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Wear behavior of Aluminium 6061 alloy reinforced with coated/uncoated multiwalled carbon nanotube and graphene

Vijee KUMAR[1], Kempaiah UJJAINI NAGEGOWDA[2], Satish Babu BOPPANA[3], Ramesh SENGOTTUVELU[3], and Palanikumar KAYAROGANAM[4,*]

[1] *Visvesvaraya Technological University, VTU Main Road, Machhe, Belgaum, Karnataka, 590018, India*
[2] *University Visvesvaraya College of Engineering, KR Circle, Devraj Urs Road, Bengaluru, Karnataka, 560001, India*
[3] *School of Engineering, Presidency University, Itgalpur, Rajanakunte, Bengaluru, Karnataka, 560064, India*
[4] *Sri Sairam Institute of Technology, Sairam College Road, Nagar, Tambavaram West, Chennai, Tamil Nadu, 600064, India*

***Corresponding author e-mail:** palanikumar@sairamit.edu.in

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Abstract

The current study deals with the fabrication and investigation of wear characteristics of Aluminium 6061(Al6061) hybrid metal matrix composites (MMCs) processed through powder metallurgy technique. Al6061 hybrid MMCs involving fixed 2 wt% of coated/uncoated multiwalled carbon nanotubes (MWCNTs) and varying weight percentages of graphene were fabricated through ball milling procedure. To enhance the scattering of MWCNTs in the matrix, MWCNTs were coated by means of copper through electroless deposition method. Dry sliding wear conduct of Al6061 MMCs was investigated using a pin-on-disc wear testing machine. It was found that at lower load, composites exhibited lower wear resistance than base alloy however at higher load, nanocomposites showed higher wear resistance. The research tried to find the effect of higher loads on the wear resistance. The composites were evaluated if they could give out reinforcements at higher loads during wear tests. The wear morphologies were reported using Scanning Electron Microscopy (SEM) and it was noticed that at lower load abrasion was superior for the composites and base alloy although at higher loads adhesion was considered to be main reason for the wear of composites.

1. Introduction

Aluminium alloys are the most extensively applicable materials in engineering fields due to its excellent properties like minimal coefficient of thermal expansion, elevated tensile strength as well as light weight [1]. However, owing to their low hardness and wear resistance their applications were however restricted [2]. Since last two decades, effort and attention has been given towards the progress of Aluminium Metal Matrix Composites (AMMC) [3-5]. AMMCs containing nanoparticulates reinforcements are attributed as capable solution to improve wear resistance to aluminum alloys [6].

AMMCs reinforced with nanoparticles such as nanotubes and graphene are found to be predominant in applications pertaining to fields like automobiles, marine, space crafts and other engineering structural applications [7]. Carbon nanotubes (CNTs) have better mechanical strength up to 1 TPa; these extraordinary properties of CNTs made them a good candidate reinforcement material for Al alloy composites [8,9]. The final desired mechanical and tribological attributes of CNTs-AMMCs rely on dispersion of CNTs in matrix phase and area matter of primary concern. CNTs in MMCs is an immense challenge owing to their agglomeration and meager scattering of the nanotubes [10]. Several methodologies have been available to manufacture MMC materials but, many researchers have reported powder metallurgy technique followed by ball milling to be the best method for synthesizing CNTs-metal matrix composites [11]. Al-Qutub *et al.* [2] have studied the wear properties and abrasion of Al6061 base alloy strengthened with 1 wt% CNT composites and indicated that the CNT content plays a major role. Choi *et al.* [12] have investigated on the mechanical and wear properties of nano based matrix of Al and revealed that MWCNTs addition appreciably improved and resulted in lower coefficient of friction probably owing to reduction in grain size and accumulation of CNTs. Manikandan *et al.* [13] deliberated on the wear performance of pure aluminium with 0.5, 1.0, 1.5 and 2 wt% of CNTs composites and revealed that the friction conduct of CNTs reinforced composites was notably reliant on the functional load. Zhou *et al.* [14] reported on the Al-CNTs composites fabricated via pressureless infiltration method and inferred that the wear loss was vastly dependent on the technique used for the distribution of CNTs. In the recent years, researchers have focused on nanomaterials. Graphene is one such new material in carbon grouping. It is a sp^2 – 2D-nanoscale matter that is well accepted with tremendous strength. It has received enormous interest owing to its outstanding mechanical and physical properties [15,16].

From the available literature, it has been noted that the dual particle reinforcement based composite is very limited. The use of carbon nanotubes and graphene for manufacturing of metal matrix composites is novel and new. A systematic analysis on the wear properties for these composites also limited and hence this study is carried out. In the current research, a novel technique is used to prepare composites; MWCNTs were initially coated with copper to improve the dispersion in the base alloy. The nanocomposites were prepared by adding 2 wt% of MWCNTs (coated/uncoated) while varying the percentage of graphene (0.5 wt% and 1 wt%). The composites were then evaluated to know the effect of addition of coated/uncoated MWCNTs and graphene on wear behavior of Al alloy composite using pin-on-disc wear tester.

2. Experimental procedure

2.1 Materials

Al6061 alloy in the form of powder was used as the matrix phase in the study, supplied from Defense Metallurgical Research Laboratory (DMRL), Hyderabad, particle size average was 300 mesh with a density of 2.7 g·cm^{-3}, Figure 1 shows the micrograph of Al6061 powder obtained through SEM (Model: TESCAN VEGA 3).

In order to characterize the specimen through microstructural studies, the specimen is cut using a hacksaw and initially filed to remove any burs and scratches. It is then subjected to polishing using a series of emery sheets and disc polishing machine until a mirror finish is obtained.

For generation of electrons in SEM, a tungsten filament is used. Ultra-high vacuum conditions are used to make sure that the tip is always free from contaminants. Using a high voltage system, the beam is then accelerated through the apertures and lens. The beam is then allowed to scan the specimen surface. Signals are generated to produce SEM images through the interaction between beam and specimen.

The MWCNTs were procured from NoPo Nanotechnologies India Private Limited, Bengaluru, and its properties are shown in Table 1.

Figure 2 shows the TEM image of MWCNTs and Figure 3 shows the FESEM image of graphene as received.

Graphene (technical grade) was procured from United Nanotech Innovation Pvt. Ltd., Bengaluru. The density of the graphene is 2.20 g·cm^{-3} with a thickness 10-12 nm and had layers from 16-20.

Table 1. Properties of MWCNTs.

Properties	**Value**
Outer diameter	30-40 nm
Length	30-50 μm
Purity	>95%
Ash	<1.5 wt%
Density	2.1 g·cm^{-3}

Table 2. Chemical composition of copper bath [17].

Chemical composition	**Quantity (g·l^{-1})**
Copper sulfate-5H_2O	40
EDTA Disodium salt	48
HCHO (add after 10 min)	25
Na_2SO_4 -10 H_2O	35
HCOONa	15
Polyethyleneglycol	6.6
PH (adjust with NaOH)	12-13

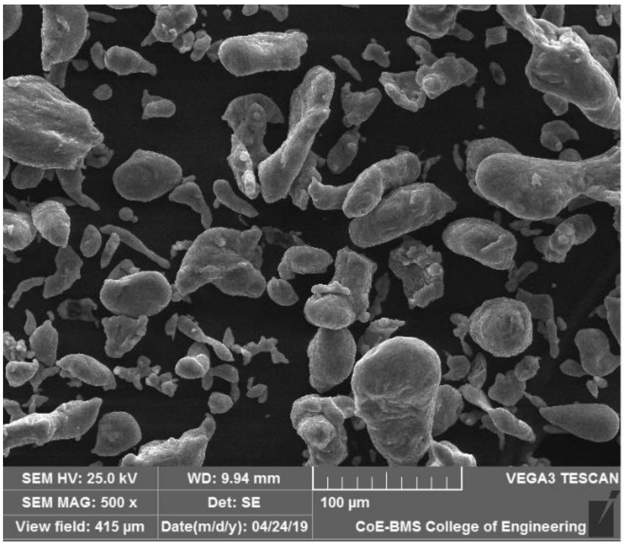

Figure 1. SEM micrograph of Al6061 powder.

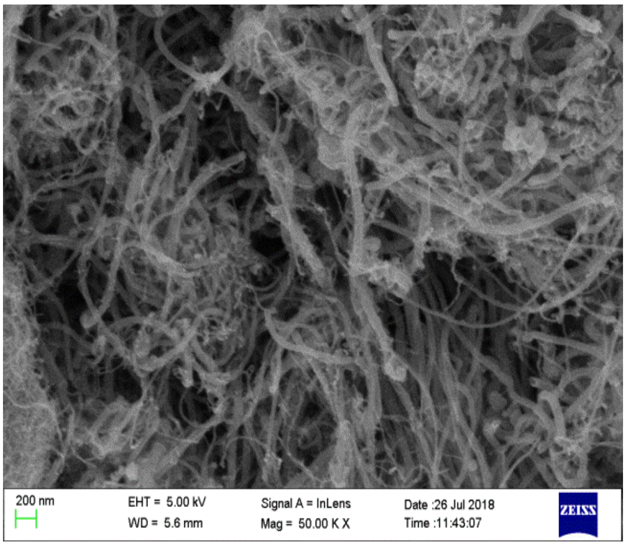

Figure 2. Nanometer scale image of MWCNTs.

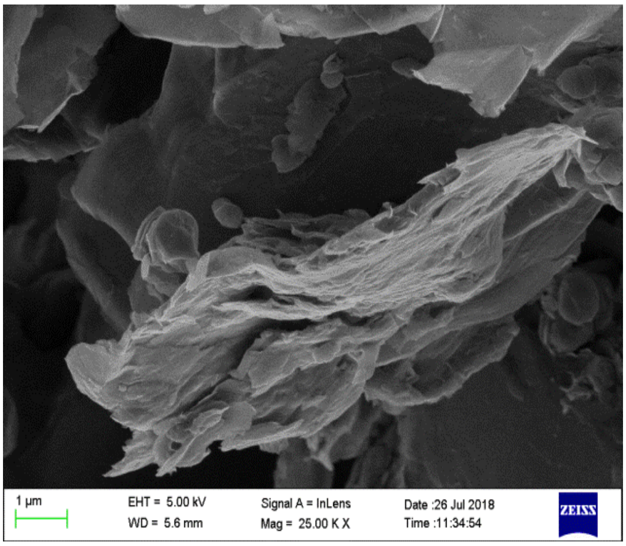

Figure 3. FESEM image of graphene.

Table 3. Sample preparations with different weight percentages.

Sample	Al6061	MWCNTs	Graphene
Al6061	100%	0%	0%
Al6061-2 % uncoated MWCNT	98%	2% (uncoated)	0%
Al6061-2 % coated MWCNT	98%	2% (coated)	0%
Al6061-0.5 % G	99.5%	0%	0.5%
Al6061-1 % G	99%	0%	1%
Al6061-2 % uncoated MWCNT-0.5 % G	97.5%	2% (uncoated)	0.5%
Al6061-2 % coated MWCNT-0.5 % G	97.5%	2% (coated)	0.5%
Al6061-2 % uncoated MWCNT-1 % G	97%	2% (uncoated)	1%
Al6061-2 % coated MWCNT-1 % G	97%	2% (coated)	1%

2.2 Electroless copper coating to MWCNTs

The purified MWCNTs were subjected to copper coating by electroless deposition. In order to achieve uniform copper coating thickness on MWCNTs, oxidization, sensitization, activation and decoration of copper was carefully carried out. Purified MWCNTs were oxidized with H_2SO_4 (98%) and HNO_3 (70%) at 140°C for 8 h and tubes were cleaned using millipore water and desiccated at 120°C. The oxidized MWCNTs were then subjected to sensitization followed by activation using stannous chloride ($SnCl_2$)/hydrochloric acid (HCl), later the tubes were activated by palladium chloride ($PdCl_2$)/HCl solution by magnetic stirring for 40 min. The activated MWCNTs were subjected to electroless copper coating for 30 min in an electroless copper bath at a pH value 12±0.7 for uniform deposition of copper. The copper bath composition of the chemicals used for study is revealed in Table 2.

2.3 Preparation of nanocomposites

In this study, Al6061alloy powder, 2 wt% MWCNTs (coated and uncoated) and 0.5/1 wt% graphene were mixed through ball milling to disperse MWCNTs/graphene in Al6061matrix. The milling time was maintained at 2 h with 200 rpm speed while the process control agent used is ethanol. The ratio of ball to powder of 10:1 [18,19] was used during the mixing process. The samples were compacted in hardened steel die-punch. The powder samples were initially pre heated and compacted with a compaction load of 90-110 kN; compacts of 20 mm diameter and 30 mm length were thus prepared. The green compacted samples were subjected to sintering at 580°C with a dwell time of 60 min in a conventional furnace with nitrogen atmosphere. Table 3, shows the compositions of nanocomposites samples prepared.

2.4 Wear studies

Dry sliding wear tests were performed on Al6061 base alloy and Al6061-MWCNTs (coated/uncoated)-graphene composites by means of a pin-on-disc wear tester. The dry sliding condition relates to the friction at the interface between pin and disc counter face when no lubrication exists at the point of contact between the two said surfaces. This is practically done to know the effect of such dry friction in specific applications like antifriction bearings, which operate without a liquid lubricant and experience high wear and friction.

The counter surface of the disc was made up of 201 stainless steel. Pin size of 8 mm diameter and 30 mm length were carefully placed in vertical direction. Before testing, the specimen polishing is carried out using abrasive grit size of 400, 600, 800 and 1000. The test environment was kept at room temperature [20]. After each trail of test, the specimen weight was precisely recorded using an electronic weighing balance having the accurateness of ±0.01 mg. The test was conducted under normal loads of 20, 30 and 40 N, at a fixed time period of 10 min, speed of 300 rpm and track diameter of 150 mm. Test was also carried out by varying speed settings of 100, 200 and 300 rpm for a fixed time of 10 min, load of 30 N and track diameter 150 mm. The data was recorded for wear rate through weight loss means. Worn surface micrographs were investigated based on the SEM observations of wear tracks and wear debris on base alloy and nanocomposites.

2.5 Hardness

Vickers Hardness testing machine was used to evaluate the microhardness of the samples prepared. The hardness value was selected as the average of 3 samples, while the hardness of each sample was found from at least 10 indents at a load of 40 gf.

3. Results and discussion

3.1 Electroless copper coating of MWCNTs

Al6061/MWCNTs composites fabricated through powder metallurgy process had consistent distribution and strong interfacial bonding of MWCNTs with alloy powder and were reasoned to be achieved through electroless coating. A tough interfacial bonding amid MWCNTs and metal powders is possible through the chemical response of MWCNTs with ions on the metal. Hence, MWCNTs were decorated with copper particles. The morphology of coated MWCNTs and EDS are shown in Figure 4(a), (b) and (c).

Figure 4(a) exhibits the FESEM images of coated MWCNTs, Figure 4(b) and (c) shows SEM image and corresponding EDS studies reveal the presence of copper on copper coated MWCNTs. The elemental Table 4 also shows the presence of Cu element.

The reinforcements and matrix used are in nanosizes. There is no evidence of insitu formation of any new phases as evidenced from XRD. Hence, the composite is considered to be a nanocomposite.

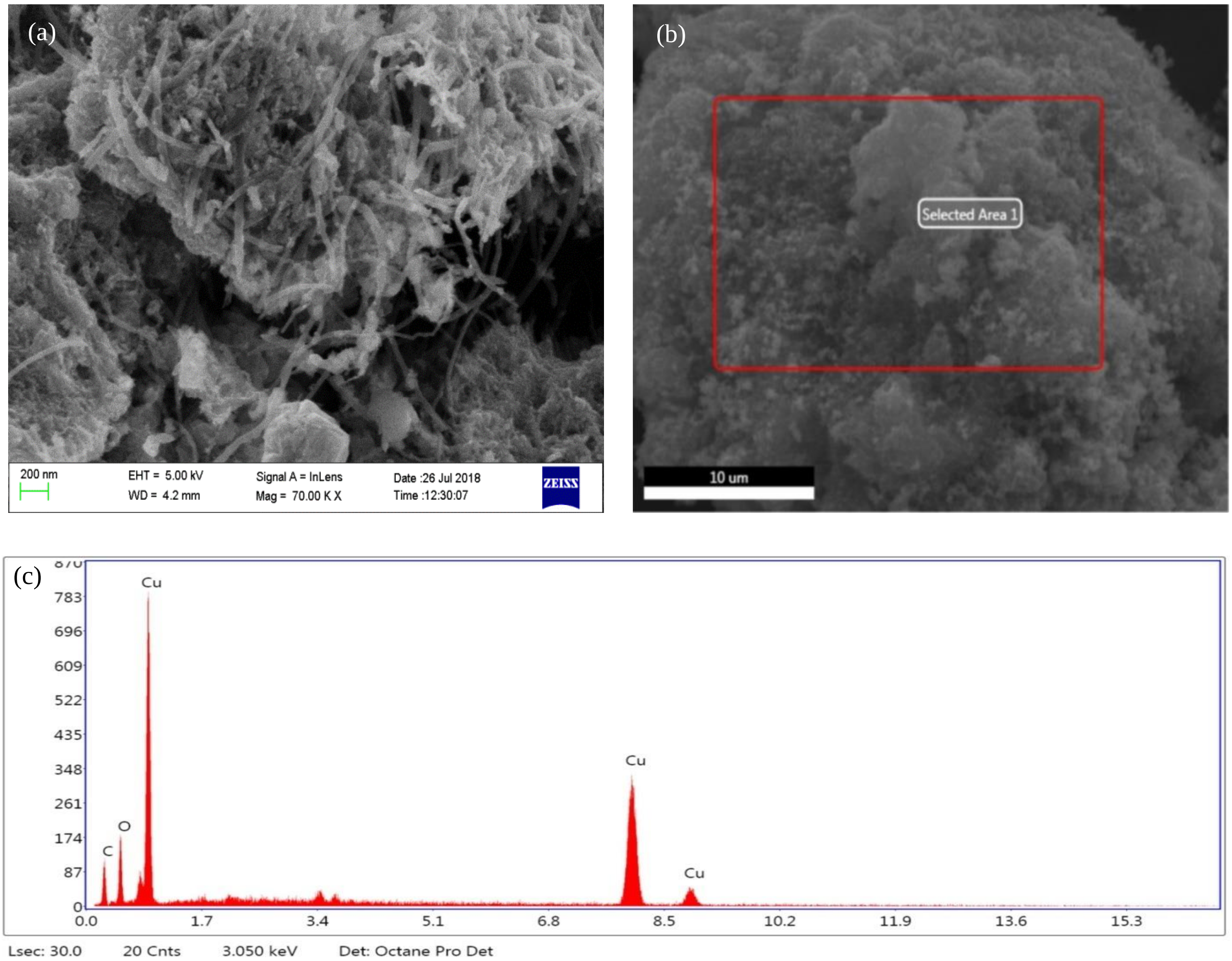

Figure 4. (a) FESEM image of coated MWCNTs, (b) SEM image of Cu-coated MWCNTs, and (c) EDS of Cu-coated MWCNTs.

Table 4. Elemental table for Cu-coated MWCNTs.

Elements	Weight %	Atomic %
C	28.06	53.62
O	20.23	29.44
Cu	51.71	17.79

3.2 Wear behavior of materials

Weight loss of Al6061 matrix material and composite with 2 wt% of MWCNTs (coated/uncoated) reinforcement at different loads of 20, 30 and 40 N and at a constant speed 300 rpm, track radius 150 mm, time of 10min are presented in Figure 5.

It was observed that weight loss for 2 wt% MWCNTs (coated/uncoated) with Al6061 samples exhibited lower weight loss at relatively lower loads from 20-30 N; though, at elevated loads from 30-40 N, the weight loss is recorded was quite high. The weight loss with respect to the varying graphene content in the composites as a relation with varying load is illustrated in Figure 6.

The rate of wear improved in relation to applied load. From Figure 6, it can be clearly seen that at higher loads, graphene (1 wt%) strengthened with Al6061 composite showed higher weight loss than 0.5 wt% graphene reinforced composite and base Al6061 alloy, due to uneven dispersion of graphene and lack of strength between matrix and reinforcement.

The weight loss of hybrid nanocomposite containing 2 wt% MWCNTs (coated/uncoated) and 0.5 wt% and 1 wt% graphene with respect to varying load is illustrated in Figure 7.

Weight loss improved linearly; at minor loads of 20-30 N, the composite portrayed better wear obstruction. Nevertheless, at elevated loads [23] of 30-40 N, the weight loss is found to be high for hybrid nanocomposites compared to Al6061 alloy as provided in Figure 7. Al6061-coated MWCNTs-graphene nanocomposites shows improved wear resistance than uncoated MWCNT reinforced nanocomposites. The coated MWCNTs reinforced composite samples at lower load condition exhibited mild wear conditions; this can be ascribed to the elevated hardness [24-26] associated with MWCNTs/graphene and also attributed to the strong interfacial bond linking the coated MWCNTs/graphene and Al6061 matrix material. At superior loads (extreme conditions), pores were seen in the combination of alloy and reinforcement due to sintering of samples and possible damages caused to MWCNTs (coated/uncoated). MWCNTs agglomerates are also a cause for resulting in poor wear resistance. Hence, at transition load (lower load) hybrid composites showed better wear resistance than Al6061 alloy but at higher load uncoated MWCNTs have negative effect on wear resistance compared to coated nanotube reinforced composites.

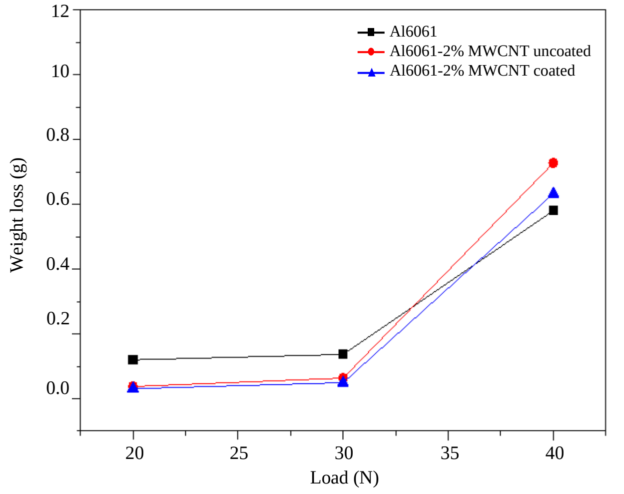

Figure 5. Effect of varying load v/s weight loss for Al6061 alloy, Al6061-2 wt% MWCNTs (coated/uncoated) samples**.**

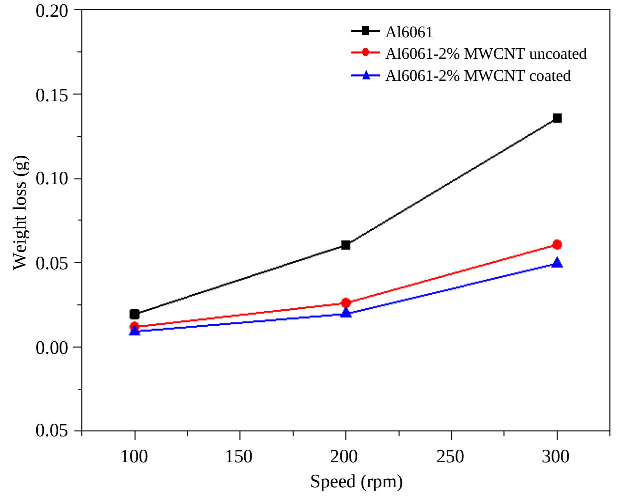

Figure 8. Effect of varying speed v/s weight loss for Al6061 alloy, Al6061-2 wt% MWCNTs (coated/uncoated) nanocomposite samples.

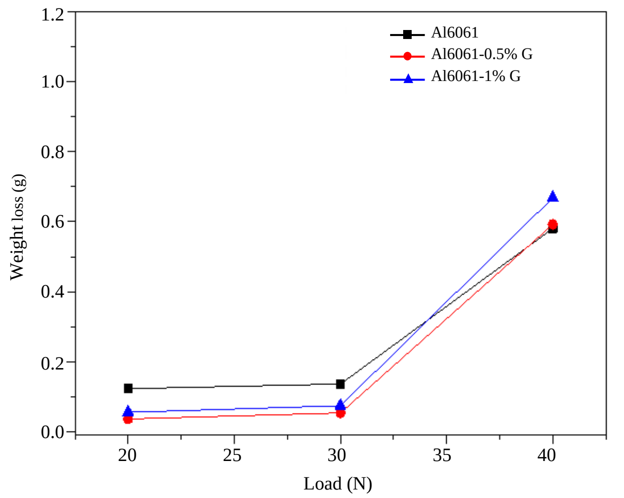

Figure 6. Effect of varying load v/s weight loss of Al6061 alloy, Al6061-0.5 wt% graphene, Al6061-1 wt% graphene samples.

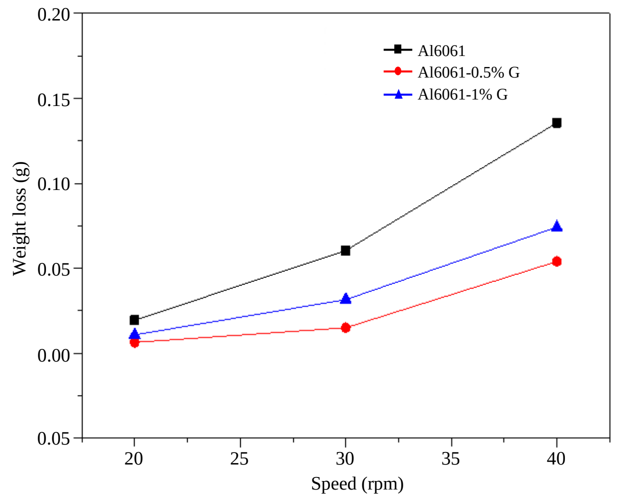

Figure 9. Effect of varying speed v/s weight loss for Al6061 alloy, Al6061 -0.5 wt% and 1 wt% graphene nanocomposites samples.

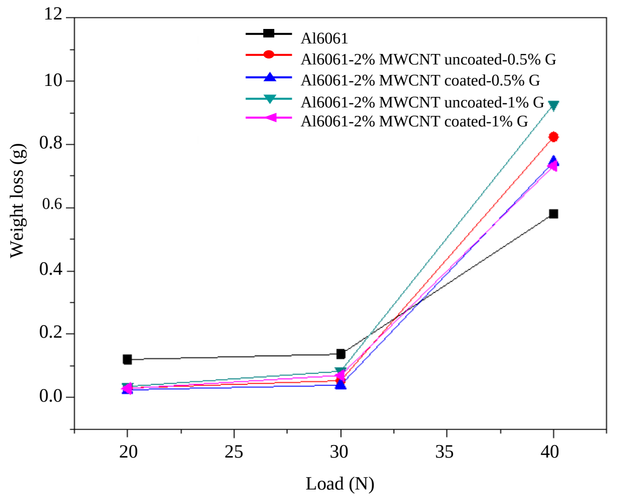

Figure 7. Effect of varying load v/s weight loss for Al6061 alloy, Al6061-2 wt% MWCNTs (coated/uncoated)-0.5 wt% graphene and Al6061-2 wt% MWCNTs (coated/uncoated)-1 wt% graphene samples.

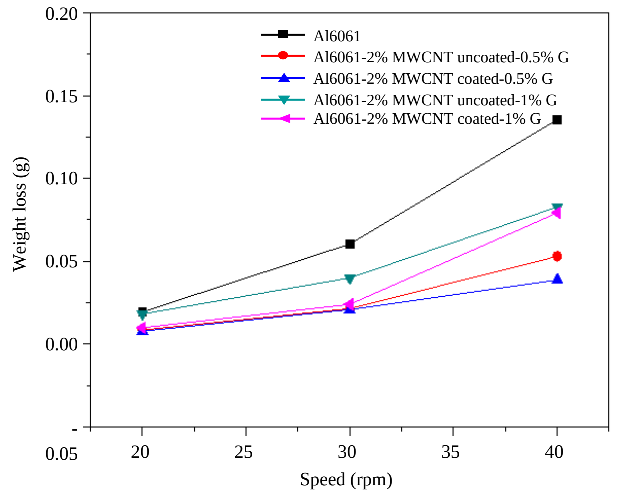

Figure 10. Effect of varying speed v/s weight loss for Al6061 alloy, Al6061-2 wt% MWNTs (coated/uncoated)-0.5 wt% graphene and Al6061-2 wt% MWCNTs (coated/uncoated)-1 wt% graphene samples for varying speed of 100, 200 and 300 rpm.

The research reveals that the composite offered good resistance until 30 N. However, upon increasing the load to 40 N, the weight loss was abrupt and could be due to the limitations of sintering process. The pores would further open up thereby exposing the reinforcement and leading to additional wear due to abrasion and adhesion. Furrows would form that would also add up to removal of layers in the advanced stages of wear due to friction.

The varying speed on weight loss of Al6061 matrix material and nanocomposites at different speeds of 100, 200 and 300 rpm at a constant load of 30 N, track radius 150 mm with a time period of 10 min was studied. Figure 8 illustrates the wear of Al6061 strengthened with 2 wt% MWCNTs (coated/uncoated).

It is reflected from Figure 8, that, weight loss linearly improved with increasing speed and coated MWCNTs reinforced composites had displayed higher wear resistance than Al6061 base alloy and uncoated MWCNTs composite at a load of 30 N.

It is seen from the Figure 9, that Al6061-0.5 wt% graphene composites have exhibited less weight loss than Al6061-1 wt% graphene composites at a load of 30 N. Further, it is concluded that there is noteworthy decrease in the volumetric weight loss, this is evident due to lubricating nature of graphene.

Figure 10, clearly exhibits the deviation of weight loss with varying percentage of graphene reinforcement at a load of 30 N.

It is observed that 0.5 wt% graphene-coated MWCNTs reinforced with Al6061 composite exhibited lesser weight loss when contrasted with 1 wt% graphene-(coated/uncoated) MWCNTs reinforced composites. It is noticed that, MWCNTs coated hybrid nanocomposites exhibited less weight loss than that of Al6061 alloy. The presence of graphene in the Al6061 composite influences the wear distribution owing to the incidence of graphene on the adjoining surfaces all through the sliding, graphene behaves as a solid lubricant which is responsible for decrease in weight loss. This is also attributed to the excellent bonding between MWCNTs/graphene and Al6061 matrix. From the analysis of figures, it has been noticed that the friction and subsequent wear loss is due to the application of load and variation of the speed. The application of the load with respect to the wear loss indicates that the wear loss is uniform upto 30 N load, whereas it increases with the increase of load. The wear loss is due to the friction which induces between the pin and disc, the results indicate that the friction is almost stable, but due to the inclusion of MWCNT and other particles, there is small deviations occur in the frictional force, which leads to the wear. The speed also indicates, almost the same trend.

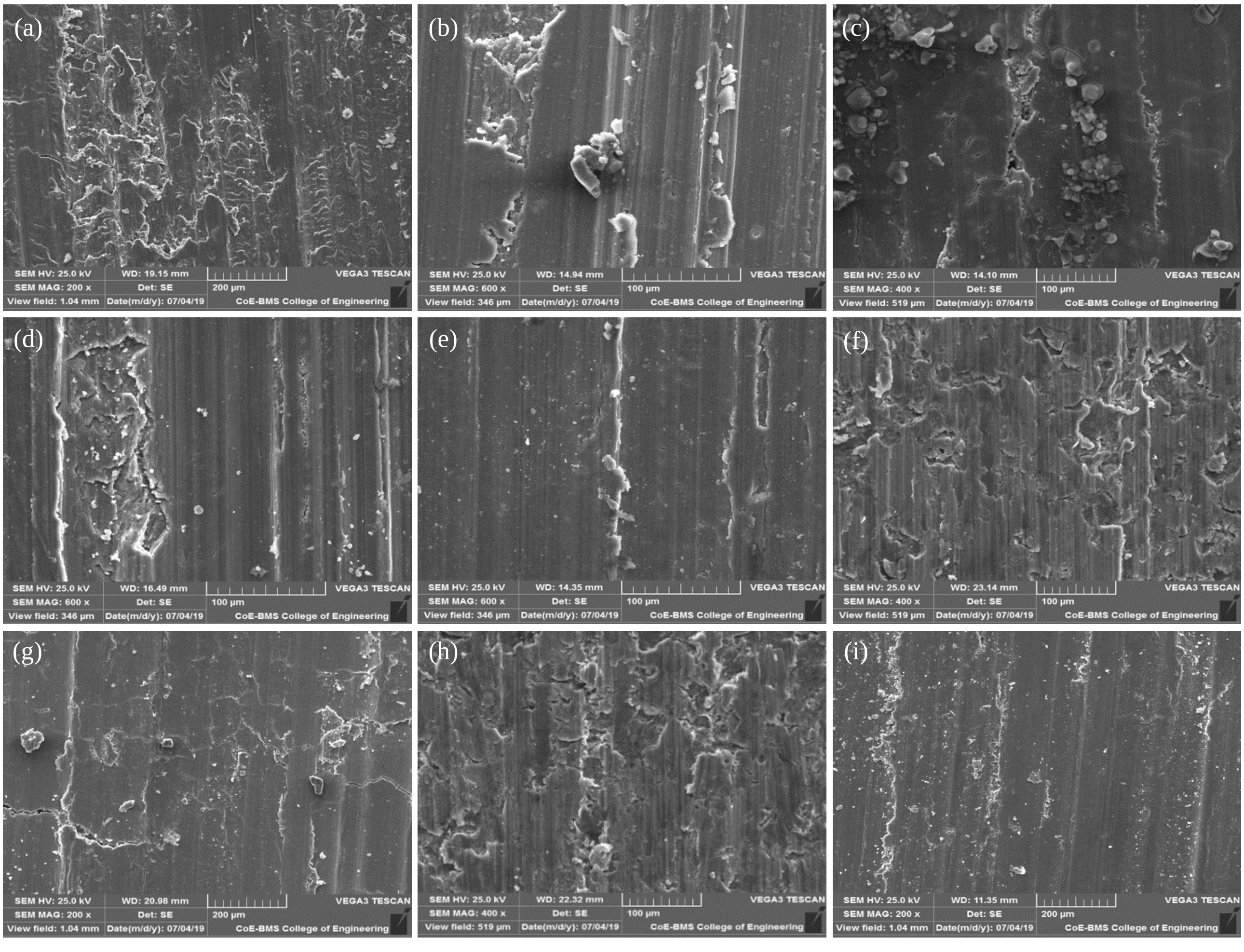

Figure 11. SEM image of worn surface of (a) Al6061 alloy; (b) Al6061-2 wt% MWCNTs (uncoated) composite; (c) Al6061-2 wt% MWCNTs (coated) composite; (d) Al6061-0.5 wt% of graphene composite; (e) Al6061-1 wt% graphene composite; (f) Al6061-2 wt% MWCNTs (uncoated)-0.5 wt% graphene composite; (g) Al6061-2 wt% MWCNTs (coated)-0.5 wt% graphene composite; (h) Al6061-2 wt% MWCNTs (uncoated)-1 wt% graphene composite; and (i) Al6061-2 wt% MWCNTs (coated)-1 wt% graphene composite.

3.3 Microstructure examination of worn surface

Worn surface of Al6061 alloy, nanocomposites and hybrid composites were examined by SEM micrographs. Figure 11(a) to (i) of worn surfaces of Al6061 alloy and hybrid nanocomposites with a normal load of 30 N and speed of 300 rpm were observed. The micrographs reveal wear debris of the Al6061 alloy and nanocomposite samples. It clearly indicates shallow craters with grooves of different sizes in the worn surface of samples.

Figure 11(b) and (c) reveals worn surface of composites reinforced with 2 wt% of MWCNTs (uncoated/coated) and Figure 11(d) and (e) reveals worn surface of composites reinforced with 0.5 wt% and 1 wt% graphene under normal load of 30 N. Wear debris of different sizes with small micro cracks are found in the micrographs. Figure 11(f) to (i) show SEM micrographs of worn surface subjected a load of 30 N at a speed of 300 rpm. The abrasive wear under normal load of 30 N results in showing up particles on the worn surface. Graphene and coated MWCNTs nanoparticles were also seen between the pin and disk surface. The wear mechanism may be a combination of delimitation and abrasion. A skinny prosperous thin film was seen to be formed from the graphene particles which help in preventing direct metal contact thereby indicating that the weight loss depends on the available graphene film layer that could well serve as a protective layer which in turn prevents the breaking of hard coated MWCNTs particles finally resulting in lesser surface damage and delimitation wear in limited regions.

Graphene behaves as a solid lubricant which is responsible for decrease in wear rate due to the formation of a protective barrier. Addition of MWCNTs would certainly increase the hardness of the composite and this has determinantal effect on the wear behaviour of the composite. The two reinforcements do not react to form a separate phase in the matrix. However, both individually act against wear.

3.4 Hardness

The hardness of nanocomposites were evaluated and exhibited in Table 5 and Figure 12.

The hardness of the alloy reinforced with 2 wt% coated MWCNTs and 1 wt% graphene revealed maximum micro hardness value compared to the other samples processed. The coated MWCNTs were responsible for the improvement in hardness. Coating would have necessarily enhanced the interfacial bonding and aided in the formation of dislocations. Amongst the nanocomposites reinforced with only graphene, it was noticed that increasing the wt% of graphene from 0.5 to 1, would decrease hardness. This may be attributed to the agglomeration of graphene in the matrix with increasing graphene content.

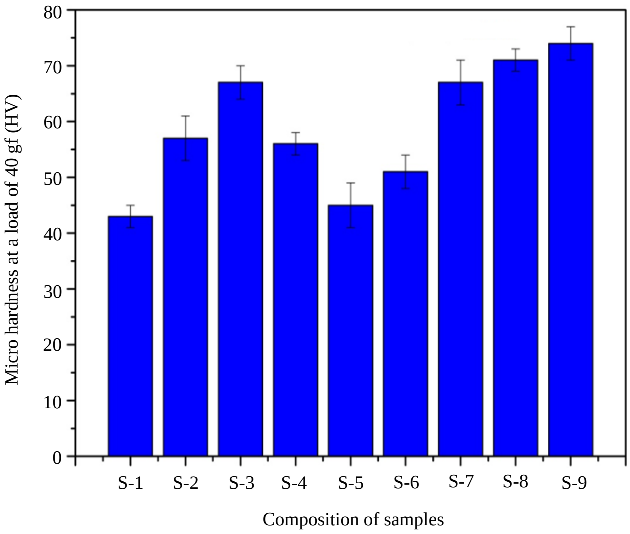

Figure 12. Microhardness of nanocomposites.

Table 5. Microhardness of the nanocomposites.

Sample No	Particulars	Micro Vickers Hardens value (HV)
S-1	Al6061 alloy	43±2
S-2	Al6061-2 wt% MWCNTs (uncoated) composite	57±4
S-3	Al6061-2 wt% MWCNTs (coated) composite	67±3
S-4	Al6061-0.5 wt% graphene composite	56±2
S-5	Al6061-1 wt% graphene composite	45±4
S-6	Al6061-2 wt% MWCNTs (uncoated)-0.5 wt% graphene composite	51±3
S-7	Al6061-2 wt% MWCNTs (coated)-0.5 wt% graphene composite	67±4
S-8	Al6061-2 wt% MWCNTs (uncoated)-1 wt% graphene composite	71±2
S-9	Al6061-2 wt% MWCNTs (coated)-1 wt% graphene composite	74±3

4. Conclusions

The present research on coating of MWCNTs, processing and assessment of wear properties of the Al6061-MWCNTs (coated/uncoated)-graphene metal matrix composites processed through power metallurgy techniques has led to following conclusions.

1. MWCNTs were successfully copper coated by two-step electroless deposition method.

2. Nano hybrid composites with 0.5 and 1 wt% of graphene, 2 wt% of MWCNTs (coated/uncoated) in Al6061 base matrix using powder metallurgy technique are fabricated.

3. Weight loss was lower in minor load condition (20-30 N) for the nanocomposites compared to base alloy, however as load increased from 30-40 N the nanocomposites displayed higher weight loss than Al6061 alloy. At higher loads, wear behaviour of Al6061-MWCNTs (coated)-graphene nanocomposites showed better wear resistance than Al6061-MWCNTs (uncoated) nanocomposites.

4. The weight loss of the Al6061 nanocomposite specimens' increases with increasing speed. MWCNTs (coated) and 0.5/1 wt% graphene particulate reinforced composites exhibited a considerable reduction in the volumetric weight loss.

5. The examination of worn sample surfaces at major load, revealed that adhesion mechanism was dominant for the Al6061 alloy, while delamination was responsible in for wear in the fabricated nanocomposites. During minor load, abrasion was the leading wear characteristic for both nanocomposites and Al6061 alloy.

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