethanol. It is important to note that the initial particle sizes of aluminum powder was found to be 75 micron. However, after 6 hours of milling, the size and morphology of the aluminum powders changed significantly. In the literature [19], it has been demonstrated that the particle size of powders decreases by milling process. In addition, the morphology of the aluminum powders after milling by attritor mill change to flaky shape. The graphene nanoplatelets distribute uniformly between flaky shape aluminum powders in matrices. By adding less than 1wt.% graphene to the aluminum matrix, a good distribution of graphene in the metal matrix can be achieved. The morphology and size of the powders after 6 hours of milling was investigated by SEM. Fig. 2 shows the SEM micrographs of flaky shapes Al-1wt.%GNP powders after 6 hours of milling. The dried powders were consolidated by single action cold compaction with 200 MPa at room temperature followed by single action hot compaction with 500 MPa at 525 °C such that a 25.4 mm diameter cylinder with a height of 10 mm was produced. Unreinforced pure aluminum was also produced by the same method to compare with the properties of the composite samples. In the present research, it is important to indicate that the GNP amount is restricted to 1 wt.% in the aluminum

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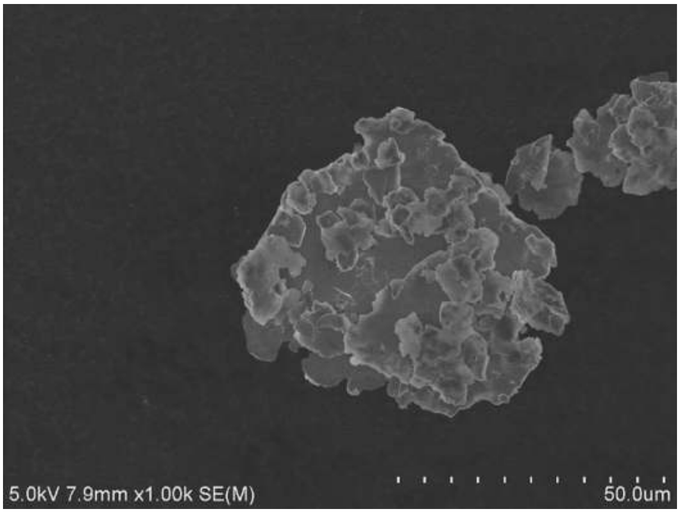

Fig. 2. Morphology of Al-1wt.%GNPs powder after 6 hours of milling.

matrix. This is because a larger amount of graphene in the aluminum matrix results in poor mechanical properties. Efforts were also made to synthesize samples with GNP amount up to 5 wt.% in the aluminum matrix as a criteria for choosing optimum GNP amount in the aluminum matrix. It was found that the amount of graphene in the aluminum matrix is substantial, a good distribution of reinforcement in the matrix did not occur. Furthermore, the mechanical properties of GNPs sample above 1 wt.% were decreased significantly with increasing GNP. This is because of agglomeration and unsuitable distribution of reinforcement into the aluminum matrix. The mechanical properties are important factors that affect tribological behavior of MMNCs, the research was further focused only to Al-1wt%GNPs samples.

To investigate the microstructure of the Al-GNP sample, TEM was conducted on cross section of the sample. TEM sample was prepared using in-situ lift-out technique by an FEI Strata 400 Dual Beam FIB/SEM. The sample was imaged in FEI Tecnai TF-20 FEG/TEM operated at 200 kV.

To study the mechanical properties, the hardness measurements of these samples were performed using a Rockwell hardness tester. For each sample, five hardness measurements were recorded and the average of hardness values was considered. Table 1 presents the properties of the materials used in the tests. It is important to indicate that increasing the graphene content more than 1% into aluminum matrix decreased the hardness values significantly. The reason could be due to difficulty in making uniform distribution of reinforcement into the matrix that ultimately lead to aggregation of graphene in the matrix and consequently caused the decrease in the mechanical properties of composite.

To investigate the tribological behavior of the samples, pin-on-disk tests under dry condition were conducted. In the tests, cylindrical pins with dimension of 6 mm in diameter and 8 mm in height were utilized from the hot compacted samples. The counterpart disk material was made of stainless steel 440C with a dimension of 55 mm in diameter and 10 mm in thickness. The pin-on-disk experiment was conducted for different normal loads (5, 10 and 15 N) and sliding speeds (50, 100 and 150 rpm) at a constant sliding distance of 1.13 km. The coefficient of friction (COF) and volume loss (wear rates) were measured during the wear tests. The statistical analysis was conducted to ascertain the p-values of significance. If the calculated p-value falls below the significance value (0.05) it shows that the considered factor (normal load or sliding speed) has a substantial effect on the response variable (COF or wear rate). The worn surfaces of the samples were investigated using Optical Microscope (OM) and Scanning Electron Microscope (SEM). The debris obtained after the wear test were investigated using Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS).

3. Results and discussion

Fig. 2 shows the morphology of Al-1wt.%GNPs powder after 6 hours of milling. The morphology of aluminum powder changed from regular shape (Fig. 1(a)) to flaky shape (Fig. 2) after 6 hours of milling. As a result, after consolidation processing of flaky shape powders, a layered microstructure can be expected. TEM micrographs of layered microstructures of Al-1wt.%GNP sample at two different magnifications were shown in Fig. 3(a) and (b). The Al matrix and GNPs were presented by red arrows in the figures.

Fig. 4 shows the variation of coefficient of friction (COF) with normal load for pure aluminum, Al-0.1 wt.% GNP and Al-1 wt.% GNP ($p < 0.001$) at constant sliding speed of 100 rpm. The results indicate that the COF decreases with increasing normal loads in all cases and follows the same decreasing trend. Furthermore, the rate of decrease in COF with normal load is significant at higher normal loads when compared to lower normal loads. The results show that the COF does not change significantly by adding 0.1 wt.% of GNP to the aluminum matrix because of insufficient amount of solid lubricant available at the contact surface. However, higher weight percentage of GNP (1 wt.%) decreased the COF of the composite sample significantly in comparison with other samples.

The variation of wear rate (weight loss) with normal load for pure aluminum, Al-0.1 wt.% GNP and Al-1 wt.% GNP ($p < 0.01$) at constant speed of 100 rpm is presented in Fig. 5. As shown in the figure, the wear rate of the samples increased with increasing normal load. Also, the wear rate of Al-1wt.% GNP is more than the pure aluminum. As indicated earlier in Table 1, Al-1wt.% GNP has the lowest hardness (86.08 ± 0.58 HR_F) while the pure aluminum has the highest hardness (92.48 ± 0.45 HR_F). The hardness of the material plays an important role to explain the wear behavior of materials. Generally, the softer materials have higher wear rates compared to the harder materials [6,20]. Further, in the literature, it is well known that there is an inverse relation between wear rate and hardness of the materials. For this reason, Al-1wt.% GNP shows the highest wear rate compared to other samples. The reduction in hardness is believed to decrease in the load bearing capacity of the Al-1wt%GNP and consequently increased their wear rate. As regard to the variation of wear rate with normal load, in the literature, the well known Archard equation [21] demonstrated that the wear rate is directly proportional to the applied normal load. From the above discussions, it is clear that the current results are in accordance with

Table 1
Properties of the materials used for the tribological experiments.

	Material	Diameter	Height	Relative density (%)	Hardness
Disk	Stainless steel 440C	55 mm	10 mm	–	96.3 $HR_B \approx 228$ H_v
Pin	Pure Al	6 mm	8 mm	98.03	92.48 $HR_F \approx 111$ H_v
	Al/0.1wt% GNP			98.83	87.1 $HR_F \approx 98$ H_v
	Al/1wt% GNP			98.87	86.08 $HR_F \approx 97$ H_v

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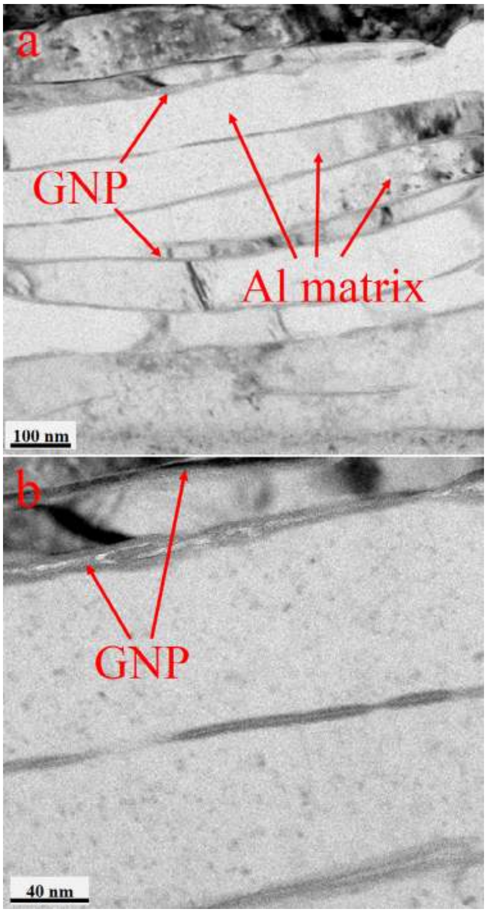

Fig. 3. TEM micrographs of layered microstructures of Al-1wt.%GNPs at different magnifications.

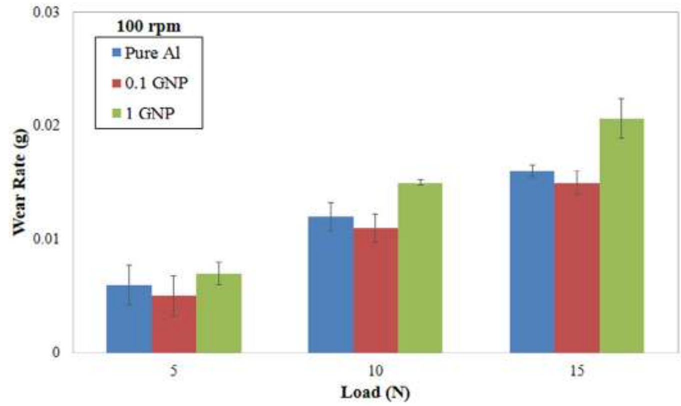

Fig. 5. Variation of wear rate with normal load (N) at sliding speed of 100 rpm for pure Al, Al-0.1wt.% GNP and Al-1wt.% GNP.

the results presented in the literature on the variation of wear rate with normal load and hardness of the materials. As the Al-1wt%GNP has better coefficient of friction when compared to other samples, further investigations are concentrated on this self-lubricating nanocomposites.

The worn surfaces of the pure aluminum and Al-1wt.% GNPs samples were analyzed by using optical microscope, and the micrographs are presented in Fig. 6. Fig. 6(a) presents the optical micrograph of pure aluminum sample. The plowing marks that are formed during dry sliding conditions can be clearly seen in the figure. Fig. 6(b) shows the optical micrograph of Al-1wt.% GNP sample. The formation of graphene film (black) on the worn surface of Al-1wt.% GNP sample is clearly seen. The decrease in the COF of Al-1wt.% GNPs in Fig. 4 is because of formation of the graphene film on the worn surface of the sample. Formation of the graphene film causes to decrease the direct contact between the aluminum matrix

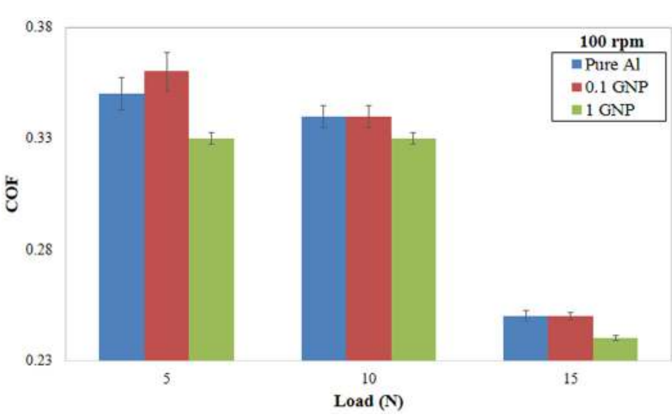

Fig. 4. Variation of COF with normal load at sliding speed of 100 rpm for pure Al, Al-0.1wt.% GNP and Al-1wt.% GNP.

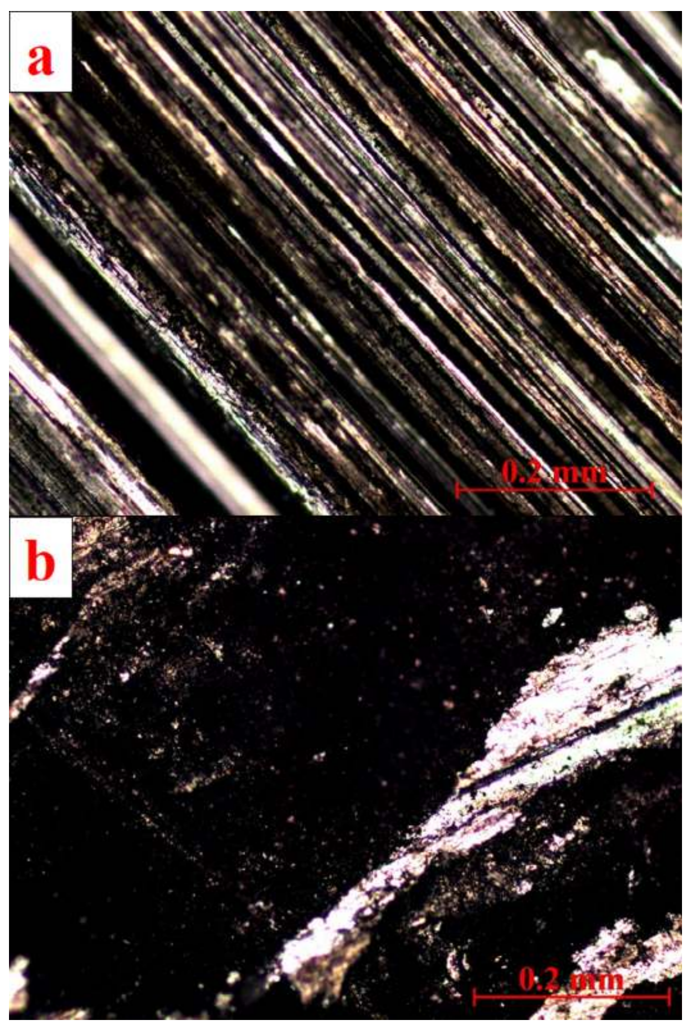

Fig. 6. Optical micrographs of worn surfaces of (a) pure aluminum and (b) Al-1wt.% GNPs.

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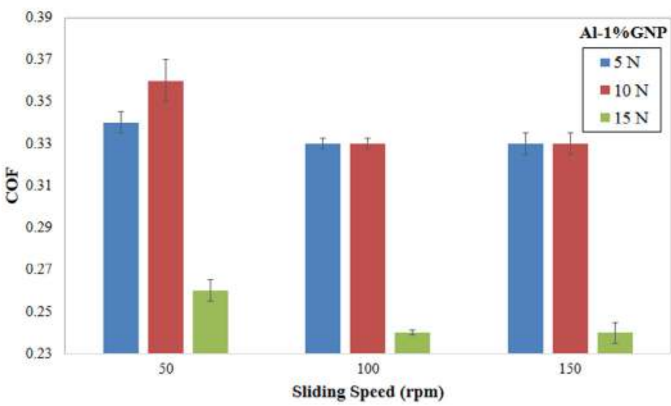

Fig. 7. Variation of COF with sliding speed at different normal loads for Al-1wt.% GNP.

and the steel disk; as a result, the COF friction of Al-1wt% GNP decreased when compared to other samples.

To investigate the effect of sliding speed on the COF of Al-1wt.% GNP, experiments were conducted at various sliding speeds (50, 100 and 150 rpm) using normal loads of 5, 10 and 15 N. The variation of COF with sliding speed at different normal loads for Al-1 wt.% GNP ($p < 0.001$) is presented in Fig. 7. The COF of Al-1wt.% GNP slightly decreases with increasing sliding speeds at different normal loads. It can be seen that the influence of sliding speed on coefficient of friction is significant only at lower sliding speeds. Further, the influence of sliding speed on the coefficient of friction is less significant when compared to the effect of normal load on the coefficient of friction. At higher normal loads, more graphene is projecting out from the pin surface due to plowing between pin and disk. Consequently, the direct contact between surfaces of the sample and disk is decreased by the graphene layer and this ultimately decreased the COF. Fig. 8 shows the variation of wear rate with sliding speed for Al-1wt.% GNP composite ($p > 0.05$). The wear rate of Al-1wt.% GNP is much higher at 15 N load when compared to 5 and 10 N loads (Fig. 5). This higher amount of wear rate has a direct influence on the COF. As the amount of graphene nanoplatelets between the contacting surfaces increases, the COF decreases owing to lubricating tendency of the graphene nanoplatelets available at the sliding interface (Fig. 7).

It can be seen that the wear rate of Al-1wt.% GNP increases with increasing normal load from 5 to 15 N (Fig. 8). Increasing normal load causes to increase the amount of plastic deformation on the surface and hence increase the real area of contact at the surface during the wear test [6]. As stated in the Archard equation [21], the wear rate is directly proportional to the applied normal load. The current results are in accordance with the results presented in the literature on the variation of wear rate with normal load. It can be seen in Fig. 8 that the wear rate first decreases slightly and then increases with increasing the sliding speed. Although, the exact reason for this variation is unknown, it can be believed that there could be a transition from severe wear to mild wear and then back to severe wear as the sliding speed is increased. More research needs to be made in this direction to understand this variation and exact reason; however, similar trends were also reported by Kozma [22] and Al-Samarai et al. [23].

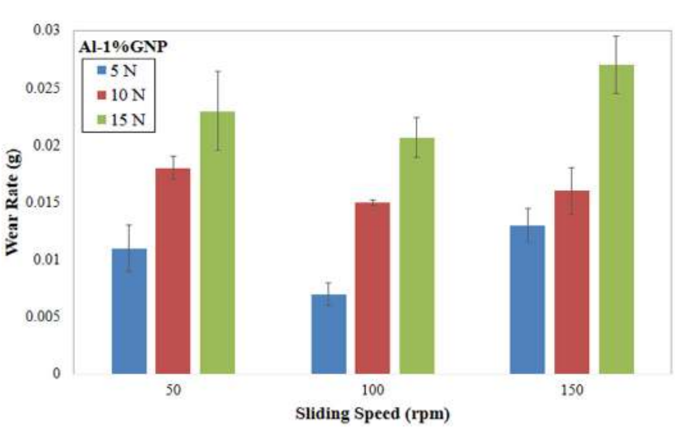

Fig. 8. Variation of wear rate with sliding speed at different normal loads for Al-1wt.% GNP.

The worn surfaces of the Al-1wt.%GNP samples after the wear experiments were investigated using SEM. Fig. 9 shows the SEM micrographs of the worn surfaces of the Al-1wt.% GNP samples at 100× magnification at various normal loads and sliding speeds. The worn surfaces have parallel grooves in the direction of sliding with varying groove width and depth that depend on the normal load and sliding speed. These types of grooves which show on the worn surfaces in Fig. 9 are due to abrasive wear during sliding conditions. More specifically, the wear mechanism in the Al-1wt.% GNP is abrasive wear even at low loads. In this current sliding situation, the contribution of adhesion could be significantly less when compared to abrasion. When self-lubrication or lubrication action is effective at the interface, basically the low sliding speed experiments represent that the tests were conducted under boundary lubricated regime [24]. At this situation, the adhesion is minimized (if not eliminated) due to the presence of lubrication effect and thus the contribution of abrasive wear mode is the key factor [24,25].

The shallow depth and narrow width of the groove were observed on the surface of the sample during the wear test at 5 N and 50 rpm. The deepest depth and broadest width were observed on the worn surface of the sample during the wear test at 15 N and 150 rpm. For a given normal load, the groove width and depth increased with increasing sliding speed; as a result, the highest sliding speed recorded extensive damage on the worn surfaces compared to the lowest sliding speed. Similarly, for a given sliding speed, the groove width and depth increased with increasing normal load. At higher normal loads, the damage on the worn surfaces was more considerable when compared to the lower normal loads. From the above discussions, it can be inferred that the abrasive wear of Al-1wt.% GNP increases with increasing normal loads at a constant sliding speed. In a similar manner, the abrasive wear of Al-1wt.% GNP increases with increasing sliding speeds at a constant normal load.

Fig. 10 shows the SEM micrographs of the worn surfaces of pure aluminum and aluminum matrix composite reinforced by graphene nanoplatelets samples after the wear test at normal load of 5 N and sliding speed of 100 rpm. The SEM micrographs show that the Al-1wt.% GNP reinforced composite had the significant amount of damage on the worn surface and the grooves produced on the worn surface of this composite sample were the deepest when compared to the worn surfaces of aluminum sample. In addition, the SEM micrographs revealed that the worn surface of the Al-0.1wt.% GNP has the least damage compared to aluminum and Al-1wt.% GNP samples. These SEM results on surface damage correlate well with the wear data obtained in the pin-on-disk tests where the data points in Fig. 5 at normal load of 5 N showed that the wear rate is highest for Al-1wt.% GNP (hence severe damage on the pin surface as shown in Fig. 10) and the wear rate is lowest for Al-0.1wt.% GNP (hence, mild damage on the pin surface as shown in Fig. 10). The debris obtained after the wear test has been investigated by SEM and EDS (Fig. 11). Fig. 11(a) shows the SEM micrographs of Al-1wt.% GNP at 200× magnification. The size of the debris varied in a broad range from submicron size to more than 100 microns. During the wear

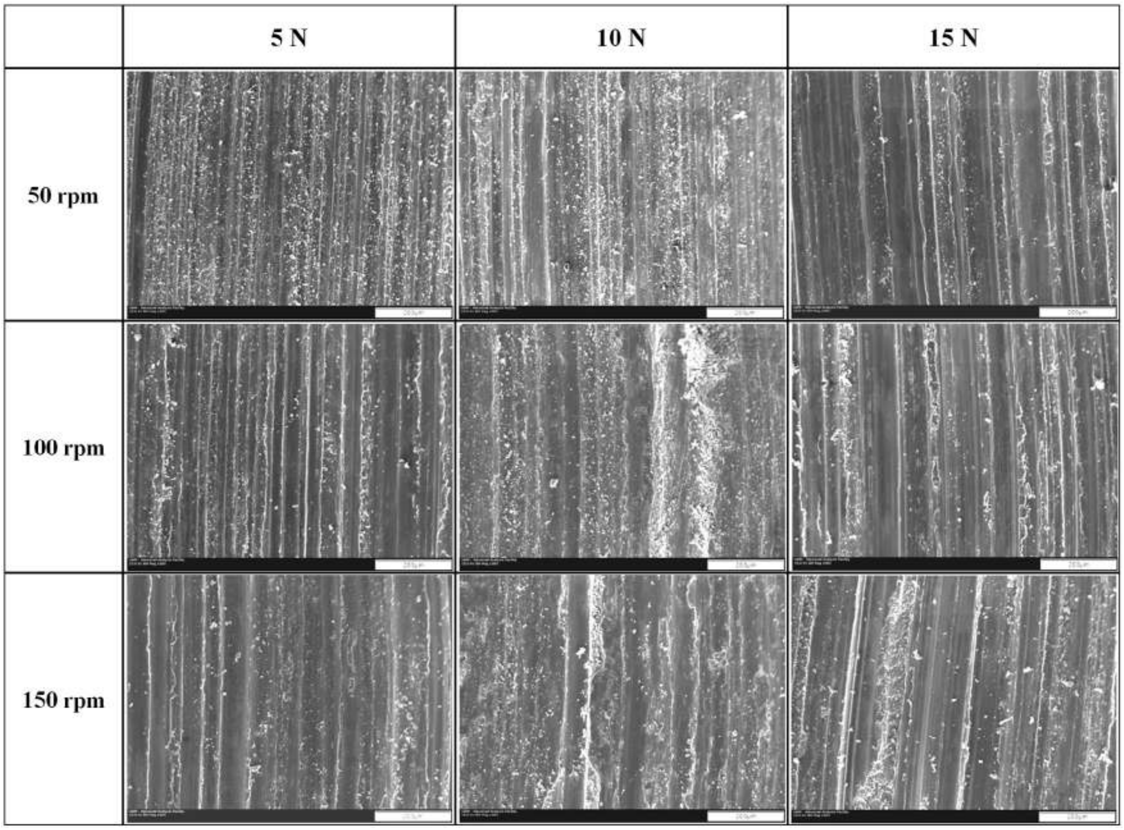

Fig. 9. SEM micrograph of Al-1 Wt.% GNP at different loads and sliding speeds at magnification of 100×. (The sliding direction is from bottom to top in the SEM images).

test the temperature at the interface of the pin and disk increases. The fresh surface of aluminum would be exposed during sliding and become active, and the development of high temperature at the interface during sliding leads to the formation of aluminum oxide in the wear test. The EDS result (Fig. 11(b)) confirms the formation of oxide during wear test (Al = 77.51 atomic% and Oxygen = 22.14 atomic%).

From the above analysis, it can be seen that the Al-1wt.% GNP recorded the highest wear rate and the lowest coefficient of friction during sliding. Higher wear rate leads to higher amount of graphene particles released between the contacting surfaces during sliding. These graphene particles act as solid lubricants at the interface and enhance the lubricating effect. For this reason, the Al-1wt.% GNP composite recorded the lowest coefficient of friction. Thus, it can be inferred that the Al-1wt.% GNP composite can be considered as a promising self-lubricating composite materials as this materials showed similar frictional and wear response as that of other self-lubricating composite materials [26].

4. Conclusions

In this study, Al matrix nanocomposites reinforced by GNP was synthesized and the tribological behavior of these composites was investigated. The experimental findings are summarized as follows:

- The COF of the pure aluminum and aluminum matrix nanocomposites reinforced by GNP decreases with increasing the normal load.
- Addition of 0.1 wt.% of GNP to the aluminum matrix did not change the COF significantly when compared to the pure aluminum sample. However, the increasing amount of GNP to 1wt.% significantly improved the COF of the pure aluminum.
- Among the three materials, namely, aluminum, Al-0.1wt.% GNP composite and Al-1wt.% GNP composite, the Al-1wt.% GNP composite recorded the lowest COF.
- The influence of sliding speed on coefficient of friction is significant only at lower sliding speeds.

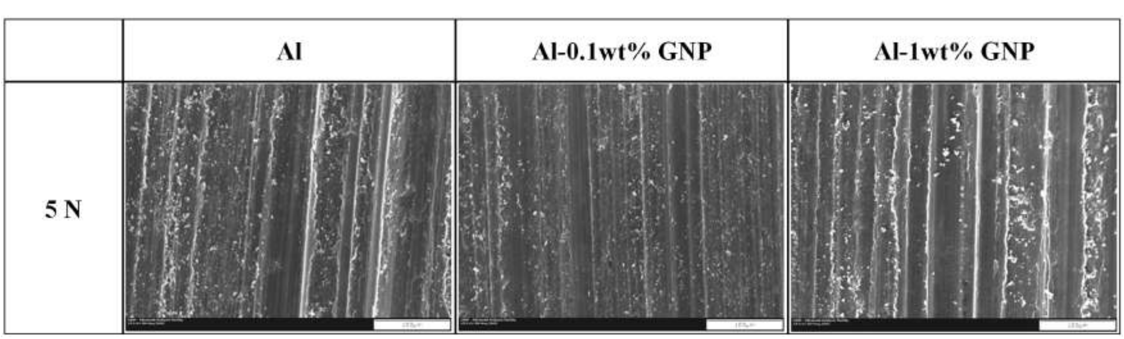

Fig. 10. SEM micrographs of the worn surfaces of different samples at speed of 100 rpm and load of 5 N at magnification of 200×.

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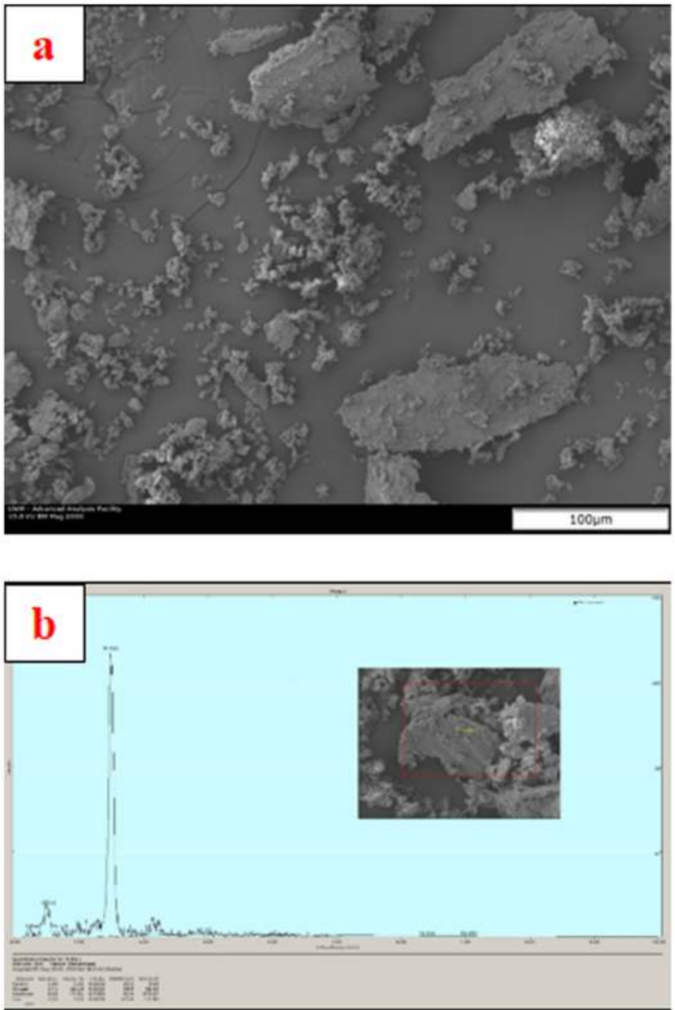

Fig. 11. (a) SEM micrographs and (b) EDS results of wear debris of Al-1 Wt.% GNP sample.

- The wear rate of the pure aluminum and aluminum matrix nanocomposites reinforced by GNP increased with increasing the normal load.
- Among the three materials, namely, aluminum, Al-0.1wt.% GNP composite and Al-1wt.% GNP composite, the Al-1wt.% GNP composite recorded the highest wear rate.
- The wear rate of Al-1wt.% GNP is higher at higher normal loads when compared to lower normal loads. However, the COF of the Al-1wt.% GNP is lower at higher normal load.
- The SEM investigation of the worn surfaces had shown that abrasive wear was the main wear mechanism in these composites.
- The highest wear rate and lowest coefficient of friction of the Al-1wt.% GNP is attributed to self-lubricating behavior of the composites.

Acknowledgments

This material is based upon work supported by the U.S. Army Research Laboratory under Cooperative Agreement No. W911NF-08-2-0014. The views, opinions, and conclusions made in this document are those of the authors and should not be interpreted as representing the official policies, either expressed or implied, of Army Research Laboratory or the U.S. Government. The U.S. Government is authorized to reproduce and distribute reprints for Government purposes notwithstanding any copyright notation herein.

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Please cite this article in press as: Meysam Tabandeh-Khorshid, Emad Omrani, Pradeep L. Menezes, Pradeep K. Rohatgi, Tribological performance of self-lubricating aluminum matrix nanocomposites: Role of graphene nanoplatelets, Engineering Science and Technology, an International Journal (2015), doi: 10.1016/j.jestch.2015.09.005