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Experimental Investigation on Graphene Reinforced Aluminium Matrix Composites for Tribological Characterisation Using Pin-On-Disc Apparatus

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Abstract

The tribological behaviour of powder metallurgy-synthesised aluminium/Al_2O_3/Graphene has been investigated. The crystal structure, mechanical, and dry sliding wear behaviour of the manufactured composite were investigated. Advanced characterisation techniques like optical microscopy, SEM, and optical profilometer analysis were employed to investigate microstructure and worn surface morphology. The Universal Testing Machine's die and punch assembly was used to create four different cylindrical pre-forms with different Al_2O_3 content levels (0 wt.% %, 1 wt.% %, 3 wt.% %, and 5 wt.% %) at a compaction pressure of 120 KN. Different sintering temperatures of 490°C, 520°C, and 550°C have been achieved by using an Electrical Muffle Furnace. A pin-on-disc wear tester was used to study the sliding wear behaviour of a cylinder with a diameter of 12 mm against an EN-32 steel disc at a speed of 300 rpm and a distance of 4000 m in dry circumstances. Rockwell's Hardness machine with a 100 kg load applied at ball point is used to test hardness. Scanning Electron Microscopy (SEM) is used for micro-structural characterisation in order to look into the grain structure. Elevating the sintering temperature and adding more Al_2O_3 and 1% graphene made the specimen harder. Increasing the Al_2O_3 concentration and sintering temperature in experiments reduced wear. Microscopy by SEM showed that the Al_2O_3 particles were evenly dispersed throughout the aluminium matrix.

Major Findings: *Density increases with 5% Al_2O_3 content from 450°C to 490°C compared to 0%, 1%, and 3%. The specimen at 5% Al_2O_3 has low friction due to reduced wear than at 1%, 3%, and 5%. Innovative framework integration for mechanical and dry sliding behaviour enhancement.*

Keywords: *Aluminium, Al_2O_3, Coefficient of Friction, Hardness, SEM Analysis, Wear Properties*

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1.0 Introduction

Composites as unique engineering materials represent one of the most important advancements in the field of materials during the last several decades. Composition, dispersion, and interaction of the constituent parts of a composite have a considerable influence on the qualities of the result. Composites are a sort of multi-phase material created by mixing many components to produce properties that would be unattainable with a single material. Composites possess a variety of desired properties, including low density, excellent electrical conductivity, high temperature stability, high stiffness and strength, a variable coefficient of thermal expansion, and enhanced wear and corrosion resistance, among others. Composites are also regarded as varied products since they are composed of two or more distinct ingredients. On a molecular level, however, they are homogenous, meaning that each component of the molecule has the same physical characteristics. While a lot of different metals and alloys have been used as matrix materials, nickel and its alloys have received most of the research and development attention. Aluminium stands out from other materials in its class because it has a unique combination of features. It costs less than magnesium and titanium, is easier to make, and is cheaper. Due to its potential as a structural material in numerous fields, including automotive and aerospace, aluminium alloys have recently piqued the interest of several scientists, engineers, and designers[1-7].

The materials characterisation of silicon carbide reinforced titanium (Ti/SCS-6) metal matrix composites was thoroughly described by Liaw, Diaz[8]. The mechanical characteristics of nanocrystalline nickel created by electrodeposition were studied by Ebrahimi, Bourne[9]. In a handbook, Kutz[10] described material selection in different applications. Nanocrystalline nickel pulse electrodeposition was described by Qu, Zhu[11], employing a high peak current density and an ultra-narrow pulse width. The fabrication of nanostructured materials using aided electrodeposition was described by Bera, Kuiry[12]. Thermal plasma deposition from micro-to-nanostructure and from thick to thin coatings was covered by Fauchais, Vardelle[13]. In a handbook, Bhushan[14] talked about material nanotechnology. A review of the use of mechanically alloyed nanoparticles was conducted by Chawla, Prakash[15]. Using the Sol-Gel technique, Mackenzie and Bescher[16] talked about chemical pathways in the creation of nanomaterials. Ionic liquid electrodeposition for various materials was covered by Schubert, Zein El Abedin[17]. The tensile and fracture characteristics of an Al–Cu–Mg alloy that is discontinuously reinforced with silicon carbide particles (SiCp) were examined by Manigandan, Srivatsan[18]. The effects of ZrO_2 concentration and casting temperature on the mechanical characteristics and fracture behaviour of A356 Al/ZrO_2 composites were examined by Abdizadeh and Baghchesara[19]. Using the stir casting method, A356 aluminium alloy matrix composites reinforced with 5, 10, and 15% ZrO_2 were created at different casting temperatures, specifically 750, 850, and 950 °C. The creation and mechanical analysis of metal matrix composites made of aluminium alloy, alumina (Al_2O_3), and boron carbide were described by Ramnath, Elanchezhian[20]. Aluminium is a matrix metal with qualities including great strength, low weight, and simplicity of machining. Because of its special mechanical and thermal characteristics, graphene has recently been regarded as the perfect reinforcement material for the creation of nanocomposites[21]. Arora, Astarita[22] described the successful use of an aluminium plate and molybdenum powder in the Friction Stir Process (FSP) to fabricate Metal Matrix Composite (MMC). The objective was to improve the mechanical characteristics of the Al plate as received by creating a surface MMC layer on it. Singh, Singh[23] Using a pin-on-disc wear measuring equipment, the wear and friction characteristics of silicon carbide-based aluminium metal matrix composites and aluminium matrix alloy under dry conditions at various sliding distances (i.e., 1979.20 m and 2262 m) with loads ranging from 10 N to 30 N have been investigated. Kumar and Jayahari[24] investigations were conducted into the wear behaviour and mechanical characteristics of aluminium 6063 matrix composites supplemented with high carbon steel machining chips. Steel chips with an average size of 50–100 microns, weighing 5–10 weight per cent, were used to reinforce aluminium 6063. Idusuyi and Olayinka[25] discussed Aluminium Metal Matrix Composites (Al MMCs) are composites of aluminium reinforced with continuous or discontinuous fibres, whiskers, or particles. Samal, Vundavilli[26] researchers from all around the world shifted their focus from monolithic to composite materials due to the need for materials with excellent

performance and low cost. An enormous amount of work has been done recently to create a variety of metal matrix composite combinations. According to research by Zhang, Zhang[27], strengthening aluminium alloys is a useful technique to enhance their qualities. Because of its exceptional all-around performance, graphene is the perfect reinforcement for the creation of Aluminium Matrix Composites (AMCs). According to research by Kumar, Lokesh[28], graphene-reinforced Al 6061's mechanical properties have significantly improved. Using the stir casting method, Al 6061 composite material including graphene particles was effectively created by adjusting the weight percentage of graphene between 0 and 0.25, 0.5, 0.75, and 1 weight per cent. Sadhu, Mandal[29] explained development of aluminium-based composite materials has been driven by the increased need for lightweight engine and structural materials for automotive and aerospace applications. Iron and copper-based composites are probably going to be replaced by these composite materials. Yu, Zhang[30] researched samples of 7075 aluminium alloy, CFRP, and 7075-Al/CFRP hybrid were chosen for the pin-disk friction test in order to investigate the impact of fibre reinforced resin matrix composites on the friction characteristics of aluminium alloy. The surface morphology of the friction samples was examined using scanning electron microscopy and white light interference. The significance of this research lies in its novel approach to integrating a new framework, applying an innovative technique, or addressing an unexplored gap. Unlike previous studies, this work investigates Aluminium/Al_2O_3/Graphene has been investigated. The findings offer valuable contributions to the automobile and aerospace field by providing new perspectives on the enhancement of mechanical and dry sliding behaviour and suggesting practical applications in automotive braking systems and aerospace components. These insights pave the way for further research and development in said area, making this study a significant addition to the existing body of knowledge.

2.0 Methodology: Experimental Set-Up and Procedure

Research on the influence of sintering temperature and microstructure on the various compositions of an Aluminium Metal Matrix composite is being conducted. After experimentation was conducted on pure aluminium powder and graphene powder from Sigma-Aldrich.

2.1 Design for Experiment

Design experiments have been done for different compositions at different temperatures. Table 1 represents the design of experiments.

Table 1. Design of experiments

T1 (490°C)	T2 (520°C)	T3 (550°C)
T1 (0% Al_2O_3/ Graphene)	T2 (0% 0% Al_2O_3/ Graphene)	T3 (0% 0% Al_2O_3/ Graphene)
T1 (1% Al_2O_3)	T2 (1% Al_2O_3)	T3 (1% Al_2O_3)
T1 (3% Al_2O_3)	T2 (3% Al_2O_3)	T3 (3% Al_2O_3)
T1 (5% Al_2O_3)	T2 (5% Al_2O_3)	T3 (5% Al_2O_3)

2.2 Sample Preparation

The material of Al_2O_3 and graphene, sample preparation and sintering temperatures were varied. Since the Die and the Punch might often fuse under pressure, either with the green sample or the Die material, a lubricant was employed to make sample preparation easier; this was accomplished by spreading graphite powder over white petroleum jelly on the Punch.

2.3 Process of Experiment

Graphene powder was combined with a variety of alumina mixtures. Similar to the other components, the alumina content varies by weight %, as was previously mentioned. Alumina content may be found at 0%, 1%, 3%, or 5%. Each sample is lubricated with graphite powder mixed with white petroleum jelly. Sigma Aldrich Limited was where we made our powder buy.

2.4 Cold Compaction and Sintering

Axial pressing in a steel die with hydraulic pressure is the most prevalent method of compacting. Components of complex geometry may be pressed in a single step at a high rate of speed. After the component reaches the required level of strength, it is expelled from the tool die.



Figure 2. Compaction process using hydraulic UTM and final samples.

The combined samples were subsequently cold crushed using a 10-mm diameter die and a continuous uniaxial load of 120 KN. With a UTM machine, powder may be compacted (compaction procedure was done at a very slow feed rate for uniform compaction without any damage to the punch and sample). Powder particles fuse into a single mass after being compacted, taking on the form of a die. If you want to avoid breaking the compressed product, you should handle it carefully. The experiment was repeated with Al_2O_3 concentrations of 0%, 1%, 3%, and 5% to ensure consistency in observations. According to the foregoing, a total of 12 samples were collected. In a short amount of time, the created green compacts were transferred to the sintering process. Figure 1 shows the compaction process using hydraulic UTM and final samples.

2.5 Sintering

The compacted samples were taken and sintered in a muffle furnace at temperatures of 490°C, 520°C and 550°C to densify the compacted powder. Figure 2 shows an electrical furnace. Also, Table 2 represents the value of time to reach sintering temperatures and the still temperature time at different sintering temperatures.



Figure 2. Electrical muffle furnace.

2.6 Finishing Operations

The sintered parts were then polished and finished manually using emery paper and a surface grinding tool,



Figure 3. Surface grinding tool.

Table 2. Design of experiments

S. No.	Sintering Temperature (°C)	Time to reach sintering temperature (min)	Still temperature time (min)
1	450	30	30
2	470	30	30
3	490	30	30

followed by cloth polishing. Figure 3 shows a surface grinding tool for finishing operations.

2.7 Optical Microscopy

In order to check the reinforcement visibility after polishing, carried out by optical microscopy. Figure 4 represents the dispersion of Al_2O_3 over the aluminium.

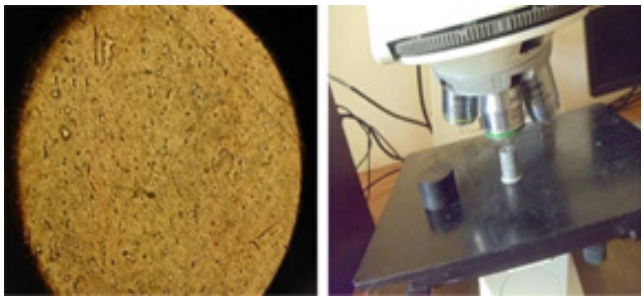


Figure 4. Dispersion of Al_2O_3 over the Aluminum.

2.8 Hardness Test

Using a machine developed by Rockwell, the hardness of the samples was determined using an HRB scale (make FIE). Before the hardness test, the specimen was metallographically polished, and its two sides were brought to precise parallelism. After subjecting the specimen to 100 kgf, the 1/16" steel ball indenter was used to measure the Rockwell hardness of the material. The average of the results from three separate measurements of hardness was recorded.

2.9 Wear and Friction Test

Wear behaviour of composite samples was analysed using a disc wear testing system (TR-20 provided by Megnun Engineering, Bangalore, India) outfitted with a 160 mm in diameter, 10 mm thick steel disc. Insert the sample pin between the jaws, then adjust its height about the test circle using the thumbscrew provided on the sample holding plate. The sample pin is held firmly in place by tightening the clamping screws on the moveable jaws, after which the height adjustment piece is used to ensure that the stacking arm is parallel to the plane of the worn disc. You may adjust the minimum necessary track span of 60 by adjusting the navigation of the scale-slider gathering. Securely fasten the slider bracing nuts and check the assembly's clipping. It's important to maintain the 10N test weight on the stacking dish. Flip the controller's "POWER" switch to the "ON" position. Remove the LVDT screw from the bolt. Adjust the thumb nut to move the LVDT plunger halfway out while monitoring the WEAR display. The WEAR show has to be in a balanced state within +/- 5 micrometres. Secure the LVDT in place with the provided bolt screw. Activate the MODE determination switch to choose the mode of operation (Time) for 20 minutes. Figure 5 represents the schematic and experimental arrangement of on disc apparatus, respectively.

Reduce the "wear" and "frictional force" displays to zero by moving the relevant Potentiometers to the OFF position. Join the thermocouple's male end to the hardware's female end. Include a thermocouple sensor of the K type in the jaw of the sample holder. To start the test, release the push catch on the controller's front board. To choose the desired rotational speed, just turn the SET 300 RPM handle in a clockwise direction. The SPEED display on the controller's front board indicates rotational speed in revolutions per minute. After the allotted time has elapsed, the test will end automatically. Also, the system may be manually stopped by pressing the STOP

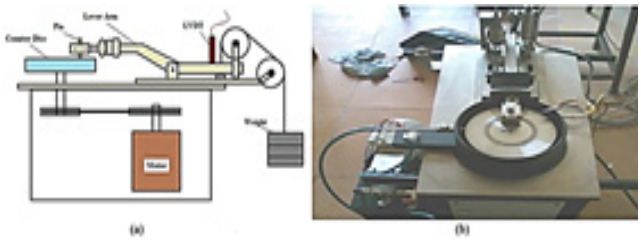


Figure 5. **(a)** Schematic diagram of pin on disc apparatus, **(b)** Experimental pin-on-disc apparatus.



Figure 6. **(a)** Wear and friction tester, **(b)** digital controller box.

button. Do the aforementioned procedure many times with different samples. All of the tests were conducted with a plate speed of 300 rpm, a 60 mm track sweep, and a connected load of 10 N. It takes around 12 minutes to test each sample, and the total distance covered throughout the test is about 4000 metres. Figure 6 represents the wear-friction tester and the digital controller box, respectively.

3.0 Results and Discussions

3.1 Hardness Test Results

Using a machine developed by Rockwell, the hardness of the samples was determined using an HRB scale (make FIE). Before the hardness test, the specimen was metallographically polished, and its two sides were brought to precise parallelism. After subjecting the specimen to 100 kgf, the 1/16" steel ball indenter was used to measure the Rockwell hardness of the material. The average of the

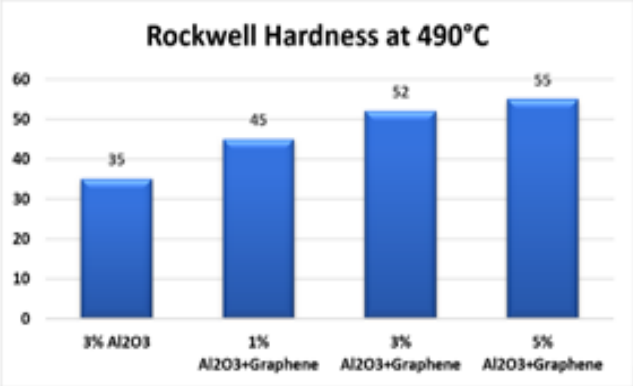


Figure 7. Rockwell hardness at 490 °C sintering.

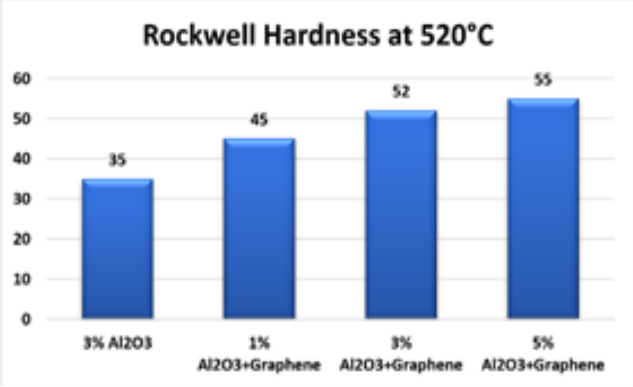


Figure 8. Rockwell hardness at 520 °C sintering.

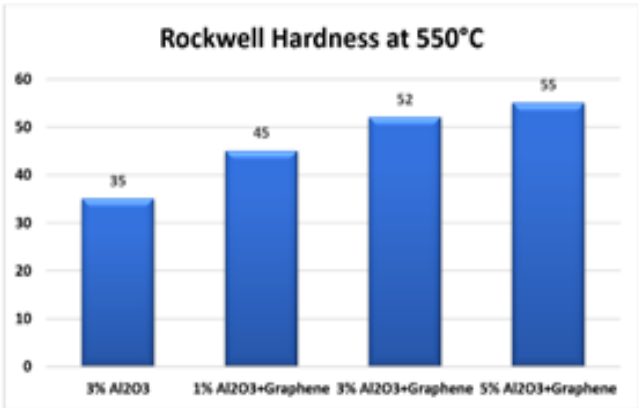


Figure 9. Rockwell hardness at 550 °C sintering.

results from three separate measurements of hardness was recorded. The hardness test results are shown below. Figures 7, 8 and 9 represent Rockwell hardness at 490°C, 520°C and 550°C respectively. From the results and compared to 0%, 1%, and 3%, Al_2O_3 content confirms dense structures at 5%.

3.2 Wear Test Results

After wear test, the sample at 550°C affirms the least wear and least COF when contrasted with 490°C and 520°C. Compared to specimens at 0%, 1%, and 3%, wear is minimal at 5% Al_2O_3. Figure 10 shows the variation in COF with sliding time for 3% Al_2O_3. Figures 11, 12 and 13 show variation in COF with sliding time for 1%, 3% and 5% Al_2O_3, respectively, along with 1% graphene.

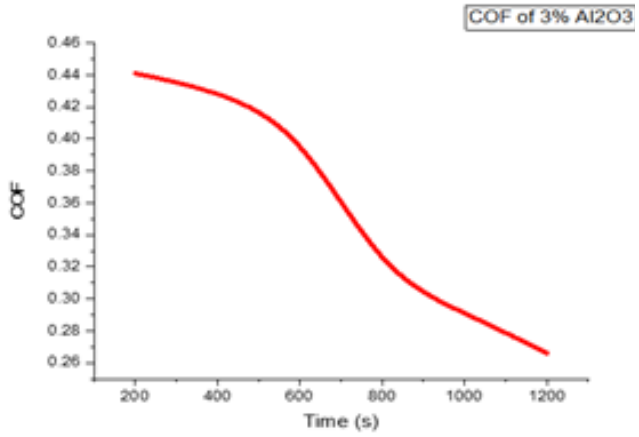


Figure 10. Variation in COF with sliding time for 3% Al_2O_3.

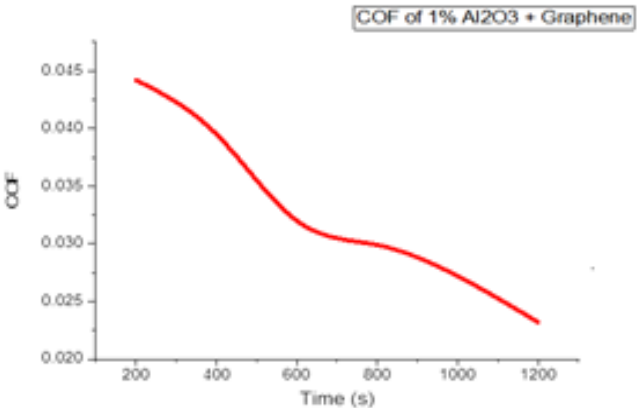


Figure 11. Variation in COF with sliding time for 1% Al_2O_3 and 1% graphene.

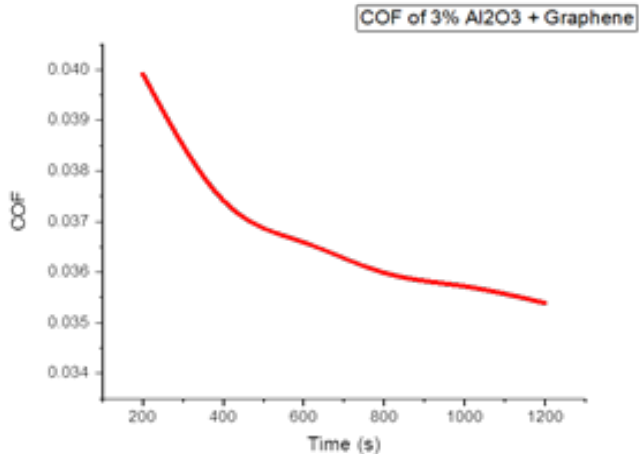


Figure 12. Variation in COF with sliding time for 3% Al_2O_3 and 1% graphene.

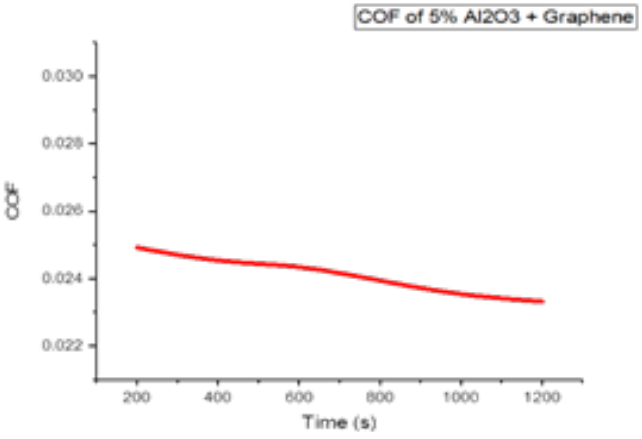


Figure 13. Variation in COF with sliding time for 5% Al_2O_3 and 1% graphene.

3.3 SEM Examination of Wear Surface

3.3.1 Wear Surface Study of Pure Aluminium

The characteristic asperity-to-asperity contact between the sliding surfaces is shown by the worn surface of pure aluminium. Sliding over the softer aluminium surface, the harder asperity (in this case, the steel surface at 62 HRC) forms grooves. The normal wear surface of an aluminium sample is less than 10 N.

3.3.2 Wear Surface Study of Composites

The incorporation of Al_2O_3 particles into an aluminium matrix has been shown to improve the composite's thermal stability. It has a consistent pattern of grooves and pits. This occurs because of the combination of brittle particles and pliable matrix interphases. As the grooves became shallower, it was discovered that increasing the amount of reinforcing material improved the wear resistance of the material. Several particles, detached from the matrix, were also visible on the surface. Surfaces that are 3% and 5% Al_2O_3/graphene composite, as well as their structures, exhibit wear.

4.0 Conclusion

The following are the conclusions:

- Wear, porosity, density, and the microstructure of the Al-Al_2O_3-Graphene composite were examined in this article.
- The sintering temperature and Al_2O_3 both increased the hardness.
- Sample at 550°C affirms the least wear and the least COF when contrasted with 490°C and 520°C.
- Compared to specimens at 0%, 1%, and 3%, wear is minimal at 5% Al_2O_3.
- Due to less wear than at 1%, 3%, and 5% Al2O3, the specimen at 5% Al_2O_3 demonstrates minimal COF.
- Compared to 0%, 1%, and 3%, Al_2O_3 content confirms dense structures at 5% from 450°C to 490°C, density rises.

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