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## Effect of Reinforcement Surface Area on Tribological Behaviour of Aluminium Alloy Nanocomposites

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### Abstract

Current research work is focused on evaluating the influence of Graphene/SWCNT/MWCNT addition on the tribological properties of developed composites. AA 6061 with varying alloying elements, well known for structural application are used as matrix. XRD analysis, relative density and hardness measurement are performed on the nanocomposites. Dry friction wear test is conducted to evaluate the wear performance of developed nanocomposites under various normal load (N) and sliding speeds (m/s) at ambient temperature. Corresponding wear loss (gm) and friction coefficient were measured to evaluate the efficiency of the composite in terms of tribological performances. Wear tracks and wear debris were analyzed through scanning electron microscopy (SEM) and Raman spectroscopy.

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*Keywords:* Graphene; Carbon Nanotube; Nanocomposites; Tribology

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## 1. Introduction

Nano-technology has enabled the revolutionary research on developing materials with improved strength; lightweight combined with wear resistance properties. This cutting edge technology created an impact on global materials research especially in sports, aircraft and automobile industries.

### Nomenclature

SWCNT	Single wall carbon Nanotube
MWCNT	Multi wall carbon Nanotube

The recent decade has witnessed research on fabrication of composite using various carbon allotropes such as Graphite, Single walled carbon nanotube (SWCNT), Multi-walled carbon nanotube (MWCNT) and Graphene etc. The addition of CNT to Al and its alloys resulted in significant improvement in the hardness due to high densification, decrease in coefficient of friction, drastic decrease in wear rate [1] and decrease in friction stability [2], but these composites are mainly influenced by applied load [3]. It was also reported that the CNT possess self-lubrication property, which creates the solid lubricant layer under dry wear conditions [4]. Graphene being a new class of material in carbon group with strong sp<sup>2</sup> – hybridized 2D- sheet like nanomaterial is endowed with extreme strength. Further it has attracted massive attention due to its excellent physical and mechanical properties (Tensile strength 130 GPa & Thermal conductivity  $-5.3 \times 10^3 \text{ Wm}^{-1}\text{K}^{-1}$ ). [5] In order to make an effective utilization of the astounding properties of carbon allotropes, they are currently used in the form of reinforcements in various proportion in aluminum and its alloys to fabricate the composites [6]. It is also being reported that Graphene is incorporated in oil and grease in nano range, which results in higher load carrying capacity compared to that of raw grease and oils without additives. Which shows that Graphene possess self-lubricating capacity. But very few literatures are available on Graphene reinforced aluminum alloy MMCs with reference to the tribological applications. Since, the structure of all carbon allotropes varies and each of them exists in unique physical structure it leads to diverse strengthening mechanism pertaining to varying tribological potential. The tubular structure of the CNT exhibits only point contacts, less carbide formation and when it is subjected to high pressure its structure will collapse compared to Graphene, which has planar sheet like structure (2D), with more contact surface area in the developed composites [7-8]. So, this lucidity is to be irradiated by advanced processing methods, which can make them suitable for industrial applications with well-established tribological performance. Study on AA 6XXX based composites reinforced with carbon-based elements is not abundantly reported in terms of various tribological applications with improved strengths [9]. In the current study AA 6061 reinforced with the SWCNT, MWCNT and Graphene - Metal Matrix Composite (MMCs) are fabricated by vacuum hot press. The reinforcements and matrix particulates are homogeneously dispersed through probe ultrasonic method and the developed composites are tested for its tribological potential. Comparison and analysis is carried out to investigate the effect of friction, wear and structural damage on the added carbon allotropes during various testing conditions.

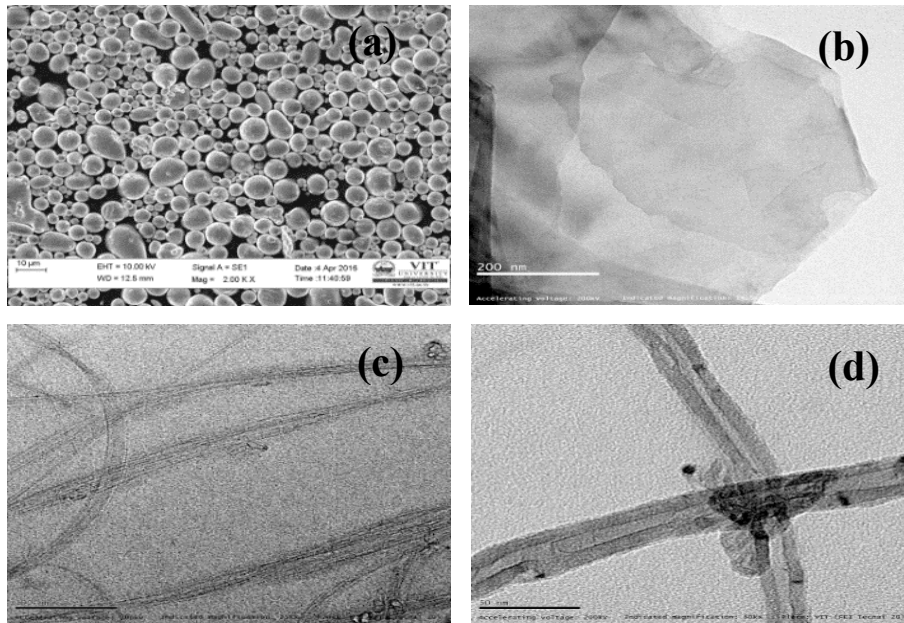
## 2. Materials and methods

### 2.1. Materials and fabrication

Gas atomized, near spherical shape AA 6061 (~ 10 μm in dia, >99.98% purity) (Fig.1) supplied by Ampal Inc. NJ, USA were employed as matrix precursor. Table.1 summarizes the various chemical compositions of aluminum alloy corresponding to the measured value. Graphene from Angstrom Materials Inc., Dayton, OH, SWCNT, MWCNT supplied by UNI Pvt, Ltd., INDIA were used as reinforcement. Graphene, SWCNT & MWCNT (Fig.1 a,b,c) respectively which were initially ultra-sonicated for 45 min each separately (0.5 wt. % each) in a beaker containing acetone to get individual exploitation and uniform dispersion. Calculated amount of AA alloy powder is stirred in a shear mixer for 60 min to obtain uniform slurry, then Graphene and CNT dispersions were added separately dropwise on individual alloy powder slurry, filtered to remove the acetone and vacuum dried for 2h at 60°C. Thus obtained precursors are vacuum hot pressed at 500 °C with a holding time of 1hr at 200 MPa pressure to get Ø8mm\*20mm long billet followed by extrusion.

Table. 1 Chemical composition of AA 6061 (Wt. %)

Aluminum Alloy	Zn	Cu	Mg	Si	Cr	Fe	Ti	Mn	Al
AA 6061	0.25	0.9	0.23	0.6	0.35	0.73	0.15	0.34	Bal.



**Fig.1** SEM of (a) AA 6061 alloy powder particle; TEM of (b) Graphene (c) SWCNT (d) MWCNT

## 2.2. Characterization and testing

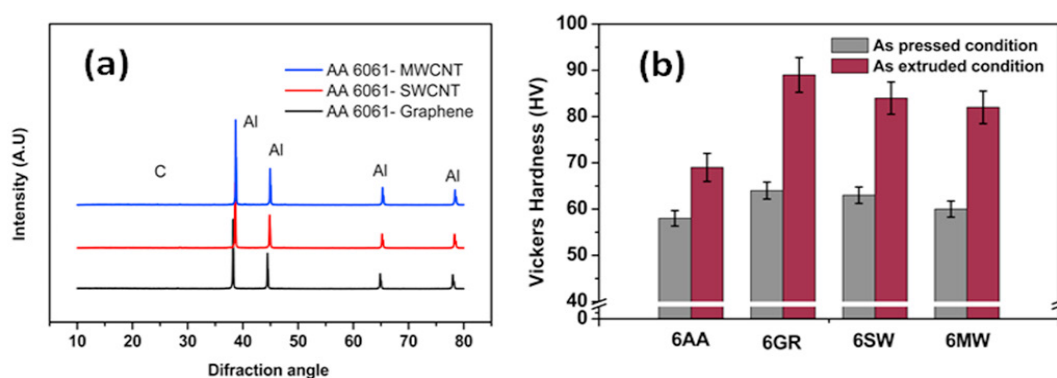
The square bars are fine polished to remove contaminants and microgrooves arisen on the surface during processing. X-ray diffraction analysis (XRD; BRUKER D8 advance) was carried out to analyze the internal phase constituents on all samples with the step size of  $\Delta = 0.0250$ . Vickers hardness (HV) test was conducted according to ASTM E-384 with (Matsuzawa MMT-X, micro diamond indenter) maximum indenting load of 500 gf for a dwell time of 10s, readings were taken with the stepping of 100  $\mu\text{m}$  between each measurement. Tribological test of extruded AA 6061 composites were performed through pin-on-disc wear and friction testing machine (DUCOM - TR-201LE, INDIA) with disc material: EN-31 steel (hardness: 60HRC) in dry sliding conditions according to ASTM G 99-95 standards at ambient temperature and 60- 65% humidity. Wear debris are collected to study morphology and structural changes of various reinforcement separated through vacuum setup. The tests are repeated four times for each experiment and the average outcomes are reported. Further, the significant changes on the wearing surface due to incorporation of Graphene SWCNT & MWCNT were characterized through scanning electron microscope (SEM; Carl Zeiss EVO 18). Further, EDS (Energy Dispersive Spectroscopy) analysis was performed to detect the various elements on the worn surface.

## 3. Results and discussion

### 3.1 Hardness and XRD analysis

XRD analysis outcomes of extruded AA 6061 Graphene/SWCNT/MWCNT composites in Fig. 2a

respectively shows the Aluminum peak, and other peaks due to unusual reactions. The elongated view shows (inset) the region  $2\theta$  from  $10.0^\circ$  to  $30.0^\circ$  which confirms the presence of carbon peaks in the extruded composites. The extruded sample of AA 6061 with various reinforcements shows the strong Al peaks and no other noticeable reflections were observed for carbide formation due to the presence of low content carbon allotropes. The micro hardness data were recorded by micro indenting on cross sections of the samples after vacuum pressed and extruded condition. Fig. 2b corresponds to micro hardness of pure AA6061 and AA 6061 – Graphene/SWCNT/MWCNT composites. With the same wt. %, Al 6061 - Graphene composites are observed to have higher hardness compared to as pressed condition, monolithic alloys, Al 6061- SWCNT and Al 6061- MWCNT under same processing conditions, superior to raw alloy and pressed state owing to dispersion strengthening mechanism irrespective of the Al<sub>4</sub>C<sub>3</sub> formations. The significant difference between Graphene and MWCNT reinforced composites are observed for both the alloys. This trend is attributed to various factors like, effect of various strengthening mechanisms, huge thermal mismatch between matrix and reinforcement, Orowan looping and strong interaction with additives [10-11]. Another reason for the difference in hardness is the existence of various interfacial phenomena due to the difference in specific surface area (SSA) of individual carbon allotropes with various physical forms (Graphene – sheet form, SWCNT & MWCNT – tubular form).



**Fig. 2** XRD patterns of (a) AA 6061- Graphene/SWCNT/MWCNT  
(b) Vickers hardness; AA 6061 with Graphene/SWCNT/MWCNT composites

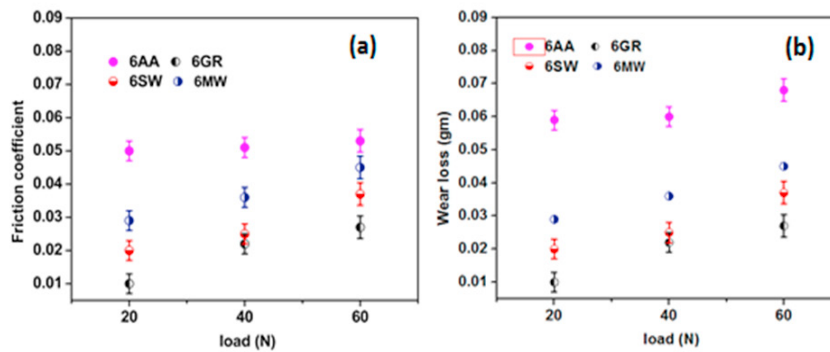
### 3.2 Effect of load on wear loss and friction coefficient

The wear test is performed on extruded AA 6061 and its composite reinforced with Graphene/SWCNT/MWCNT. The test had made it possible to validate the wear resistance of synthesized composites reinforced with different carbon constituents having varying physical forms and specific surface area. Many researchers have reported that wear loss is inversely related to hardness values of the composites or alloys [12]. It was also mentioned that physical form and surface contact area are also essential parameters that influences the wear loss. **Fig. 3** shows the plots comprising magnitude of weight loss (mg) and friction coefficient against load (N) with sliding distance of 1000 m.

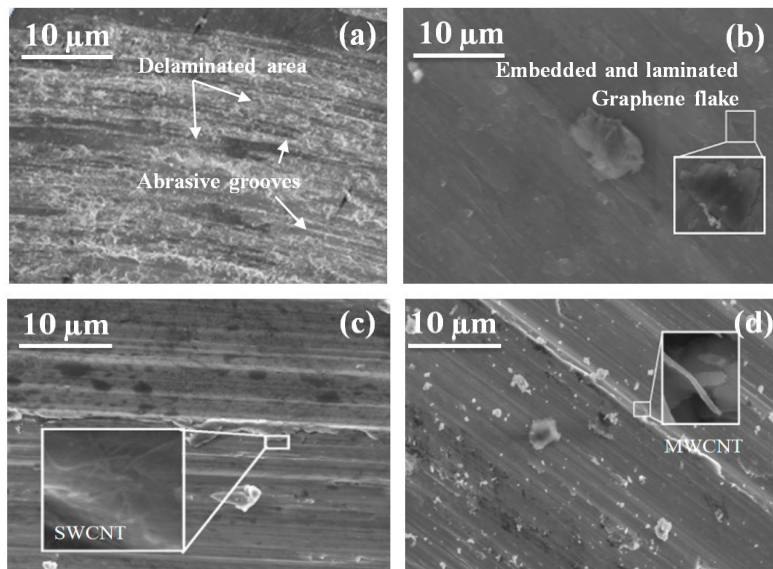
**Fig. 3a** presents the plot between wear loss and load for AA 6061 composites and it was found that wear rates increased dramatically for an increase in increasing load. This is due to increase in sliding forces, which will plow the wearing mechanism and increases the material removal rate. Further, AA 6061- Graphene composites are found to have lesser wear rate when compared to AA 6061 – CNTs composites and base alloy, which is attributed to difference in the interfacial bonding. Graphene composites exhibit more grain boundary pinning and more uniform distribution, which prevent the crack propagation. Such a boundary obstructive leads to increase in fracture toughness, thermal conductivity, heat and stress dissipation [13-14] which bring down the wear losses.

**Fig. 3b** shows the proportional increase in average friction coefficient with increasing load (20 to 60 N) at

a constant sliding speed of 0.15m/s for AA 6061 respectively. Composites posed lesser wear loss and friction coefficient compared to base alloy materials due to the presence of nanomaterials in the composites. The significant differences in the friction coefficients due to high protective nature of Graphene will lower the shear force and reduction in the material losses. Further, 2D Graphene stances high surface area and superoleophilic nature which leads to drastic reduction in the coefficient of friction compared to CNTs hydrophilic nature [15]. The reduction in the friction and associated mechanism is explained by puckering influence on Graphene sheet and CNTs when strongly bonded to the matrix. **Fig. 4** shows the SEM micrographs of worn surfaces of AA 6061 composites, which depict the effect of interaction of Graphene/SWNT/MWCNT and its influence on the worn-out surfaces after various loading condition. **Fig. 4a** represents AA 6061 base alloy in which more severe delamination of material and deep abrasive grooves are observed compared to composites. Analysis of worn surface of AA 6061- Graphene (**Fig. 4b**) shows the significant change in the wear mechanism from adhesion to abrasion. But this abrasion is arrested by the influence of plane puckering mechanism discussed earlier; no abrasive scars are inferred on the surface. Thus, Graphene in the composite (embedded or laminated on the surface; shown inset) will act as efficient wear resistant film there by lowering the friction as well as wear losses (in dry wear conditions). This type of solid lubricating film will result in enhanced wear resistant platform upkeep of delamination of the Graphene layer.



**Fig. 3** Wear loss; (a) AA 6061 Graphene/SWCNT/MWCNT Friction coefficient; (b) AA 6061 Graphene/SWCNT/MWCNT composites varying with the load (N)



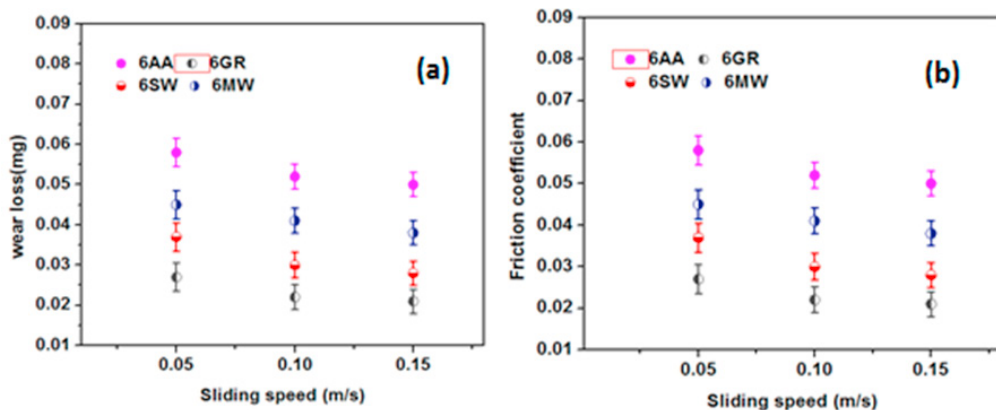
**Fig.4** SEM micrographs of worn surfaces (a) AA 6061 (b) AA 6061 – Graphene (c) AA 6061 – SWCNT (d) AA 6061- MWCNT

**Fig. 4c** shows the SEM micrographs of AA 6061-SWCNT; similarly, **Fig. 4d** shows the SEM micrographs of AA 6061-MWCNT, which were tested under different loading conditions. Few literatures are available on CNT based composites and its worn surface characterizations, but the current study, explain few other facts about tubular structure of CNTs in the composites during wear test. Minor abrasion scars were noticed on the worn surfaces, but majority of CNTs are positioned at squeezed out abrasive grooves valleys edges. This shows that tubular structure is more prominent to de-bonding behavior compared to planar and 2D Graphene at higher applied loads. However, the minimal differences in surface roughness compared with planar Graphene; CNTs composites are restricted with only line contacts and more cushioning whereas, Graphene establishes plane contact with the counter surfaces.

### 3.3 Effect of sliding speed on wear loss and friction coefficient

**Fig. 5** presents the plots comprising the magnitude of weight loss (mg) and friction coefficient against sliding speed (m/s) with a constant sliding distance of 1000 m. **Fig. 5a** shows the wear loss of AA 6061 varying with sliding speeds at constant applied load (40N). Decreasing trend of wear rate is observed for both the composite on increasing the sliding speeds. Base alloy experienced higher wear loss compared to carbon constituent's composites, which signifies the effect of added nano materials and their influence on wear resistance mechanism. More heat generation is expected while increasing the sliding speeds, but the presence of high thermally conductive reinforcements (Graphene- $5.3 \times 10^3 \text{ Wm}^{-1}\text{K}^{-1}$ , CNT - $35 \text{ Wm}^{-1}\text{K}^{-1}$ ) in the composites will take a role in dissipating the heat energy through the specimen in faster rate. Further, it will reduce the shear fatigue life of the reinforcements where, severe damages, pullout and structural damage of CNTs are observed before failure which is experimented for longer sliding durations. It implies that increase in the sliding speed has less influence to wear rate for AA 6061 composites compared to its base alloys.

**Fig. 5b** represents the plots between friction coefficients against sliding speed (m/s) of AA 6061 with sliding distance of 1000 m at constant applied load (40N). It is a common inference that lower degree of friction coefficient is addressed at higher speeds [16]. Initially it seems to be a higher but once a constant higher sliding speed is reached softening of both disc and pin material take place which results in lower friction values. More noise is observed for the composites compared to base alloy, which indicate that the pin is trying to just slide on the disc to overcome the wear. Further, rolling up of debris is seen at higher speeds resulting in damage and fatigue failure of CNTs. Also, embedded Graphene gives planar edges compared to spherical or sharp edge (asperities) of the CNT in the composites, which are prone to high stress interacting sites.



**Fig. 5** Wear loss; (a) AA 6061 Graphene/SWCNT/MWCNT Friction coefficient; (b) AA 6061 Graphene/SWCNT/MWCNT composites varying with the load (N)

#### 4. Conclusions

One of the common engineering research challenges in material is to determine ways and means to improve the wear resistance between any coupling surfaces, thereby reducing the energy and wear losses. In the current research work AA 6061 with Graphene/SWCNT/MWCNT are processed separately through vacuum hot press (gives high-density compacts > 80 to 90%) followed by hot extrusion successfully. With the same 0.5 wt. % reinforcement addition Al 6061 - Graphene composites are observed to be superior in hardness compared to monolithic alloys and MWCNT reinforced composites owing to uniform dispersion and higher dispersion strengthening mechanism. Graphene exhibit more grain refinement compared to CNTs, which improves the fracture toughness of the composites. This feature has reduced wear losses and therefore Graphene reinforced composites are suitable for various tribological applications. AA 6061- Graphene composites are found to experience lesser wear rate compared to CNT based composites and base alloy. This is attributed to the difference in interfacial bonding due to varying specific surface area (SSA). The significant differences in the friction coefficients due to high protective nature of Graphene will lower the shear force and reduce the material losses. Further, 2D Graphene stances high surface area and superoleophilic nature which leads to drastic reduction in the coefficient of friction compared to CNTs.

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