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Al/Graphene/CNT Hybrid Composites: Hardness and Sliding Wear Studies

Graphene and carbon nanotubes are two carbon based materials known for their unique wear and friction properties. It would be quite interesting to understand the wear behavior of aluminium hybrid composites when these two nanosize reinforcements are incorporated into it. The hybrid composites with varying weight fractions of graphene (1, 2, 3 and 5 wt.%) and fixed CNT content of 2 wt.% were produced using powder metallurgy technique. The effect of varying graphene content on hardness and sliding wear of hybrid composites was studied. The wear tests were done as per ASTM G-99 standard with fixed sliding velocity (2 m/s) and sliding distance (1200 m) but varying applied load (10 - 30 N). Worn surface analysis was conducted using scanning electron microscope to arrive at wear mechanisms responsible for wear of aluminium and its hybrid composites. Increase in graphene content led to increase in bulk hardness with highest value of 61 RHN for hybrid composite with 3 wt.% graphene content. The wear rate of hybrid composites was found to be decreasing with enhancement in graphene content. Lower wear rate in hybrid composites was due to the formation of lubricating layer on the worn surface.

Keywords: *Graphene; Carbon nanotubes; Powder Metallurgy; Hardness; Sliding Wear*

1. INTRODUCTION

It is well known that aluminium matrix based composite materials are quite popular and significant amount work is done on exploring mechanical and tribological properties [1-9]. The main advantage of using aluminium as matrix materials is its low density, good strength, and corrosion resistance. Conventional aluminium matrix composites were reinforced with micron sized reinforcements while latest development is using the same reinforcements but in nanosize. In last two- or three-decades lot of efforts has gone into research in which nanosize reinforcements are SiC, TiO_2, B_4C, h-BN and CuO. However, the most explored nanosize reinforcements are carbon-based materials like carbon nanotubes, graphene oxide and graphene. These materials are known to have exceptionally high modulus, tensile strength, thermal conductivity, and electrical conductivity [10]. These materials are known to strengthen the aluminium matrix materials in a significant way. But when comes to engineering components strength is not only the factor to be considered but wear also play and important role. In order to reduce wear rate and friction related issues, many solid lubricants are introduced in the aluminium matrix. Earlier works reported development of self-lubricating composites reinforced with lubricating materials like graphite and

carbon fibers [11,12]. However, the non-wetting and large size graphite particles tend to form defects resulting in premature failure of composites. In this regard nanosize carbon-based reinforcements are being tried to replace the micron size counterparts in self-lubricating composites.

Both graphene and CNTs have emerged as solid lubricants and are gaining a lot of attention as strong candidate materials for development of self-lubricating composites [13,14]. The addition of these carbon-based materials to aluminium matrix has shown positive outcomes like reduced wear rate and coefficient of friction. The atomically smooth surface and easy shear capability are prime attributes for excellent lubricating behaviour and highly desirable in composite for wear protection. Many works have been reported on improvement in wear resistance and decrease in coefficient of friction of aluminium matrix composites after addition of graphene and CNTs. In their work, Kim et al. [15] reported the sliding wear behaviour of CNTs reinforced aluminium composites. It was found that higher the hardness of composites the lower was the wear rate. Oxidation wear was found to be predominant wear mechanism operating in the composites. Wu et al. [16] studied the wear behaviour of AlSiMg matrix reinforced graphene composite prepared using selective laser melting process. With increasing sliding speed from 0.3 – 0.9 m/s, the wear rate was found to decrease, and the wear mechanism changed from abrasive to delamination wear. The change in wear regime was attributed to formation of mechanical mixed layer. Apart from this, some work on wear behaviour of aluminium hybrid composites consisting of either graphene or CNTs as one of the reinforcement

materials was also reported. Zhang et al. [17] studied tribological properties of graphene encapsulated SiC particles reinforced aluminium composite. Compared to unreinforced aluminium the hybrid composite showed reduced wear rate and coefficient of friction which was due to the formation of lubricating tribolayer. Overall, these studies suggest that the incorporation of graphene or CNTs tends to reduce the wear rate of aluminium composites by forming a lubricating layer.

In this work, development of graphene/CNTs reinforced aluminium composite is reported. As far authors are concerned very meagre amount of work has been done using graphene/CNTs hybrid combination. However, these works focused majorly on improving the strength, while tribological behaviour is rarely explored [18,19]. The wear rate as function of varying load is reported with worn surface analysis conducted through scanning electron microscope (SEM).

2. EXPERIMENTATION

2.1 Materials

Commercial purity aluminium powder has been chosen as matrix material due to its extensive application in automotive and engineering applications. The CNTs having diameter in the range 10-20 nm, length 3-8 μm, and graphene having lateral dimension of 5-10 μm are used as reinforcements. The SEM micrographs of graphene and CNTs are shown in Fig. 1. Hybrid composites having different wt%. of graphene (1 wt%, 2 wt%, 3 wt% and 5 wt%,) and fixed CNTs (2 wt%) were fabricated by powder metallurgy route.

2.2 Fabrication

Initially, aluminium powder, CNTs and graphene were mixed thoroughly to get different compositions of hybrid composites using a planetary ball mill. The ball milling was done for 120 minutes at 250 rpm to obtain mixture of hybrid composite powder. The mixture was cold compacted in a die assembly made of stainless steel (Grade: H13) by applying a pressure of 350 MPa. Green compacts were then sintered in a tubular furnace under argon gas atmosphere. The photographs of instruments/tools used in this entire experimentation right from cold compaction to finished hybrid composite compacts are depicted in Fig. 2. The sample designation with varying graphene content is shown in Table 1.
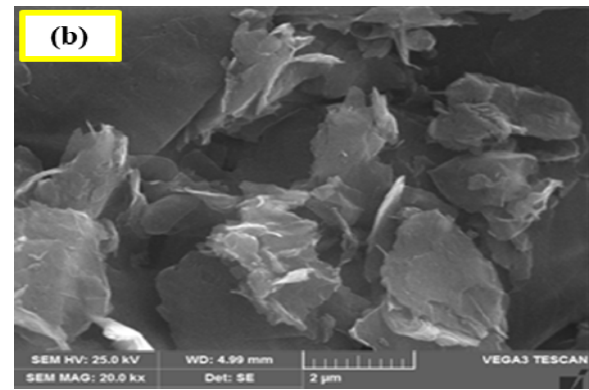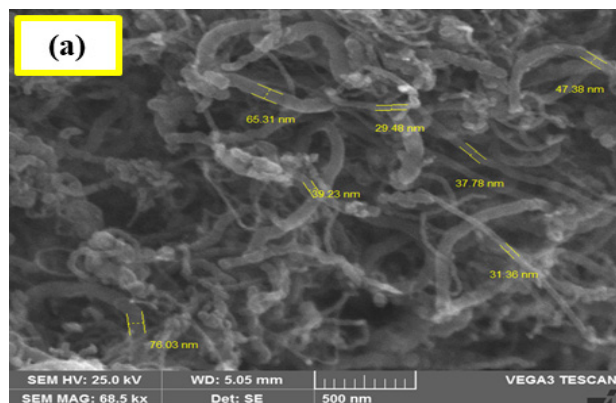

Figure 1. SEM micrographs of procured (a) CNTs and (b) graphene.

Table 1. Designation of hybrid composite samples

S.No.	Composition	Designation
1	Pure Aluminium	AL
2	Al + 2 wt.% CNT	B1
3	Al + 2 wt.% CNT + 1 wt.% Graphene	C1
4	Al + 2 wt.% CNT + 2 wt.% Graphene	D1
5	Al + 2 wt.% CNT + 3 wt.% Graphene	E1
6	Al + 2 wt.% CNT + 5 wt.% Graphene	F1

2.3 Characterization and testing

The microstructural characterization of initial materials, sintered hybrid composites and worn surface analysis was carried out using SEM. The density measurement was done according to Archimedes principle in accordance with ASTM standard B962. The bulk hardness measurements were done using Rockwell hardness tester, which is done in accordance with ASTM standard E18. The average value of three indentations is reported here.
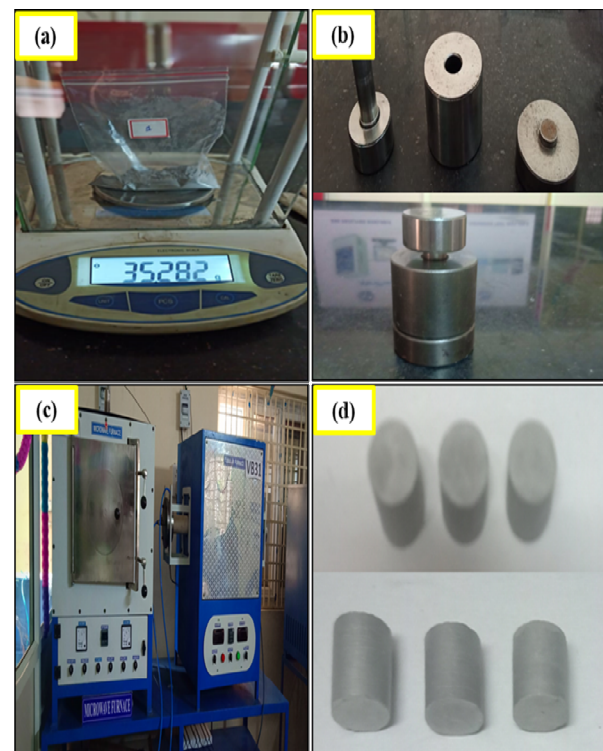

Figure 2. Photographs of different devices/tools used to produce of Al/Graphene/CNT composites (a) weighing balance, (b) compaction die, (c) tubular furnace for sintering and (d) sintered hybrid composite samples.

The wear tests were done as per ASTM standard G99 using pin on disc test apparatus. The samples used for wear testing were having dimensions of Ø 6 mm x 30 mm. Three loads of 10 N, 20 N, and 30 N with fixed sliding velocity of 2.0 m/sec and sliding distance of 1200 m were selected for conducting sliding wear experiments. The countersurface used is alloy steel disc of with HRc 60 in the heat-treated condition. The samples were properly cleaned, dried and measured during each test. A microbalance has been used to weigh the samples. Weight loss was converted into volume loss per unit of sliding distance to calculate wear rate.

All the tests were conducted at room temperature and average of three tests specimen for each testing condition is presented here.

3. RESULTS AND DISCUSSION

3.1 Microstructure and density

Fig. 3 (a) and (b) shows the SEM micrographs of aluminium hybrid composites with 1 wt% and 3 wt% graphene content. Both the hybrid composites showed dense structure with clear grain boundaries and minimal pores. It is found that graphene is located at the grain boundaries of aluminium grains. At few places some clusters are also seen. However the clustering of graphene is comparatively high in hybrid composite containing 5 wt% graphene. With increase in content the clustering explains that this is critical volume fraction above which any addition will be redundant. The clustering can be attributed to π-π interactions and strong van der Waals attraction [20,21].
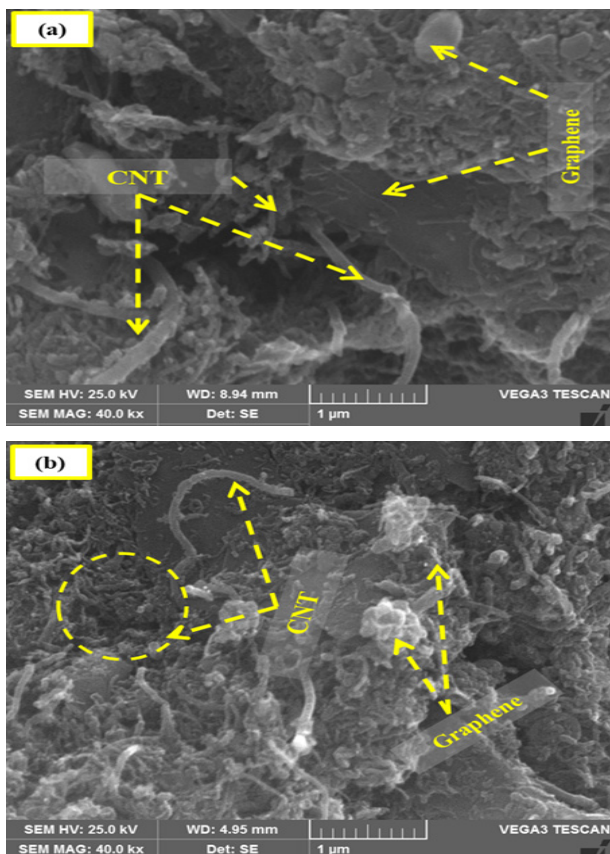

Figure 3. SEM micrographs of Al hybrid composites with (a) 1 wt% and (b) 3 wt% graphene content.

Fig. 4 shows the density values of Al/Graphene/CNT hybrid composites after sintering. It is seen from this figure that pure aluminium has shown highest density of 2.5892 g/cm^3. With the addition of graphene and CNT, the density of hybrid composites tend to decrease. For instance, B1 composite showed decrease in density by 13.56% compared to AL. Similarly, C1, D1, E1 and F1 composites showed decrease in density values by 12.89%, 16.25%, 17.99% and 18.20%, respectively over AL. This decrease in density values is due to addition of graphene and CNTs which have density values of 2.26 and 2.1 g/cm^3 respectively. The addition of low density reinforcements led to aluminium decrease in the density of resulting hybrid composites.
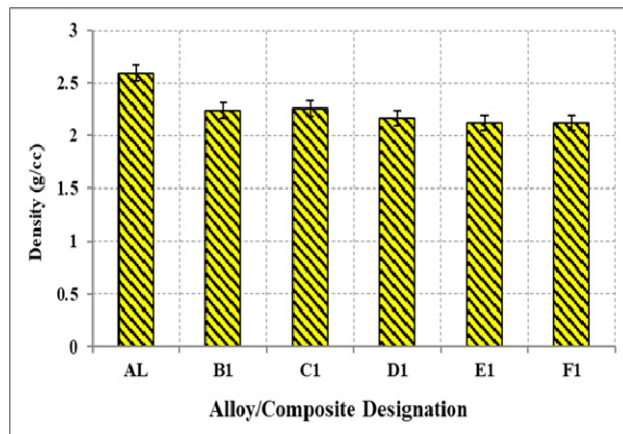

Figure 4. Density values of unreinforced and graphene/CNT reinforced aluminium hybrid composites.

3.2 Hardness

The bulk hardness of Al/Graphene/CNT composites is shown in Fig. 5. It is evident that hardness increases after addition of graphene and CNT. Further increase was observed when the graphene content was increased from 1 wt% to 3 wt%. B1composite shows an increase in HRA by 8.58%, while C1, D1, E1 and F1 composites exhibit an increase by 14.93%, 20.95%, 27.29% and 23.38%, respectively, when compared to AL. The hardness was found up to E1 composite and thereafter there is a slight dip in the hardness value for F1 composite by about 2.68% compared to E1 composite. The reason may be attributed to the fact that increased wt% of graphene may result in agglomeration of in F1 thus leading to decrease in the hardness value. In their work, Varol and Canakci [22] found that due to soft nature of graphene, they tend to agglomerate at the grain boundaries of copper grains. Due to this the Brinell hardness of nanocomposite was found to decrease. Bobic et al. [23] developed hybrid A356/SiCp/Grp composites via compo-casting and investigated effect of microstructure and mechanical properties i.e., phases on hardness of the composites. Also, Vencl et al.[24] analysed the effect of SiC and graphite particles i.e., secondary phases in the A356 MMCs on mechanical properties by means of macro and nanoscales and found that they had a beneficial effect on all the mechanical properties examined. However, in the present work it is quite clear from the results that both graphene and CNTs are contibuting to enhancement in hardness of hybrid

composites. Improvement in hardness can be attributed to the effect emanating from thermal mismatch and grain refinement. The dislocations are generated due to difference in coefficient of thermal expansion mismatch between Al ($\sim$23.6$\times$10^{-6} /K), graphene ($\sim$1.0$\times$10^{-6} /K) and CNTs ($\sim$1.0$\times$10^{-6} /K).With the increase in graphene content the dislocation density could also increase. On the other hand the presence of graphene and CNTs can effectively pin the grain boundaries by inhibiting their motion during sintering. The higher the graphene content the possibility of grain refinement enhances leading to fine grain sizes of aluminium. This will eventually help in impeding the dislocation motion which in turn increases hardness of hybrid composites [25,26].
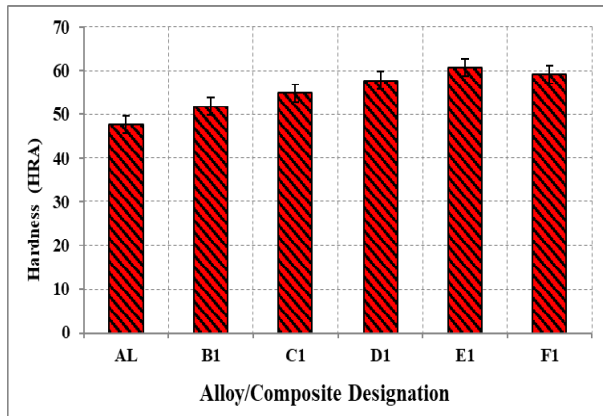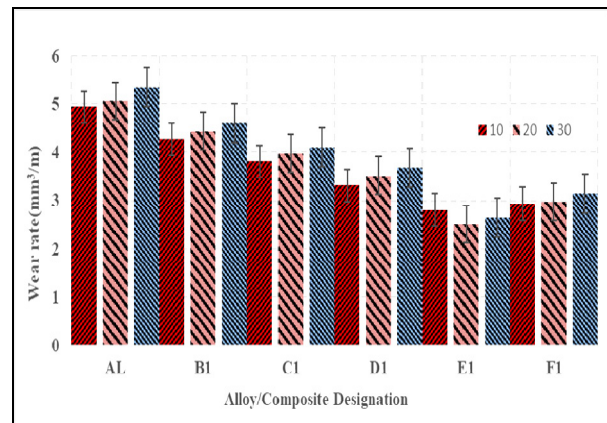

Figure 5. Hardness of unreinforced and graphene/CNT reinforced aluminium hybrid composites.

3.3 Wear behaviour

Fig. 6 shows the wear rate of Al/Graphene/CNT hybrid composites for three different loads 10, 20 and 30 N and sliding distance of 1200 m. The high sliding speed was chosen as a result of the experiment carried out by several researchers [27,28]. Also, as the sliding velocity increases to certain values, the wear rate decreases due to the forming of a mechanically mixed layer (MML) between contact surfaces [29-32].

It is observed that wear rate tend to increase with the increase in load from 10 N to 30 N. For instance, AL sample showed wear rate values of 4.95$\times$10^{-3}, 5.0$\times$10^{-3} and 5.30$\times$10^{-3} mm^3/m, respectively, for 10, 20 and 30 N. From the load point of view, the increase in load helps in increased penetration of countersurface asperities into the surface of AL and its hybrid composites. This increases the material removal rate in all materials especially for unreinforced AL. This observation is well in line with the Archard principle which states that the increase in load tends to increase wear rate. With the addition of 1wt% of graphene and 2% wt CNT, the wear rate of C1 were 3.70$\times$10^{-3}, 3.95$\times$10^{-3} and 4.10$\times$10^{-3} mm^3/m respectively for 10, 20 and 30 N. The wear rate values for C1, D1, E1 and F1 were found to decrease significantly after addition of both graphene and CNT. Analogous results have been published by Vencl et al. [33-35] and several other investigators [36-38]. From the reinforcement point of view the decrease in wear rate of hybrid composites is attributed to main reasons, one is increase in hardness of hybrid composites over AL, load bearing effect and lubricating nature of

graphene and CNTs. The material hardness plays an important role in determining the material loss during in contact with other surface in motion. So, it is a common notion that harder the material higher is the wear resistance. In case of metals like aluminium, they are quite soft in nature and tend to wear out easily. Addition of graphene and CNTs tends to increase the hardness of soft and ductile aluminium matrix. Due to this, the resistance of aluminium matrix tends to increase resulting in low wear rate compared to AL [38]. On the other hand, the high strength graphene and CNTs strengthen the aluminium matrix due to which it is capable of withstanding the applied normal load and resist bending load. Due to this load bearing nature, the wear rate of hybrid composites is minimized. The lubricating nature of graphene and CNT also contribute to reduced wear rate. During sliding of two contact surfaces both graphene and CNT tend to tear and unwrap leading to the formation of lubricating layer. In addition to this the debris containing smeared graphene and CNT formed bridges the subsurface gap, thereby reducing the wear rate [39].

Figure 6. Wear rate of unreinforced and graphene/CNT reinforced aluminium hybrid composites for varying load.

3.4 Worn surface analysis

Fig. 7 (a) – (f) shows the worn surface of AL and its hybrid composites taken at 30 N load, sliding velocity of 2 m/s and sliding distance of 1200 m. In case of AL, the worn surface as seen in Fig. 7 (a) showed delaminated patches and deep scratches. The soft nature of AL and deformation due to applied load causes subsurface crack formation. Due to this crack propagates the weak region leading to debonding of material in the form of large flakes. In addition to this presence lot of white particles does imply the oxidation taking place. Most of the white spots are seen in the delaminated region. This implies that at higher load of 30 N, the predominant mechanism operating are adhesion and oxidation followed by slight abrasion. As shown in Fig. 7 (b) to (f), the worn surface showed scratches with minimal delamination. With the addition of graphene and CNT the extent of delamination is found to be reduced significantly. Fig. 7 (b) showed that the B1 composite containing only CNTs exhibited slight delamination but large number of scratches. Due to increase in hardness the composite showed utmost resistance to delamination. Here abrasion was dominant wear mechanism followed by adhesion.
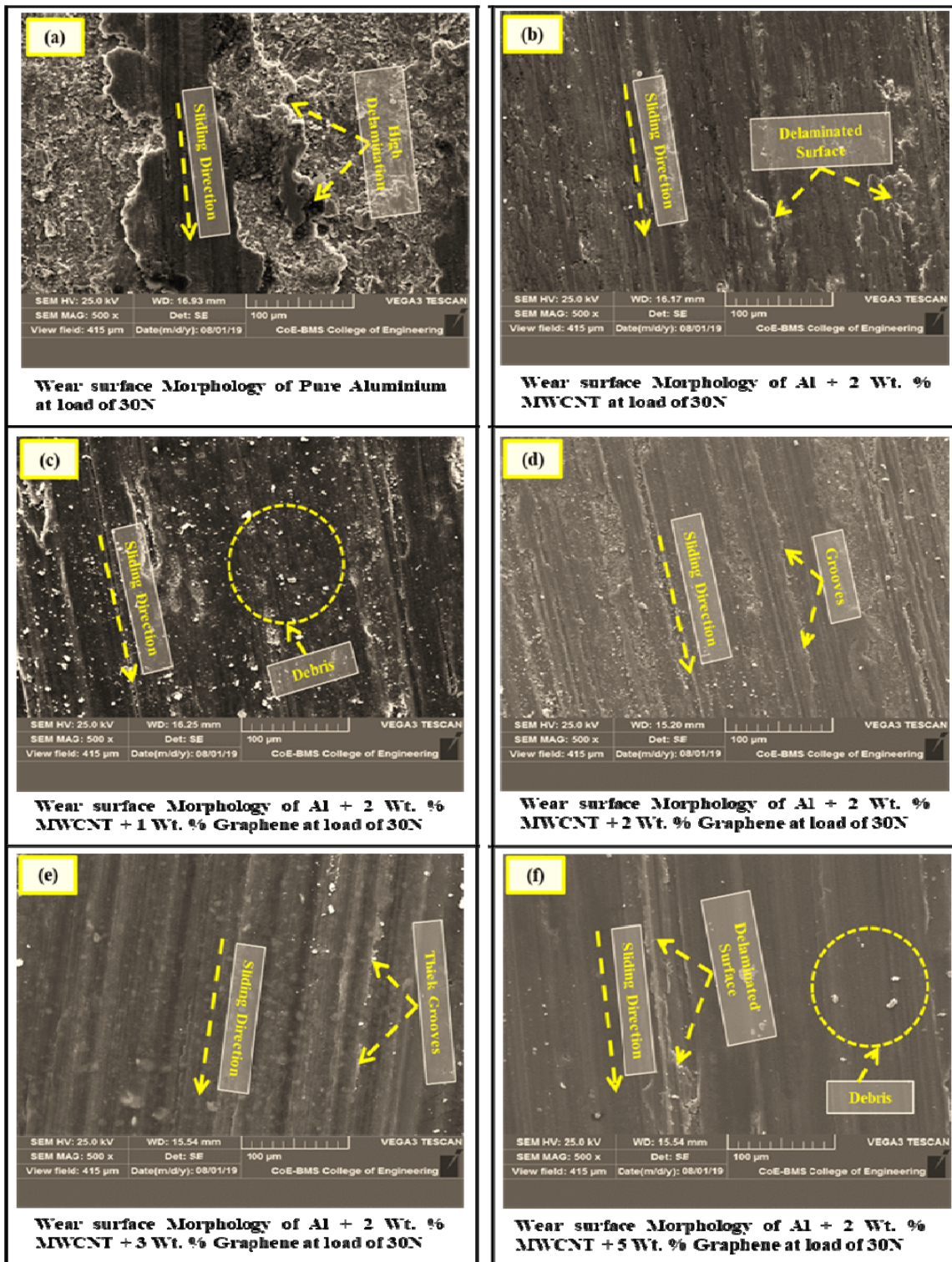
Figure 7. Worn surface of different aluminium and its hybrid composites at sliding velocity of 2 m/s, sliding distance of 1200 m constant and load of 30N.

In the case of hybrid composites, the extent of delamination was minimized by both graphene and CNTs. All hybrid composites showed minimal delamination along with narrow scratches. The width and depth of scratches tend to decrease with the increase in graphene content. The hybrid composites E1 and F1 had near smooth surface due to their high hardness when compared to AL and its other counterparts. As mentioned earlier both graphene and CNTs tends to act as solid lubricant. During the course of sliding wear the tearing and worn out of reinforcements

form a lubricating layer on the surface of hybrid composites. This layer tends to reduce the direct contact between the composite surfaces and countersurface. In addition to this hard hybrid composite surface posed significant resistance to plastic deformation due to which the scratch depth and width tend to decrease. This is why the extent of adhesion is reduced significantly and scratches got narrower as evident from the worn surfaces. Overall hybrid composites showed abrasion as dominant wear mechanism. This observation is well supported by the

work reported by Bastwros et al. [40] where they found abrasion as main wear mechanism for Al/CNT composites. Further it is interesting to note that the extent of oxidation was also less in hybrid composites. This is mainly because, in case of AL the soft surface tends to delaminate easily exposing the fresh surface which in due course of testing tend to react with oxygen present is atmosphere and form oxidation products. However, in the case of hybrid composites the extent of delamination is greatly reduced due to high hardness and presence of lubricating layer. Hence no fresh surface is exposed to atmosphere and no oxidation products are seen on the worn surface.

In order to confirm the oxidation taking place in Al and formation of lubricating layer in hybrid composites, EDS analysis was carried out and is shown in Fig. 8 (a) and (b). The contribution of oxidative wear is confirmed by the presence of oxygen element in the EDS analysis. In the case of hybrid composites, the EDS showed large peaks pertaining to carbon and aluminium. These results are in good agreement with the observed wear rate values and those reported in the literature [41].

4. CONCLUSION

In the present work an attempt was made to develop Al/Graphene/CNT hybrid composites and study there wear behaviour. The conclusions drawn from this work are as follows,

i. The hardness of hybrid composites was found to be higher than that of unreinforced aluminium. The increase in hardness was attributed to grain refinement and difference in thermal expansion coefficient.

ii. The wear rate was found to decrease significantly after addition of graphene and CNTs. The increase in hardness and lubricating property of reinforcements was found to be major contributors for increased wear resistance.

iii. The wear mechanism was adhesion and oxidative wear for unreinforced aluminium while for hybrid composites it was abrasion.
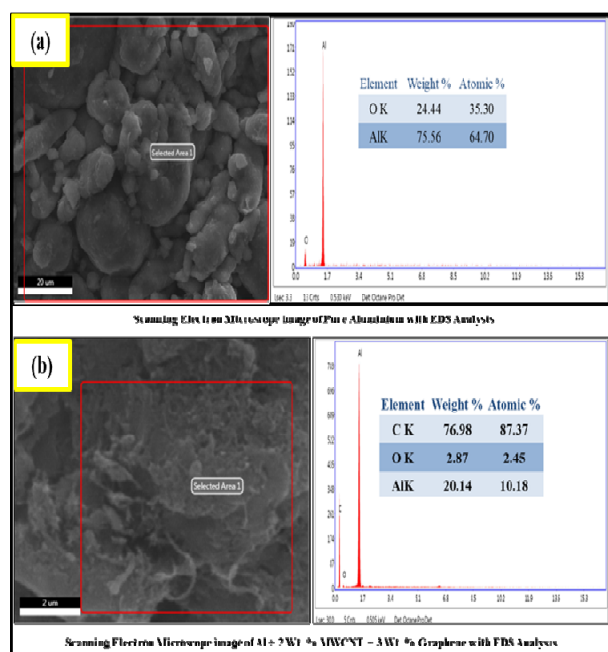

Figure 8. EDS image of (a) AL and (b) E1.

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ХИБРИДНИ КОМПОЗИТИ НА БАЗИ АЛУМИ-НИЈУМА, ГРАФЕНА И УГЉЕНИЧНИХ НАНОЦЕВИ: ИСПИТИВАЊЕ ТВРДОЋЕ И ХАБАЊА ПРИ КЛИЗАЊУ

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Графен и угљеничне наноцеви су два материјала на бази угљеника позната по јединственим својствима хабања и трења. Од интереса је разумевање понашања хибридних композита на бази алуминијума у условима хабања када се наночестице ова два материјала користе за ојачање композита. Техником прашкасте металургије произведени су хибридни композити са променљивим тежинским уделом графена и стабилним садржајем угљеничних наноцеви. Испитан је утицај променљивог садржаја графена на тврдоћу и хабање при клизању код хибридних композита. Ипитивање хабања је обављено применом стандарда ASTM G-99 при константној брзини хабања (2м/сец) и растојању код клизања (1200 м) али променљивом оптерећењу (10 – 20 N). Истрошене површине су анализиране скенирајућим микроскопом да би се открили механизми хабања одговорни за трошење алуминијума и хибридних композита. Пораст садржаја графена довео је до пораста запреминске тврдоће (највећа вредност: 61 RHN код хибридног композита, садржај графена: 3 теж.%). Утврђено је да брзина хабања опада са повећањем садржаја графена. Нижа вредност брзине хабања је резултат стварања филма од мазива на истрошеној површини.