Development and Mechanical Characterisation of Al6061-Al2O3-Graphene Hybrid Metal Matrix Composites

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Abstract: MMC based on aluminium (Al) were produced for light-weight applications especially in aviation and automobile areas. Present paper deals with the fabrication and mechanical performance of AA6061 matrix composites fortified with Al2O3 (alumina) and graphene particulates. Fluid metallurgy method namely stir casting route was employed for fabricating the hybrid composites. Al2O3p and graphene powder are mixed in different weight fractions in which graphene (1 wt. %) particle reinforcement is held consistent and Al2O3 reinforcement is differed freely with 5, 10 and 15 wt. %. Using optical analyser and SEM equipment, microstructural examination is carried out and the result reveals that the graphene and Al2O3 particles prevalently are homogeneously appropriated on the grain limits of Al matrix and Al2O3 particles are disseminated between graphene in the as-cast AA6061 MMC’s. Detailed analysis on investigation of the microstructure and mechanical aspects of Al6061-graphene-Al2O3p composites is presented by following ASTM guidelines; results uncovered that with increment in reinforcement particles, there is an enhancement in the hardness, ultimate strength, yield strength and a decline in the elongation values was however noticed when contrasted with Al6061 alloy. Fractography investigation revealed dimples in unreinforced alloy and the composite.

Keywords: metal matrix composites; Al2O3; graphene; mechanical properties; fractography

1. Introduction

In the current era, a remarkable development in materials in various applications is observed. The standard designing materials do not fit for a few ventures since they belong to unmodified solid materials. However, composite materials have been smart in gathering the requirements for such ventures [1–3]. The material of composites comprises more than one new material that conveys high strength when related to the independent material [4,5]. Researchers are engaged around utilising mixes of forefront materials and framing new procedures to hybrid another arrangement of metal matrix composites (MMCs). The primary explanation behind favouring the Al composite is due to its mechanical properties, higher strength with low weight, which is mostly utilised in aviation projects [6,7]. Matrix 6xxx-related material is a standout amongst the other appropriate materials in the Al family. Amongst them, AA6061 is phenomenal in solidarity to weight proportion which is liked in automotive and aviation industries. Aluminium (Al) metal matrix composites (AMMCs) essentially improved the mechanical properties when contrasted with the as-cast
aluminium alloys. Different reinforcements are utilised in metal matrix to enhance various properties of composites. Aluminium (Al)-based MMCs have been widely concentrated as an attractive decision of auto, aviation and military ventures owing to their high strength with light weight and protection from high temperatures [8–10].

However, the miniature ceramic-reinforced particles, ($\text{Si}_3\text{N}_4$, $\text{B}_4\text{C}$, $\text{TiB}_2$, $\text{AlB}_2$, $\text{SiC}$, $\text{TiC}$, $\text{TiN}$, $\text{Al}_2\text{O}_3$ and so on) are utilised to enhance the ultimate strength and yield strength of the metal [11–13]. Anyway, the malleability of the MMCs breaks down with high ceramic particle concentration. Nano-structural reinforcement (particulate, fibre and sheet and so on) can essentially upgrade the mechanical strength of the base matrix by adequately advancing the fortifying and hardening material than micron size reinforcement [14]. Numerous analysts had recommended that synthesizing aluminium-based composite through fluid metallurgy strategy is financially achievable. However, in the liquid metallurgy route, non-uniform dispersion of the reinforcement presence brings about blow openings and porosity [15].

Lot of research work has been performed to get ready aluminium matrix composites added up with nano reinforcement for improvising the mechanical strength of the matrix. Amongst the diverse particulate reinforcement used, aluminium oxide ($\text{Al}_2\text{O}_3$) is perhaps the most modest material that can hold top caliber at higher temperature and offers splendid wear and mechanical properties. Numerous specialists have chipped away at graphene particles as a reinforcement to acquire magnificent mechanical properties because of its Youngs modulus that ranges from 0.5–1 TPa and owing to its tensile strength [16–18]. Al-Salihi et al. [19] arranged composites Al6061 with $\text{Al}_2\text{O}_3$ nano reinforcement and announced that there was a critical upgrade in the mechanical and hardness properties at an addition of 10 wt. % alumina. Bharath et al. [20], analysed the microstructural portrayal of the Al6061 metal matrix composite with 6, 9, and 12 wt. % of $\text{Al}_2\text{O}_3$ nanoparticles and noticed a uniform appropriation of $\text{Al}_2\text{O}_3$ nanoparticles. G. Sivakaruna [21], analysed the impacts of reinforcement on aluminium metal alloy composites. It was seen that expanding the level of reinforcement brought about the upgrade of mechanical properties and wear resistance. P. V. Rajesh [22] dealt with Al6061-$\text{Al}_2\text{O}_3$ composites utilising stir casting course, and presumed that composite specimens have improved mechanical properties than the aluminium alloy. M. Senthil Kumar [23] dealt on AA2024-$\text{Al}_2\text{O}_3$-$\text{SiC}$-reinforced hybrid composites by employing squeeze casting process, and revealed that hybrid composites showed improvements in mechanical and wear opposition appropriate for motor chamber liner in automobile applications.

Graphene has exceptional properties with high unequivocal surface zone and high Young’s modulus. On account of its decent thermal conductivity property, graphene plays as a nice competitor for the stronghold of Al matrices to redesign the thermal conductivity [24]. Various experts have worked away at conveyance of graphene particulates in aluminium matrix, owing to their interfacial exposure area when contrasted with the other various reinforcement particles with various assembling techniques, since the expansion of graphene particulates into Al alloy matrix impacts both tribological and mechanical behaviour of the composite. The weight % of graphene particles can be chosen depending on the type of application prerequisite. A restriction is observed in handling graphene-related metal matrix composites using stir casting because graphene thoroughly settles down at the base and usually does not blend as expected [25]. Wang et al. [26] revealed that ultimate strength of the aluminium composite reinforced with graphene nano sheets with just 0.3 wt. %, by powder metallurgy is 249 MPa. There was considerable improvement over the as-cast aluminium matrix. Li et al. [27] revealed that the aluminium composite with 0.3 wt. % graphene oxide shows a 18% intensification in elastic modulus strength and 17% in hardness, over as-cast aluminium. Yan et al. [28] detailed that yield and ultimate strength of 0.3 wt. % graphene-strengthened aluminium matrix composite created using powder metallurgy, when contrasted with the unblemished aluminium alloy, shows an increment of 25 and 58%, respectively. Siddhartha et al. [29] revealed the readiness of Al 6061 matrix nanocomposites manufactured using graphene flake and SiC particle by
powder metallurgy and revealed that hardness of composites was better than Al 6061 alloy. Mahmut Can Senal et al. [30] conveyed on the application of graphene as lubricant in Al-based composites. Tabandeh-Khorshid M et al. [31] explored Al matrix synthesised with graphene and concluded that expanding the graphene weight fraction over 1 wt. % in Al matrix diminished the hardness value altogether. Rashad et al. [32], examined the mechanical characteristics of Al-graphene with (0.3 wt. %) mixed by hot extrusion and compaction process. The composite exhibited an expansion of hardness, yield strength when compared with the as-cast unreinforced aluminium. Researcher Shin et al. [33] reported on the sustaining behaviour of multi-layered graphene particle-reinforced Al composites made using hot rolling.

Some exploratory works were detailed over the use of hybrid reinforcement with the expansion of graphene in various forms like particulate, nanosheets and nanoplatelets through powder metallurgy procedure [34,35] and stir casting strategy. Hui-Min et al. [36] announced the readiness of Al 7075 alloy nanocomposites built up with graphene nano plates utilising flash plasma sintering. The outcomes uncovered that hardness of the Al composite expanded by 29%, individually when contrasted with the 7075AA alloys. Researchers [37] worked on aluminium strengthened with silicon nitride and graphene and concluded to see a rise in hardness levels in the synthesised material. Researchers [38] worked on hybrid material built up ZrO$_2$ and graphene particles by employing stir casting route and presumed that expansion in the wt. % of hybrid reinforcement upgraded mechanical properties. Bartolucci et al. [39] dealt with the ball-processing system to powder aluminium (Al) and graphene platelets, trailed by hot isostatic squeezing besides expulsion. Aluminium carbide was reported in the composites and it was reasoned that the hardness reduced because of the carbide arrangement available in the middle of the grains thereby debilitating the tensile strength. Reddy et al. [40] found the uniform spread of graphene and SiC reinforcements in the alloy when synthesised using ultrasonic-aided casting technique. Du X. M. et al. [41] arranged hybrid composites using graphene and SiC particles with Al7075 utilising powder metallurgy and revealed that graphene and SiC nanoparticles prevalently are homogeneously appropriated on the grain limits of Al matrix and SiC nanoparticles are conveyed between graphene sheets. There was a critical expansion in wear opposition in the composites thus formed.

It has been seen that, there is not enough literature work on hybrid composites involving graphene and ceramic Al$_2$O$_3$ particle invigorated MMCs to update the mechanical and wear conduct of aluminium-related composites. Subsequently, in the current research paper, an endeavour is made to contemplate and create the impact of the Al$_2$O$_3$ and graphene particle embedded in the Al6061 alloy.

Amongst several techniques like squeeze casting, pressure infiltration, electro plating, electro forming, foil deposition, stir casting course is chosen in the present research to synthesise composites since it is the most practical technique for large-scale manufacturing and avoids agglomeration of Al$_2$O$_3$ and graphene.

The impact of graphene and Al$_2$O$_3$ particle addition on the microstructure and mechanical properties namely yield strength, hardness, and tensile strength is studied.

2. Fabrication Procedure

2.1. Matrix and Reinforcement Material

Al6061 hybrid metal matrix composites (Al6061-graphene-Al$_2$O$_3$$_{5p}$) was set up by liquid metallurgy course. For the current assessment, Al6061 alloy was picked as matrix material in light of its wide extent of employments in auto sector and avionics industries. Al$_2$O$_3$ particles (5, 10 and 15 wt. %) and graphene (1 wt. %) were used as reinforcements for synthesizing hybrid composites. Al6061 composite billets were procured from Fenfee Metallurgical Private Limited, Bengaluru Karnataka, India. An adequate amount of reinforcement material for the current assessment is chosen as Al$_2$O$_3$ in light of its properties like higher hardness with extraordinary wear resistance. Al$_2$O$_3$ particles with 40-micron
size are acquired from United Nanotech Innovations Private Limited, Bengaluru, India and graphene was procured from BT Corp, Hoskote, Bangalore, Karnataka, India.

2.2. Composites Preparation

Prior to adding Al 6061 billets into the crucible, the billets are preheated in a muffle furnace to eliminate dampness. Prior to this, it was appropriately salted in NaOH solution to eliminate residue and oil. A known amount of AA6061 alloy billets was dissolved in a crucible in an electrical resistance heater to a required temperature range of 660–670 °C and subsequently superheated. In order to acquire distinctive weight level of hybrid composites, a known amount of Al₂O₃ powder and powder graphene was gauged, carefully stuffed in an aluminium (Al) foil and introduced into the molten melt; reinforcement was preheated or oxidised in a muffle heater at 400 °C for 150 min to eliminate dampness from particles and increase the wettability in as-cast matrix material; this aide the joining of the reinforcement particles while decreasing undesired interfacial reactions. A suitable flux was introduced to the liquefy for confining oxidation. This was also to eliminate the dissolved gases present in the liquid metal amid dissolving process. Hexachloroethane tablets (C₂Cl₆) were subsequently used as degasification tablets. Mixing was persistently carried out at normal stretches utilising a graphite material rod. The temperature of the molten melt was kept up to 800 °C and continually observed utilising a computerised temperature regulator. After the melt is homogenised, the liquefy is filled in the cast iron die and allowed to cool. The cast specimens were subsequently machined according to ASTM norms aimed at different microstructure and mechanical tests. Microstructures and essential basic examination were considered utilising a scanning electron magnifying lens equipment. Test specimens were cut from the lower part of the projecting, utilising a hacksaw blade, grinding was done on an emery paper accompanied by cleaning, utilising an alumina solution. The specimen was dried up using a hot dry air oven for 12 h for removing dampness. The specimen is then kept in an etching solution containing HCl, H₂SO₄ and H₂O.

The surface morphologies were beheld by a scanning electron microscopy (SEM) furnished with energy dispersive spectrometer (EDS) and an optical magnifying instrument (OM) related to an image analyser (Nikon Epiphot 200). Hardness values of composite specimen were checked on a Brinell hardness testing machine (HBW 10/1000, RSML, Bangalore, India), utilising a specified load and the mean normal average values of at least five values were considered at various regions of each specimen.

Tensile strength was assessed using modernised computerised pressure hydraulic-driven universal testing machine (TUE-C-1000-RSML, Bangalore, India) with a maximum limit of 200 KN according to ASTM standards. The sample was arranged and set into the machine so much that the cross head basically reached the surface of the cylindrical shaped moulded sample. Required loads were applied on the testing specimen and consistently extended from zero to up to cross-head and voyaged a distance for deformity through a velocity of 1m/s. The value recorded was the normal average of three trial tests conducted on the tensile specimens.

3. Results and Discussions

3.1. Microstructural Analysis

To understand the morphologies of reinforcement particulates, optical (Figure 1) images are used. The optical micrographs of the unadulterated Al6061 and the composites with graphene and Al₂O₃ particles are addressed in Figure 1a–d. Figure 1a depicts the optical images of the Al alloy utilised, which displays the dendritic structure. The optical micrograph of the as-cast combination shows the base construction with α-Al stage, and shows both inside and outside of the grain. The grains have equiaxed shape. The bone like Mg₂Si is seen in the matrix. As far as possible it seems to present a dendritic construction as in the images of Figure 1a–d. Because of the presence of Al₂O₃ in the hybrid composites as a result of homogeneity nucleation in the matrix it prompts more modest grains as seen in the Figure 1b,c. In the Al6061-graphene-Al₂O₃p composites, it obviously shows the
development of refined and smaller grains in contrast with monolithic alloy. From the optical micrographs of the composites, the greyish white particulates refer to the Al$_2$O$_3$ particulates, while the graphene particulates are viewed as dull greyish particulates. The shape of the particulates is normally found as round and some plate-like particles are also seen. Graphene and Al$_2$O$_3$ are consistently found on the aluminium composites and for the most part, particles are in grain limits [38,42,43].

Figure 1. Optical images of (a) Al6061; (b) Al6061 + 1 wt. % Graphene + 5 wt. % Al$_2$O$_3$ composites; (c) Al6061 + 1 wt. % Graphene + 10 wt. % Al$_2$O$_3$ composites; (d) Al6061 + 1 wt. % Graphene +15 wt. % Al$_2$O$_3$ composites.

Figure 1b–d depicts optical image of the specimen where a thick stage microstructure with restricted amount of porosity is noticed. A predominant holding in between the reinforcement particulates and lessening in porosity might provoke an increase in the load-bearing restriction of the composite’s specimen tests. The appropriation of dark Al$_2$O$_3$ particles might be likely identified with the higher thermal conductivity of the cast iron mould that upgrades cementing measure. Expanding the graphene substance would presumably build the quantity of agglomerates and porosity would likewise increase. Additionally, there is a significant conflict between aluminium and graphene/Al$_2$O$_3$, which may result in debonding at the inter-face. As a result of its incredibly higher conductivity, graphene particle might be significantly tolerant interestingly due to the enveloping microstructure of the composite specimen. The dendritic form microstructure is exhibited as a result of the reaction between the Al and graphene/Al$_2$O$_3$ particles probably due to the carbide phase that may have been furthermore represented [44,45].

Figure 2 shows the SEM picture of as cast alloy revealing the white α phase. Fine grains are found dissipated along as far as possible in the structure of Al6061 alloy [38,44–52].
3.2. Mechanical Properties

Table 1 depicts the mechanical properties of the fabricated AA6061-Al2O3-graphene composites.

| Sl. No | Base Matrix | Reinforcement | Specimen Code | Ultimate Tensile Strength | Yield Strength | % of Elongation | Hardness |
|--------|--------------|---------------|---------------|---------------------------|----------------|----------------|----------|
| 1      | Al6061       | -             | Al            | 117.36 ± 3.23             | 82.88 ± 2.63   | 11.23 ± 1.96   | 51.4 ± 3.23 |
| 2      | Al6061       | 1 wt. % Graphene + 5 wt. % Al2O3 | Al-1G-5ALU | 128.36 ± 2.87             | 91.23 ± 2.12   | 5.18 ± 1.26    | 57.6 ± 2.23 |
| 3      | Al6061       | 1 wt. % Graphene + 10 wt. % Al2O3 | Al-1G-10ALU | 157.98 ± 4.23             | 108.23 ± 3.65  | 4.62 ± 1.29    | 69.56 ± 3.24 |
| 4      | Al6061       | 1 wt. % Graphene + 15 wt. % Al2O3 | Al-1G-15ALU | 190.23 ± 3.87             | 142.21 ± 2.98  | 4.12 ± 1.06    | 86.92 ± 3.15 |

It is seen that with the existence of Al2O3 and graphene particulates in the base matrix, the ultimate tensile strength and yield strength of the developed composites increased that confirms the clear and uniform scattering of reinforced particles upheld in the Al MMC.

3.2.1. Tensile Strength

Tensile tests were carried out to evaluate the mechanical characterisation of the synthesised composites. Figure 3 shows the outcome of aluminium oxide (Al2O3) and graphene particle reinforcement on the UTS of the AA6061 composites. Due to the existence of graphene and expanding wt. % of Al2O3 particulates, the UTS of the composites increased. UTS of as-cast section of aluminium combination AA6061 is 117MPa and this augments to a limit of 190MPa for the matrix of Al-1G-15ALU composites and exhibits 62.09% additions over the unreinforced aluminium. The UTS, yield strength of the as-cast alloy was found to be lesser than predicted and may be attributed to some porosity defects during casting.
The development in the UTS may be attributed to the existence of hard Al₂O₃ particulates and graphene particle reinforcement, due to the nearby pressing and uniform transport of reinforcement, thus resulting in lesser intermolecular spaces. The types of holding at the inter-face of aluminium matrix composites amid atomic blending are important due to the useful gatherings on the carbon and Al₂O₃ particles which is valuable to the movement of load among matrix and the reinforcement. Inter-faces and solid interfacial holding amongst graphene and Al₂O₃ particle reinforcement in the AA6061 metal alloy assume a significant part in deciding tensile strength of the composites [50,53].

### 3.2.2. Yield Strength (YS)

With the tensile test, it is noticed that the YS of the composites is more critical than as-cast Al alloys (Figure 4). The applied load transfers to the insistently invigorated Al₂O₃p and graphene particulates in Al alloy. It is additionally obvious that the extended separation density is identified with the matrix-reinforcement-interface. The additional plausible explanation remains ascribed to the grain-refining fortifying component. Strength increments progressively with the expansion in wt. percentage levels of Al₂O₃ and graphene particle reinforcement in the as-cast material. This upgrade of solidarity can be grasped by various means that include Orowan, load transfer and dislocation strengthening, where existence of both reinforcements, graphene and Al₂O₃ particles have solid grip, great holding, close packing, clear and uniform appropriation of reinforcement inside the as-cast material [50,53,54].

With an ascent in weight part level, it is conceivable to move more loads to the reinforcement, which additionally adds to all the more likely yield strength.
3.2.3. % of Elongation

Figure 5 depicts the graphs of % of elongation with weight % of reinforcement. Results uncover that, while expanding the reinforcement stage of Al_2O_3 and graphene particulates, the percentage of elongation of the composite specimen declines, chiefly credited to the existence of hard ceramic substance of Al_2O_3 and graphene particulates inside the composites [50,53–55].

Figure 5. % Elongation variation in hybrid Al-Al_2O_3-graphene composites.

3.2.4. Hardness

Hardness values were estimated for Al6061 alloy and Al6061-Al_2O_3-graphene composites. Hardness of the composites improves with the expanding reinforcement content in the matrix. This is due to the hard particles inserted in the matrix that take up the load of the indenter. The particulates act against the disengagements when load is applied. Reinforcing particles are more diligent and stiffer than the matrix that fabricates restriction to plastic distortion of matrix all through the testing procedure [56].

The outcomes are summed up in Figure 6. With the increment of the Al_2O_3 substance, the hardness of the hybrid composites improves. The base alloy recorded 51.4.0 ± 3.23 as BHN, while the hardness of Al-1G-15ALU composites was 86.92 ± 3.15 BHN, showing 69.10% additions over the unreinforced aluminium alloy. The organised graphene and hard Al_2O_3 can hinder grain development in aluminium matrix through grain limit stick- ing and consequently leading to better grain construction of aluminium. Better grain constructions can bring about higher hardness values [38,57].

Figure 6. Hardness variation in hybrid Al-Al_2O_3-graphene composites.
The ductility of the Al alloys composite is influenced by strengthening. Because of void nucleation, the decline in ductility might be attributed to the expanding reinforcement content.

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![Hardness variation in hybrid Al6061-Al2O3-graphene composites.](image)

Hybrid composites revealed improvements in hardness due to the presence of ceramic content and graphene in the matrix. The reinforcements are relatively stronger than the matrix of the alloy. Alumina particles proved to be more stable and prompted the increase in imperative to plastic distortion of the lattice during the process of indentation. The increase in tensile strength of the composite is due to the α-Al dendrites that take the support of the reinforcement while they are included in the matrix. The ductility decreased due to the fact that the ceramic particle had lower flexibility when compared with matrix.

The changes in morphology modification of the matrix have resulted in variations in tensile strength [58]. The thermal mismatch between the matrix and reinforcement is responsible for increasing the dislocation density of the matrix and hence results in improvement in strength of the composite. Hardness improvements are also ascribed
to lesser amount of porosity and uniform distribution of reinforcement throughout the matrix [59].

4. Fractography

SEM assessment of the fractured surfaces uncovered a dimpled break surface for the as-cast material. The fracture surface of the as-cast material contained a somewhat lopsided dissemination of enormous dimples associated with sheets of more modest dimples (Figure 7a at a magnification of 800X).

The fractured surface of the reinforced particles material composites contained just more modest dimples than the fractured surface of the as-cast material. The fracture surfaces additionally display fine and shallow dimples, demonstrating that the break is flexible, as demonstrated in Figure 7a. Yet, higher level of graphene-$\text{Al}_2\text{O}_3$ hybrid composites showed weak fracture failure as shown in Figure 7b–d at a magnification of 700, 400 and 1500 X. The failure showed not many fracture splits longitudinally and transitionally as demonstrated in Figure 7c–d. The reason of failure in the composite might be ascribed to the increment in weight/load on the sample [44,47,60].

The fracture morphology of the hybrid composites is delineated in Figure 7b–d. They have similar boundaries, with the exception of the presence $\text{Al}_2\text{O}_3$ content. Moreover,
graphene particle is implanted along the grain limits. From Figure 7a, it is observed that a few pores in composites are without graphene and Al₂O₃p; other side pores can give spaces to the development of grains, which prompts strange grain development and inhomogeneous grain size. Further, micro fractures normally happen close to the pores, and they have an incredible unfavourable impact on the ceramic properties [44,47–61]. Accordingly, decreasing porosity is useful in enhancing the mechanical behaviour of the composites. As the load on the fracture expands, it actuates strain in the particles (Figure 7c,d), and the most intensely stacked break fractures [62]. Some break ‘pullout’ has occurred in the specimen, however the fracture gives off an impression of being at the base matrix end and not at the interfacial zones, as demonstrated by the tapered depressions with undulated surface. Obviously more ‘pullout’ occurred in the alloy composite because of the low strength of the base matrix [44,47,60,63]. The fracture method of the matrix combination which converted from tensile to cleavage type (on account of MMCs) was ruled with micro fracture nucleation as demonstrated in Figure 7b–d.

It is accepted that graphene is consistently dispersed throughout the grain limits, controlling inordinate grain development [64]. Uniform dispersion of graphene successfully improves the densification of ceramic production because of its fine properties [60,65,66]. A few pores are shaped between graphene and Al₂O₃ matrix, giving spaces for grains to develop (Figure 7b–d). Graphene particle cluster might have prompted a decreased ‘binding effect’ along the grain limits, bringing about an expansion in grain sizes. To acquire better mechanical behaviour of materials, suitable graphene wt. % content should be used amid fabrication [38,60]. Hence, the graphene content is fixed at 1 wt. % in the research study. Furthermore, as demonstrated in Figure 7b–d, a substituting dissemination of solid and frail holding interfaces instigated by graphene and Al₂O₃p is noticed, which is helpful in improving the mechanical properties.

However, Al₂O₃p agglomerates throughout the grain limits of the matrix (Figure 7c,d) with the expansion of Al₂O₃ content, making it more inclined to aggregate spalling. Consequently, the mechanical behaviour diminishes with the increasing Al₂O₃p. The thermal development coefficients of Al₂O₃p and graphene are distinctively separate. In this way, modest quantity of graphene is encased by enormous Al₂O₃ grains or appropriated along grain limits. In the interim, if the Al₂O₃p is in the break engendering way, the break can without much of a stretch arrive at the graphene-Al₂O₃p/matrix interface. The fracture might be stuck if the external force does not build, which is the hardening component related to “crack pinning”. In the event that the outside load keeps on expanding, the failure could go through the grains, prompting transgranular break, or the break direction will keep on growing along the grain limits, prompting intergranular fractures. It is observed that fracture counters are smooth and levelled, demonstrating the presence of transgranular break. Consequently, it is presumed that the break modes are a blend of both transgranular and intergranular fracture [60,67–69]. Upon modification, graphene was termed as a functional material by many researchers [70,71] and the effect of adding graphene in the matrix material is discussed in the present research.

MMCs could have been fabricated through a variety of ways like powder metallurgy [72,73], diffusion bonding, infiltration techniques [58,59], chemical vapour deposition techniques, stir casting etc. Since stir casting is relatively simple and an inexpensive method, the present work employed stir-casting technique to effectively prepare hybrid composites.

In the present work, the synthesised hybrid composites have shown better mechanical and physical properties compared to pure aluminium and rightly belong to a new class of engineering materials. They may be termed as second-generation composites in which more than one shape, type and size of reinforcements could be used to attain desirable properties. Hybrid composites need to be preferred since they have better properties when contrasted with single reinforced composites as they combine the advantages of the constituent reinforcements.
5. Conclusions

An exact report on the combination of microstructural portrayal of graphene and Al₂O₃-sustained AA6061 MMCs is represented in the present research paper. The experimental results were inspected essentially and critical facts were drawn:

- Composites were viably manufactured using Al₂O₃ and graphene particulates in the aluminium combination using fluid metallurgy route.
- Optical images show the significance of Al₂O₃ and graphene reinforcement, reliably dispersed through improved closed packing besides incredible holding bond amid particulates in the matrix alongside a few proportions of pores.
- Tensile strength and yield strength of the composite extended with the graphene support when compared to as-cast alloy. The increment in the mechanical strength is credited with the harder Al₂O₃ particles/graphene which delivers strength to the composites.
- % of elongation lessens with enhancement in weight % of reinforcement, however due to the hard particulate’s existence, brittleness occurs that diminishes the ductility of the composites. This may likewise be credited to the harder ceramic Al₂O₃p and graphene particles.
- Al6061 composites displayed higher hardness than unadulterated Al6061alloy. The hardness expanded by about 69.10% with the reinforcement of 1 wt. % graphene and 15 wt. % Al₂O₃p.
- The fracture was pliable with dimple surface showing particle debonding and particle breaking. With fractography, the failure of the composites appeared to comprise transgranular break of the graphene–Al₂O₃p reinforcement and ductile fracture of the AA6061 base matrix.

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References

1. Dayanand, S.; Babu, S.; Auradi, V. Experimental Investigations on Microstructural and Dry Sliding Wear Behavior of Al-AlB₂ Metal Matrix Composites. Mater. Today Proc. 2018, 5, 22536–22542. [CrossRef]
2. Yang, J.-G.; Ou, B.-L. Influence of microstructure on the mechanical properties and stress corrosion susceptibility of 7050 Al-alloy. Scand. J. Met. 2001, 30, 158–167. [CrossRef]
3. Dayanand, S.; Boppana, S.B.; Hemanth, J.; Telagu, A. Microstructure and Corrosion Characteristics of In Situ Aluminum Diboride Metal Matrix Composites. J. Bio Tribocorros. 2019, 5, 60. [CrossRef]
4. Boppana, S.B.; Dayanand, S. Impact of Heat Treatment on Mechanical, Wear and Corrosion Behaviour of In Situ AlB₂ Reinforced Metal Matrix Composites Produced by Liquid Metallurgy Route. J. Bio Tribocorros. 2020, 6, 33. [CrossRef]
5. Boppana, S.B.; Dayanand, S. Development of AlB₂ particles using inorganic halide salts and mechanical characterization of AlB₂ reinforced AA6061 MMC’s. Mater. Today Proc. 2020, 27, 595–602. [CrossRef]
6. Boppana, S.B.; Dayanand, S.; Ramesh, S.; Auradi, V. Effect of Reaction Holding Time on Synthesis and Characterization of AlB₂ Reinforced Al6061 Metal Matrix Composites. J. Bio Tribocorros. 2020, 6, 88. [CrossRef]
7. Dayanand, S.; Babu, B.S. A Review on synthesis of AlB₂ reinforced aluminium matrix composites. IOP Conf. Ser. Mater. Sci. Eng. 2020, 810, 12038. [CrossRef]
8. Dayanand, S.; Boppana, S.B.; Auradi, V.; Nagaral, M.; Ravi, M.U. Bharath Evaluation of Wear Properties of Heat-Treated Al-AlB₂ in-situ Metal Matrix Composites. J. Bio Tribocorros. 2021, 7, 1–11. [CrossRef]
9. Samuel, D.; Boppana, S.B.; Palanikumar, K.; Ramesh, S.; Auradi, V. Role of Heat Treatment on Hardness of Al 6061- AlB₂ Metal Matrix Composites. Int. J. Surf. Eng. Interdiscip. Mater. Sci. 2021, 9, 26–39. [CrossRef]
10. Kumar, V.; Nagegowda, K.U.; Boppana, S.B.; Sengottuvelu, R.; Kayaroganam, P. Wear behavior of Aluminium 6061 alloy reinforced with coated/uncoated multiwalled carbon nanotube and graphene. J. Mater. Miner. 2021, 31, 17–24.
11. Bharath, V.; Auradi, V.; Nagaral, M.; Dayanand, S.; Boppana, S.B. Impact of Alumina Particulates Addition on Hardness and Wear Behaviour of 2014 Al Metal Matrix Composites by Vortex Method. IOP Conf. Ser. Mater. Sci. Eng. 2021, 1013, 012018. [CrossRef]
12. Nagaral, M.; Deshapande, R.G.; Auradi, V.; Boppana, S.B.; Dayanand, S.; Anilkumar, M.R. Mechanical and Wear Characterization of Ceramic Boron Carbide-Reinforced Al2024 Alloy Metal Composites. J. Bio Trib. Corros. 2021, 7, 19. [CrossRef]

13. Boppana, S.B. In Situ Synthesis of Titanium Carbide in Pure Aluminium. J. Mater. Sci. Chem. Eng. 2020, 8, 1–10. [CrossRef]

14. Boppana, S.B.; Chennakeshavalu, K. Preparation of Al-5Ti Master Alloys for the In-Situ Processing of Al-TiC Metal Matrix Composites. J. Miner. Mater. Charact. Eng. 2009, 8, 563–568. [CrossRef]

15. Dayanand, S.; Satish, B.B. Effect of Cryolite on Microstructure of Insitu AlB3 Aluminium Metal Matrix Composites. Int. J. Recent Technol. Eng. (IJRTE) 2019, 8, 940–943. [CrossRef]

16. Yang, B.; Sun, M.; Gan, G.; Xu, C.; Huang, Z.; Zhang, H.; Fang, Z.Z. In situ Al2O3 particle-reinforced Al and Cu matrix composites synthesized by displacement reactions. J. Alloys Compd. 2010, 494, 261–265. [CrossRef]

17. Yang, Y.; Zhang, Z.; Zhang, X. Processing map of Al2O3 particulate reinforced Al alloy matrix composites. Mater. Sci. Eng. A 2012, 558, 112–118. [CrossRef]

18. Jawalkar, C.S.; Verma, A.S.; Suri, N.M. Fabrication of Aluminium Metal Matrix Composites with Particulate Reinforcement: A Review. Mater. Today Proc. 2017, 4, 2927–2936. [CrossRef]

19. Al-Salihi, H.A.; Judran, H.K. Effect of Al2O3 reinforcement nanoparticles on the tribological behaviour and mechanical properties of Al6061 alloy. AIMS Mater. Sci. 2020, 7, 486–498. [CrossRef]

20. Bharath, V.; Nagaral, M.; Auradi, V.; Kori, S. Preparation of 6061Al-Al2O3 MMC’s by Stir Casting and Evaluation of Mechanical and Wear Properties. Procedia Mater. Sci. 2014, 6, 1658–1667. [CrossRef]

21. Sivakaruna, G.; Babu, D.P.S. A survey on effects of reinforcement on aluminium metal matrix composites. Int. J. Mech. Eng. Technol. 2017, 8, 112–131.

22. Rajesh, P.V.; Sriman, P.M. Optimization of process parameters of Aluminium Alloy (Al6061)-Alumina (Al2O3) composites fabricated by stir casting. Int. J. Eng. Res. Manag. Stud. 2018, 5, 2394–7659.

23. Senthil, K.M.; Managalara, R.V.; Senthil Kumar, K.; Natrayan, L. Processing and characterization of AA2024/Al2O3/SiC reinforces hybrid composites using squeeze casting technique. Iran J. Mater. Sci. Eng. 2019, 16, 55–67.

24. Li, F.; Long, L.; Weng, Y. A Review on the Contemporary Development of Composite Materials Comprising Graphene/Graphene Derivatives. Adv. Mater. Sci. Eng. 2020, 2020, 1–16. [CrossRef]

25. Venkatesan, S.; Xavior, M.A. Characterization on aluminum alloy 7050 metal matrix composite reinforced with graphene nano particles. Procedia Manuf. 2019, 30, 120–127. [CrossRef]

26. Wang, L.Z.; Fan, G.; Pan, H.; Chen, Z.; Zhang, D. Reinforcement with graphene nanosheets in aluminum matrix composites. Scr. Mater. 2012, 66, 594–597. [CrossRef]

27. Li, Z.; Fan, G.; Tan, Z.; Guo, Q.; Xiong, D.; Su, Y.; Li, Z.; Zhang, D. Uniform dispersion of graphene oxide in aluminum powder by direct electrostatic adsorption for fabrication of graphene/aluminum composites. Nanotechnology 2014, 25, 325601. [CrossRef] [PubMed]

28. Yan, S.J.; Cheng, Y.; Hu, H.Q.; Zhou, C.J.; Bo, L.D.; Long, D.S. Research of Graphene-Reinforced Aluminum Matrix Nanocomposites. J. Mater. Eng. 2014, 4, 1–6.

29. Siddhartha, J.; Prashantha, K.H.G.; Anthony, M.X. Synthesis and characterization of AA 6061-graphene-SiC hybrid nanocomposites processed through microwave sintering. IOP Conf. Ser. Mater. Sci. Eng. 2016, 149, 12086.

30. Şenel, M.C.; Gürbüz, M.; Koç, E. Mechanical and tribological behaviours of aluminium matrix composites reinforced by graphene nanoplatelets. Mater. Sci. Technol. 2018, 34, 1980–1989. [CrossRef]

31. Tabandeh-Khorshid, M.; Omrani, E.; Menezes, P.L.; Rohatgi, P.K. Tribological performance of self-lubricating aluminum matrix nanocomposites: Role of graphene nanoplatelets. Eng. Sci. Technol. Int. J. 2016, 19, 463–469. [CrossRef]

32. Rashad, M.; Pan, F.; Tang, A.; Asif, M. Effect of Graphene Nanoplatelets addition on mechanical properties of pure aluminum using a semi-powder method. Prog. Nat. Sci. 2014, 24, 101–108. [CrossRef]

33. Shin, S.; Choi, H.; Shin, J.; Bae, D. Strengthening behavior of few-layered graphene/aluminum composites. Carbon 2015, 82, 143–151. [CrossRef]

34. Sethuram, D.; Koppad, P.G.; Shetty, H.; Alipour, M.; Kord, S. Characterization of graphene reinforced Al-Sn nanocomposite produced by mechanical alloying and vacuum hot pressing. Mater. Today Proc. 2018, 5, 24505–245014. [CrossRef]

35. Bastwros, M.; Kim, G.Y.; Zhu, C.; Zhang, K.; Wang, S.; Tang, X.; Wang, X. Effect of ball milling on graphene reinforced Al6061 composite fabricated by semi-solid sintering. Compos. B Eng. 2014, 60, 111–118. [CrossRef]

36. Hui-min, X.; Zhang, L.; Yong-chao, Z.; Na, L.; Yu-qi, S.; Ji-dong, Z.; Hui-zhong, M. Mechanical properties of graphene nanoplatelets reinforced 7075 aluminum alloy composite fabricated by spark plasma sintering. Int. J. Miner. Metall. Mater. 2020, 27, 1295–1300.

37. Şenel, M.C.; Gürbüz, M.; Koç, E. Fabrication and characterization of aluminum hybrid composites reinforced with silicon ni-tride/graphene nanoplatelet binary particles. J. Compos. Mater. 2019, 53, 4043–4054. [CrossRef]

38. Boppana, S.B.; Dayanand, S.; Kumar, A.; Kumar, V.; Aravinda, T. Synthesis and characterization of nano graphene and ZrO2 reinforced Al 6061 metal matrix composites. J. Mater. Res. Technol. 2020, 9, 7354–7362. [CrossRef]

39. Bartolucci, S.F.; Paras, J.; Rafiee, M.A.; Rafiee, J.; Lee, S.; Kapoor, D.; Koratkar, N. Graphene–aluminum nanocomposites. Mater. Sci. Eng. A 2011, 528, 793–7937. [CrossRef]

40. Reddy, A.P.; Krishna, P.V.; Rao, R.N. Tribological Behaviour of Al6061–2SiC-xGr Hybrid Metal Matrix Nanocomposites Fabricated through Ultra-sonically Assisted Stir Casting Technique. Silicon 2019, 11, 2853–2871. [CrossRef]
70. Bellucci, S.; Kruchinin, S.; Repetsky, S.P.; Vyshyvana, I.G.; Melnyk, R. Behavior of the Energy Spectrum and Electric Conduction of Doped Graphene. *Materials* 2020, 13, 1718. [CrossRef]

71. Repetsky, S.P.; Vyshyvana, I.G.; Kruchinin, S.P.; Bellucci, S. Influence of the ordering of impurities on the appearance of an energy gap and on the electrical conductance of graphene. *Sci. Rep.* 2018, 8, 9123. [CrossRef]

72. Bahrami, A.; Soltani, N.; Pech-Canul, M.; Gutierrez, C.A. Development of metal-matrix composites from industrial/agricultural waste materials and their derivatives. *Crit. Rev. Environ. Sci. Technol.* 2015, 46, 143–208. [CrossRef]

73. Deaquino-Lara, R.; Soltani, N.; Bahrami, A.; Gutierrez, E.; Garcia-Sánchez, E.; Hernandez-Rodriguez, M. Tribological characterization of Al7075–graphite composites fabricated by mechanical alloying and hot extrusion. *Mater. Des.* 2015, 67, 224–231. [CrossRef]