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ASSESSMENT OF MECHANICAL AND TRIBOLOGICAL PROPERTIES OF Al 2024- SiC - GRAPHENE HYBRID COMPOSITES

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Abstract

In the recent years, enormous research has been carried out and reported on ceramic and carbon based reinforcement. Graphene, no wonder has attracted significant research interest due to its extensive physical properties at its single atomic thickness and 2-D morphology. The current studies focus on the role of Graphene in reducing the wear and frictional coefficient of Al 2024- SiC - Graphene reinforced hybrid composites. Reinforcement chosen is 4, 8 and 12 wt. % of SiC with 0.5 wt. % of Graphene to investigate the lubricating property under dry wear conditions and processed through the ultrasonication and ball milling. The processed mixtures are hot compacted and microwave sintered. Hardness, density, X-ray diffraction and flexural strength analysis were done on developed composites. The dry frictional wear test were carried out using pin-on-disc tribometer to evaluate the effect of Graphene and SiC content in the composite under various normal load and disc speed conditions. Further, wear resistance and a reduction in the coefficient of friction (μ) values compared to pure alloys were observed. Surface roughness values (Ra) and microscopic studies on wear tracks were carried out. The possible tribological potential coupled with improved mechanical properties and surface roughness of Al 2024- SiC - Graphene hybrid composite processed through powder metallurgy were discussed.

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Keywords: Hybrid composites, Graphene, Flexural, Wear

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1. Introduction

Composites are the potential candidates for the advanced engineering applications and massive attentions have been given to further enhance their properties by combinations of novel, smart materials and advanced manufacturing techniques. The deeper knowledge of the materials is very much significant in order to achieve the strengths in the developed composite structures. Most of the studies on Aluminum and its alloys based metal matrix composite pose highest strengthening efficiency with the carbon, ceramic based reinforcements [1][2][3]. However, Aluminum alloys also have number of positive features together with low density, resistance to corrosion, thermal conductivity etc. Addition of small content of reinforcement with appropriate quantities to the Aluminum alloys can improve the stiffness, hardness, fatigue resistance and tribological properties which results in achieving the needed properties to enrich the efficiency and cost saving in industrial applications. Al 2xxx with copper as major alloying element (3.8 to 4.9 wt. %) is mainly used to manufacture aircraft fitting, couplings, shaft and gear materials for extensive industrial applications. But, the development of Al 2xxx based MMCs with uniform dispersion of reinforcements, strong interfacial bonding with controlled reaction is really in demand in the market[4][5]. Powder metallurgy practice has gained popularity and significance because of its near net shape, effective strength, alloy flexibility and its ability to reduce the complication of multileveled engineering components.

Graphene is an allotrope of carbon atom with single atomic layer of graphite organized into a hexagonal lattice **(Fig.1).** The stand-out properties is its inherent strength (at all temperatures) and found to be the strongest material ever discovered. Specifically these materials possess distinctive wear resistance properties and also superior mechanical properties leading to innovative applications. Addition of Graphene at nanoscale range becomes a promising reinforcing material in many engineering applications specifically in ceramics, polymers including metal or its alloys. Research on Graphene metal matrix composites are in infancy stage due to its challenges in homogeneous dispersion and the extensive reaction between metals interfaces during high temperature processing conditions. Unique mechanical properties of Graphene are found to be Elastic modulus; 0.5 – 1 TPa, [6][7] Specific Surface area; $2630m^2\ g^{-1}$, Tensile strength; 130 GPa [8] . Graphene serves as both solid and colloidal lubricant between the two sliding surfaces exhibiting its superior tribological advantage with very minimal usage (0.1 to 0.3%) and found to results in appreciable reduction in the wear loss and friction.[9] Graphene being used as additives to lubricant such as oils and grease in nanoscale range provides the higher load carrying capacity compared to that of oil or grease without the addition[10]. Limited studies on the aspects of effect of graphene content in Al alloy MMCs prepared by powder metallurgy approach and on its mechanical and tribological applications are available in literature. So this paper gives the outlay of the influence of Graphene content in Al2024 on tribological aspects under dry sliding conditions. Further, an attempt is made to develop the homogeneous dispersion strengthened Al 2xxx - SiC- Graphene by advanced powder processing method in liquid media. Further the precursor is uniaxial hot compacted and microwave sintered. Thus developed composite is subjected to evaluation of flexural and wear studies under various load condition to exploit the Graphene addition and microwave sintering.

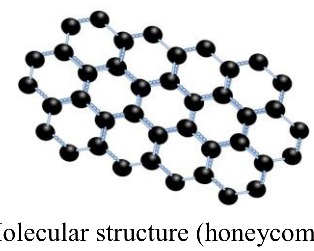

Fig.1 Molecular structure (honeycomb) of Graphene

2. Experimental

2.1 Materials

Al 2024 is synthesized through gas atomization method with density of 2.7 g/cm^3. The chemical composition of the matrix is given in the table.1. Also, Graphene with average platelets (flake) size >10 µm imported from USA with the density of 1.5 to 2.0 g/cm^3 and SiC particles with density of 3.20 g/cm^3 are used in this research work.

Table.1 Composition of Al 2024 used as matrix material (wt. %)

Element	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
%	0.5	0.5	4.0	0.4	1.5	0.1	0.25	0.15	Bal.

2.2 Al 2024-SiC –Graphene dispersion

AA 2024 – SiC – Graphene composites are produced through various steps. Initially, Graphene dispersion is carried out through an ULP (ultrasonic liquid processor) in a solvent. SiC with various (4.8 and 12) weight percentage were added to solvent containing Graphene which is dispersed in acetone. Further Graphene – SiC mixtures are kept in hot air oven to evaporate and dry the acetone. Thus obtained precursor is subjected to ball milling. Ball milling is carried out for 60 minutes with the ball to mill ratio 16:1 at 200 rpm rotating speed for all the combinations of SiC - Graphene weight fraction. Homogeneous dispersion and encapsulation of Graphene on SiC are achieved at optimized processing techniques and parameters. Al 2024 metal powder is added to ball milled Graphene - SiC precursors and ball milling process is continued with decreased ball mill ratio (5:1, to prevent the encapsulation of Graphene) for another 25 minutes. After this, the complete mixture of Al 2024-SiC-Graphene is subjected to consolidation.

2.3 Compaction, Microwave sintering and Testing

AA 2024-SiC-Graphene precursors are preheated in compaction C- 12 carbide dies with chromium tool steel die case and subjected to compaction (single action) according to ASTM B 925 – 08. The mixtures are compacted at 450 MPa compaction pressure for 30 min. High temperature lubricants such as boron nitride is applied between the punch and die walls while processing. Thus prepared compacts are subjected to sintering at 550 ˚C in an argon (inert gas) atmosphere for 3 hours followed by furnace cooling to attain room temperature. The steps followed to synthesize of Al 2024-SiC -Graphene hybrid composite is illustrated in the **Fig.2 (a. b, c, d & e).**

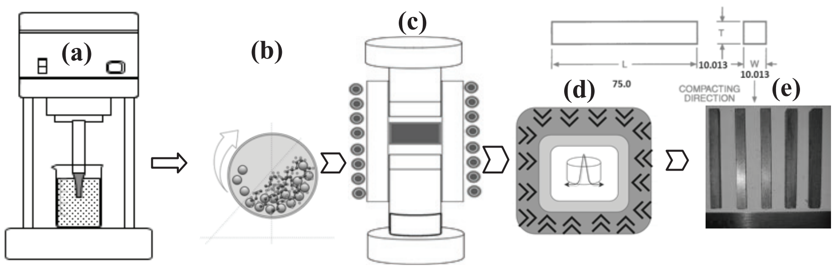

Fig. 2 (a) Powder processing through ultrasonic liquid processor (b) Ball milling (c) Powder hot uniaxial compaction in hydraulic press (d) Microwave sintering with inert gas facility (e) Al 2024 – SiC - Graphene bar samples

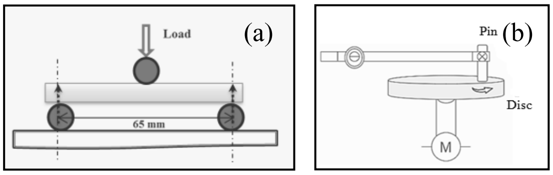

Fig.3 Schematics of (a) three point bending (Flexural) (b) pin-on-disc wear test

Hardness (Vickers-HV) values are measured for all the samples on the fine polished microwave sintered composite with 200 gf. Minimum three to five measurements on different areas and sides on the composites taken and the average values reported. Three point bending (flexural) test is carried out on samples prepared by EDM wire cut and fine polished composite sample of size 70 length (L) X 10 thickness (T) X 10 Width (W) (all in mm). The test is performed for Al 2024 and Al 2024- Graphene - SiC composite with cross head rate (strain rate) 0.1 mm/min and span length 6.6 mm between the two line supports **(Fig. 3a)** for all the composite in an axial loading testing machine (INSTRON – 8801) after fine polishing. The maximum flexural strength in MPa that the developed composite can withstand without crack initiation at room temperature is measured. Fresh samples are also prepared and fine polished at the edge for the wear analysis. The test carried out conferring to ASTM-G 99-95 standards (pin-on-disc friction and wear testing machine -DUCOM Instruments, Bengaluru, INDIA). The schematics of axial loading on the pin against the disk during the experimentation are shown in **Fig.3b**. The wear loss or mass loss is measured by measuring the sample weight before and after the experiment in an electronic balance (0.0001grams accuracy) along with coefficient of friction and surface roughness values (Ra) through surface roughness tester. Wear tracks are analyzed through electronic microscope.

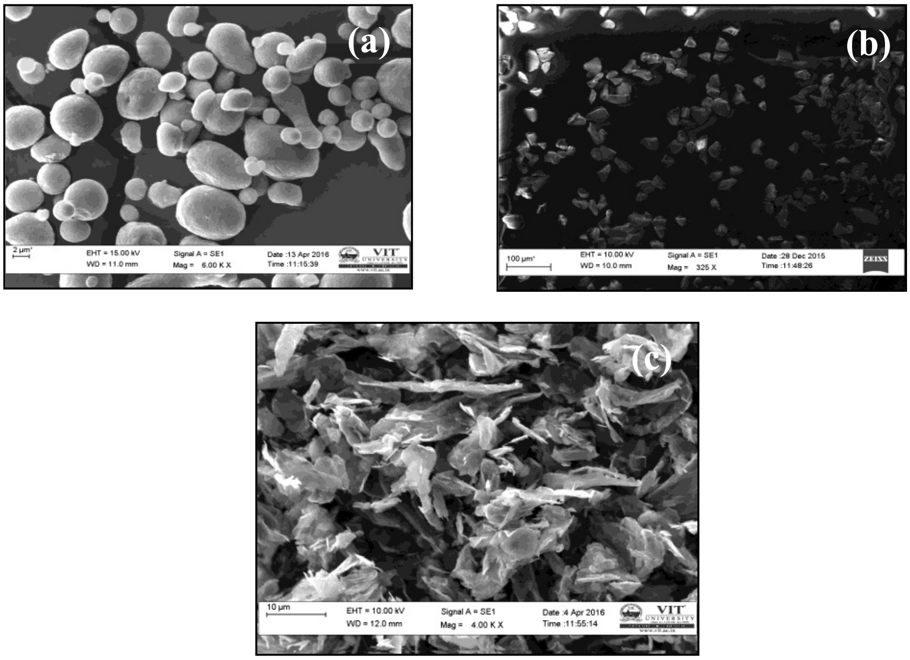

Fig.4 SEM image of (a) Al 2024 Powder morphology (b) SiC and (c) Graphene

3. Results and discussion

The received AA 2024, Graphene and SiC are observed through scanning electron microscope (SEM). **Fig.4a &b** shows the SEM micrographs of Al 2024 and SiC respectively in which Aluminum alloy particles are in spherical shape and size in the range of 20 to 30 µm whereas size of SiC ranges from 30 to 35µm. **Fig.4c** shows the sheet morphology and single layer structured Graphene in which the layers are stacked one above the other. Ball milled AA 2024-Graphene-SiC powder mixtures were analyzed under scanning electron microscope to confirm the homogeneous and partial encapsulation. The X- ray diffraction patterns of AA 2024 – SiC- Graphene of synthesized hybrid composite is shown in the **fig. 5.** The analysis is carried out from which the carbide peaks formation is visualized. Graphene peak is found to be present at 2θ equal to 26.50˚ (Very lower peak due to less concentration and depend on the sensitivity of the sample) diffraction phase of Al 2024. The formation of aluminum carbide is not occurred in any of the microwave sintered sample according to the analysis.

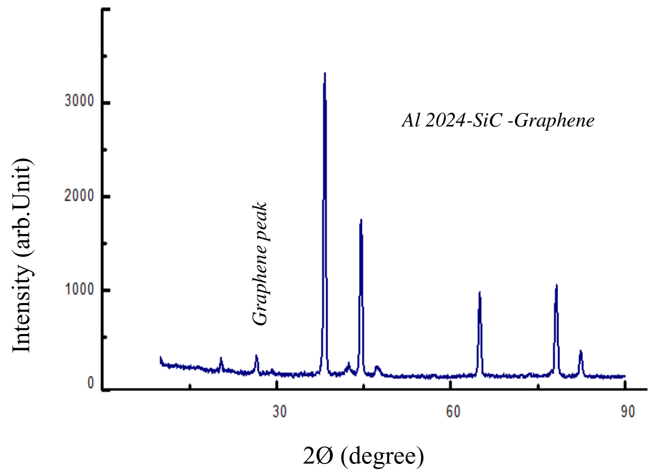

Fig.5 X – ray diffraction analysis of Al 2024-SiC- Graphene composite after microwave sintering

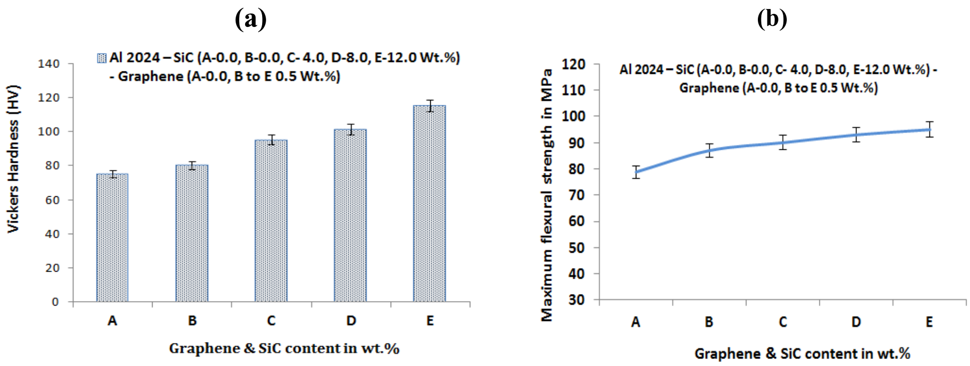

Fig.6 **(a)** Hardness values varying Graphene content (b) Flexural strength values of Al 2024 – SiC – Graphene hybrid composites

Fig.6a shows the average measured Vickers hardness values for various concentrations of Graphene and SiC in Al 2024 – SiC – Graphene nanocomposites. The hardness values for various concentrations of Al 2024 – SiC (A-0.0, B-0.0, C- 4.0, D-8.0, E-12.0 Wt. %) - Graphene (A-0.0, B to E 0.5 Wt. %) are found to be A-78, B-82, C-95, D-100, E-105 HV respectively. This shows that, the nanocomposite processed through ultrasonic dispersion and microwave sintering technique exhibit a the substantial improvement in micro hardness values compared to base alloy - AA2024 (A-82HV) also, the values are superior to results obtained from other processing method on the same material[11][12]. The grouping of SiC and Graphene are capable with excellent microwave absorption capacity [13]. Also, when Graphene is coated/encapsulated with SiC at lower concentrations (up to 0.5 wt. %) that leads to improved microwave absorption of materials which ascribed to change in dielectric parameters. Importantly, Graphene and SiC and in micro scale ranged particle that posses higher surface area which is crucial to make interfacial bonding effective and to enhance the overall densification by minimizing the porosity level. The significant improvement in hardness are may be due to formation of aluminum carbide in the nanocomposites. But, Graphene combined with microwave sintering in an inert gas atmospheric sintering method, prevented formation of other compounds.

Flexural strength values of Al 2024 – SiC – Graphene composites varying with weight percentage of SiC and Graphene content are shown in the **Fig.6b** along with the reference variation of Graphene and SiC content. Also, obtained flexural strength values of rectangular sample are related in equation.1for the three-point bending test.

$$\sigma = {}^{3PL}/_{2wt^2} \quad \ldots\ldots \quad 1$$

(Where, P=Axial load at fracture point (N), L=Length between two supporting span (mm), w=Width & t=Thickness samples (mm))

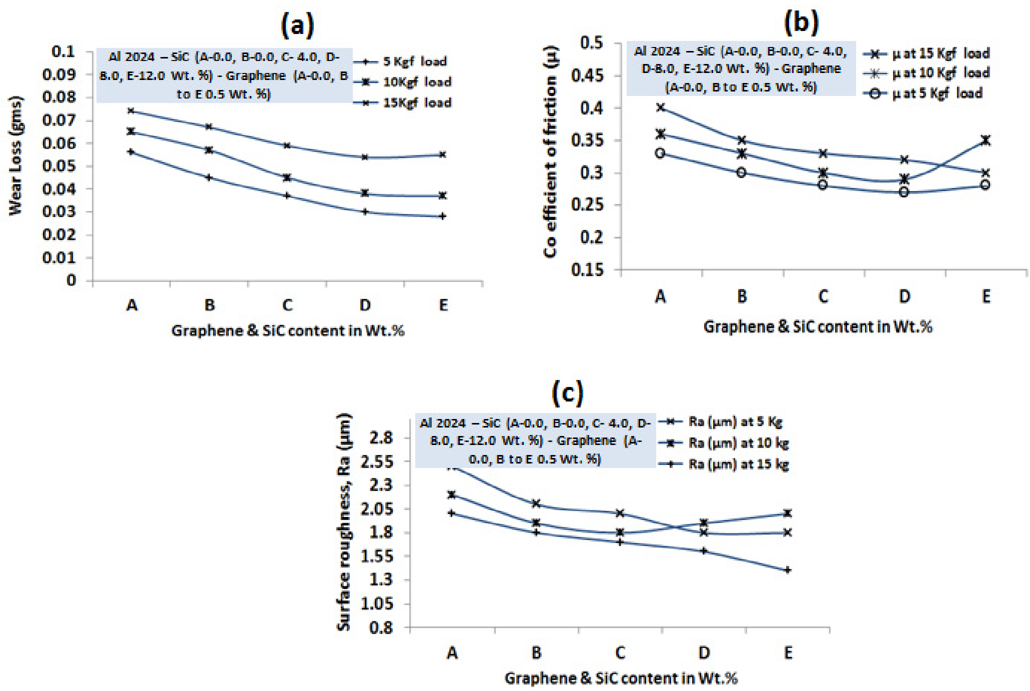

Fig.7 (a) wear loss, (b) co-efficient of friction, (c) Surface roughness with varying the Graphene and SiC content as a function of load

Since Graphene have higher surface area (2D material), individual exfoliation, uniform dispersion and integrating into it materials and integrate it into the matrix is relatively challenging. From the **Fig.6b** it is observed that there is an improvement in the flexural strength with addition of lesser quantity of combined additives Graphene (0.5 Wt. %) and SiC (0.4 to 0.8 Wt. %) in Al 2024 nanocomposites. Such increases (88 to 104 MPa) are comparable to monolithic alloy – Al 2024 (79MPa). The improved strength is attributed to effective microwave absorption during sintering and encapsulation during processing which leads to effective material transport in the composites and also added Graphene inhibit the grain growth formation leading to formation of finer grains. **Fig.7a** shows the wear loss (gms) of Al 2024 – SiC – Graphene nanocomposites with various SiC (A-0.0, B-0.0, C- 4.0, D-8.0, E-12.0 Wt. %) - Graphene (A-0.0, B to E 0.5 Wt.%) weight fractions as a function of load (kg) conditions. Addition of Graphene and SiC has positive outcome in the synthesized composites in which wear loss found to be reduced self-effacingly. Extensive wear resistance is due to strengthening of Al 2024 – SiC – Graphene composites by a combination and addition of Graphene and encapsulation with SiC which are in good agreement with the improved hardness and flexural strength values as discussed previously. **Fig.7b** shows that there is a decrease in the coefficient friction for an increase in Graphene and SiC content in the composite compared to Al 2024. Further, coefficient of frictional values (0.29 to 0.35) are found to be less for the composite compared to Al 2024 base alloy friction values (0.34 to 0.39) and are independent of applied normal loading. In general, surface roughness values are constituted by mating materials and hardness (relative). Further, added Graphene will act as a solid lubricant between the wearing surfaces and also presence of added SiC particles creates the point contact by reducing the third body abrasion. **Fig.7c** illustrates the comparison of surface roughness (Ra) values of Al2024-SiC-Graphene composites for various Graphene and SiC content. It can be inferred that the surface roughness, Ra value is found to be less when compared to that of the base material. The addition of Graphene found to result in a positive outcome in which coated / encapsulated SiC will becomes an efficient lubricating layer by smearing on the mating and wearing surface which will creates the fine and smooth surface that reduces the roughness values. **Fig.8** shows the optical micrographs of Al 2024 – Graphene -SiC nanocomposites wear out surface which are rubbed in unlubricated environments. Many macro cracks or grooves are observed **(Fig.8a)** for the samples without and very less micro surface **(Fig.8 b, c, d & e)** on Graphene (A-0.0, B to E 0.5 Wt. %) and SiC (A-0.0, B-0.0, C- 4.0, D-8.0, E-12.0 Wt. %) reinforced composites. So it can be concluded that the combination of Graphene and SiC with coating /encapsulation leads in enhancing the in tribological properties at its optimized values.

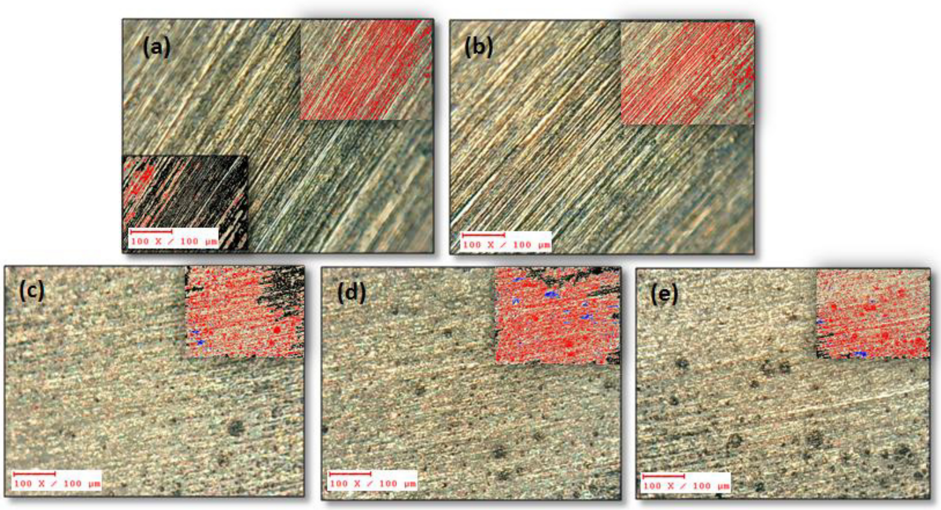

Fig. 8 Optical micrographs of worn out surface with various Graphene concentrations Al 2024 – SiC (A-0.0, B-0.0, C- 4.0, D-8.0, E-12.0 Wt.%) - Graphene (A-0.0, B to E 0.5 Wt.%)

4. Conclusions

In this research work, the Al 2024 – SiC (0.0-12.0 Wt. %) - Graphene (0.5 Wt. %) nanocomposites were successfully processed through ultrasonic liquid processing (ULP) method followed by planetary ball milling to obtain homogeneous dispersion. The mixtures are preheated and uniaxial hot compacted in the semi-solid regime and microwave sintered successfully (both in inert gas atmosphere). Addition of SiC (0.0-12.0 Wt. %), Graphene (0.5 Wt. %) and encapsulation enables significant improvement in both hardness and flexural strength compared to base alloy. Microwave sintering method can efficiently increase the diffusion of ions in the composite and thus gear up the sintering process, leading to finer grain growth and the densification. Also, Combination of SiC & Graphene in the nanocomposites has resulted in significant improvement on tribological properties where, it gives the wear resistance by creating a solid lubricant layer between the sliding surfaces. Also, results revealed that there is a decrease in wear losses, surface roughness and coefficient of frictional values which are observed to be due to Graphene and SiC content and also increasing the SiC in the composite leads to further improvement in the tribological properties.

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