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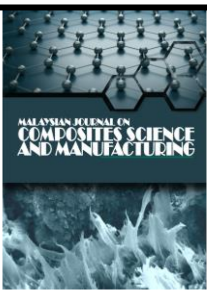

Influence of Graphene on the Microstructure and Mechanical Properties of Aluminium Matrix Composite

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ABSTRACT

The constraints of conventional aluminum alloys have prompted the investigation of strengthening substances such as carbon nanotubes and graphene fillers. The improved composite exhibits suitability for applications in the automotive and aviation industries. Graphene's popularity stems from its high strength, electrical and thermal conductivity, and chemical inertness, making it ideal for mechanical, thermal, and microstructural applications. This research varied concentrations of graphene nanoplatelets (0.3%, 0.6%, and 0.9%) to enhance the mechanical properties of aluminum metal matrix composites. A stir casting process was employed to create a graphene-reinforced aluminum matrix composite using A356 aluminum alloy. A designed experiment (DOE) investigated the impacts of graphene concentration and suitable heat treatment time on the aluminum composite. Subsequently, the specimens underwent heat treatment and X-ray diffraction (XRD). Mechanical properties were examined using a universal testing machine. The best aluminum matrix composites were produced with 0.9wt% graphene and 180 minutes of heat treatment. These parameters resulted in a microstructure with refined grains evolving from dendritic to rosette. The grains became closely packed, and reduced porosity was observed. As a result, the mechanical properties were enhanced, with a maximum ultimate tensile strength (UTS) of 250 MPa and a fracture elongation of 6%. The findings indicate that graphene nanoplatelet (GNP) concentration significantly influences the mechanical characteristics of the composite. Tensile and yield strength increase with GNP concentration, but higher concentrations reduce the composite's ductility. These insights contribute to optimizing GNP-reinforced composites and developing innovative materials with superior mechanical properties.

Keywords:
Semi-solid processing, Aluminum matrix composite, Graphene nanoplatelets

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1. Introduction

Graphene consists of carbon atoms bound in an sp2 configuration, forming a perfect two-dimensional lattice with exceptional properties. The fracture strength, measured at 125 GPa, and Young's modulus exhibit remarkably high values, approaching 1 TPa [1]. The thermal conductivity of suspended single-layer graphene was measured by Zhang *et al.* [2] to be very high at 5000 $Wm^{-1}K^{-1}$. These criteria illustrate the preeminence of graphene as a material for managing heat. Khanna *et al.* [3] had wrote a review on the effect various types of graphene and evaluated for their suitability as fillers in metal-matrix composites. Graphene nanoplatelets (GNPs) are very suitable nanofillers for producing composites with an aluminum matrix due to their distinctive form, which consists of numerous graphene sheets arranged in a stack-like. Due to its remarkable attributes like as outstanding mechanical strength, high carrier mobility, excellent electrical and thermal conductivity, chemical inertness, and linear dispersive electronic structure, graphene, which is only one atom thick, has attracted significant attention. The current scientific investigations on graphene have primarily concentrated on its correlation with superconductivity. In the context of superconducting graphene superconductor junctions, the superconducting properties of graphene are enhanced through its connection with the twisted graphene beneath or by the proximity effect.

The constraints of conventional aluminum alloys have prompted the investigation of strengthening substances such as carbon nanotubes and graphene fillers, which have demonstrated beneficial effects on the mechanical characteristics of aluminum matrix composites [4]. To meet the demands of future automotive and aerospace applications, producing composite materials necessitates more significant amounts of material than what can currently be obtained using graphene synthesis methods. Therefore, using graphene nanoplatelets (GNPs) is a promising solution for attaining the desired properties. Graphene's remarkable mechanical properties make it a compelling option for improving the mechanical performance of aluminum composites. GNPs have the potential to be used as nanofillers in aluminum-matrix composites because of their layered structure and ability to maintain important mechanical properties. This is because graphene possesses extraordinary mechanical properties, which greatly influence the mechanical performance of aluminum composites. Thus, it is possible to generate a single or several layers of graphene using this method. Significant quantities of graphene have also been synthesized under carefully regulated conditions. Recent investigations have revealed that the features are significantly influenced by the type of reinforcement, its weight percentage, and its distribution within the metal matrix [5–8]. It is crucial to ensure that the distribution of the reinforcing material inside the matrix is uniform to optimize the properties of the aluminium matrix composite (AMCs). Many mixing techniques have been employed to ensure a uniform distribution of reinforcements inside the metal matrix, such as ball milling, molten or liquid metal processes, and solution-aided mixing. Ball milling is a technology that has attracted considerable attention because of its substantial impact on the microstructure of the result. When applied at the optimal working conditions of the feed material, it enhances the affinity between the reinforcement and the metal matrix, resulting in uniformly dispersed reinforcements.

The production method also affects the final features of the AMCs. They can be produced by many techniques, such as casting, thermomechanical processing, and powder metallurgy (PM). The stir-casting process has emerged as the most practical technology due to its simplicity and low cost. Chak *et al.* [9] studied the effects of GNPs reinforced aluminum composite on microstructure and mechanical properties through ultrasonic assisted stir casting technique. It had been found that adding graphene to the base matrix led to grain refinement and a significant enhancement of around 37% in ultimate tensile strength and 27% in microhardness of the cast composites. The increase in mechanical characteristics was attributed to graphene's strength and grain-refining action. Heat treatment results in an additional enhancement of approximately 83% and 34% in the ultimate tensile strength and microhardness of the manufactured composites compared to the heat-treated base matrix. The improvement in the characteristics of the heat-treated samples was ascribed to age hardening.

Nevertheless, the constraints of conventional aluminum alloys have prompted the investigation of strengthening substances such as carbon nanotubes and graphene fillers. These substances have demonstrated favorable effects on the mechanical characteristics of aluminum matrix composites. Thus, to meet the demands of future automotive and aerospace applications, producing composite materials necessitates larger amounts of material than what can currently be obtained using graphene synthesis methods. Therefore, using GNPs is a promising solution for attaining the desired properties. Numerous studies have examined the impact of graphene content on the matrix's mechanical characteristics and microstructural evolution. Nevertheless, the study employs distinct methodologies and fabrication techniques, making it rather arduous to determine the optimal graphene composition.

In this present work, the mechanical properties and the microstructural evolution of GNPs GNP-reinforced MMCs fabricated through mechanical stirring were investigated as stated in the methodology. Then, the effect of using different GNPs contents and duration of heat treatment were studied to find the optimum mechanical properties of the matrix composite.

2. Methodology

This study implements the Taguchi Method by utilizing orthogonal arrays to systematically evaluate all design factors while reducing the quantity of executed tests. The parameter and design summary of Taguchi Methods were summarized in Table 1 and Table 2. Fabricating the AMCs involves applying the Design of Experiment (DOE) methodology, wherein various weight percentages (wt%) of graphene and durations of heat treatment are incorporated. This process is carried out using the stir casting technique, as outlined in Table 1.

Table 1
The condition of the parameter

Input Parameter	Level 1	Level 2	Level 3
GNP (Wt-%)	0.3	0.6	0.9
Heat Treatment (min)	60	120	180

Table 2
Design summary of the Taguchi method

Design Summary	
Taguchi Array	L9(3^2)
Factors	2
Run	9
Columns of L9(3^4) array 1 2	

The AMCs were fabricated with the standard casting technique. The aluminum ingot underwent a heating process in the furnace at a temperature of 700 °C until it transitioned into a liquid state. Simultaneously, the graphene nanoplatelets (GNPs) and magnesium powder were subjected to preheating. Upon attaining this particular state, the temperature subsequently decreased to 650°C, followed by the initiation of stirring and mixing at a velocity of 500 revolutions per minute for a duration of 15 minutes. The molten slurry was introduced into the casting mold and left unattended until it reached solidification. Heat treatment was employed to improve the mechanical properties [9-10]. The technique is illustrated in Figures 1 and 2. The samples underwent solution treatment for a duration of 1 to 3 hours at a temperature of 540 °C, as indicated by the samples. Subsequently, they were rapidly cooled by quenching in water at ambient temperature, followed by an ageing process for 3 hours at 180 °C.

Table 3
Experimental layout of Taguchi method L9 orthogonal array design

No. Run	GNP Content (Wt-%)	Short Heat Treatment (min)
1	0.30	60
2	0.30	60
3	0.30	60
4	0.60	120
5	0.60	120
6	0.60	120
7	0.90	180
8	0.90	180
9	0.90	180

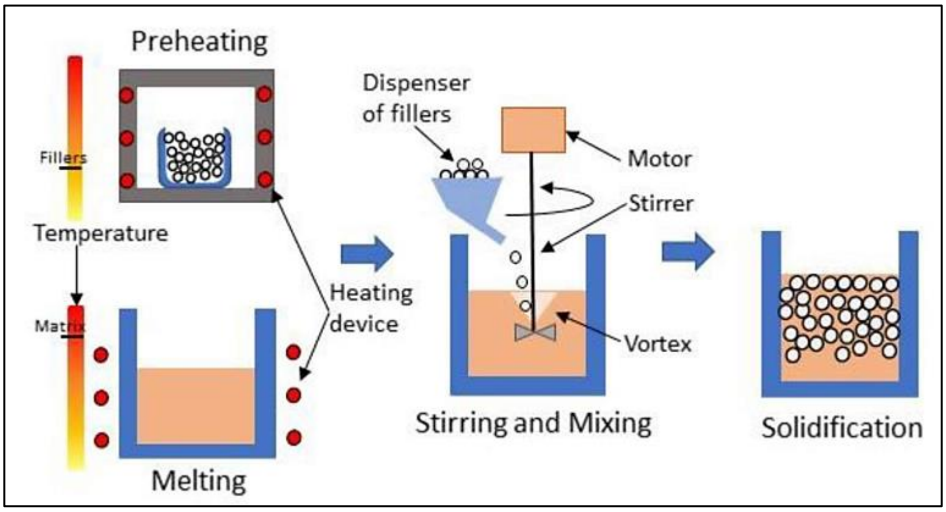

Fig. 1. Conventional Stir casting process [12]

After the heat treatment process, the prepared AMCs were characterized using a range of testing and analysis techniques, including X-ray diffraction (XRD) and tensile load testing. These experiments are used to investigate the microstructural and mechanical properties of the composites. The observation of microstructural characterization and distribution of GNP of the composites was performed by Nikon optical microscope and FESEM (ZEISS Sigma 500, Germany) after grinding (400, 600, 800, and 1200 grits), polishing with diamond solution (6, 3, and 1µm), and etching with Keller's reagent solution. Tensile tests were conducted at room temperature using a 100 kN universal testing machine. The samples were prepared following the ASTM E8M standard for each group, as shown in Figures 3 and 4. Three samples were used in this test to obtain a reliable data analysis. The yield stress used in this experiment was according to the 0.2% plastic strain offset.

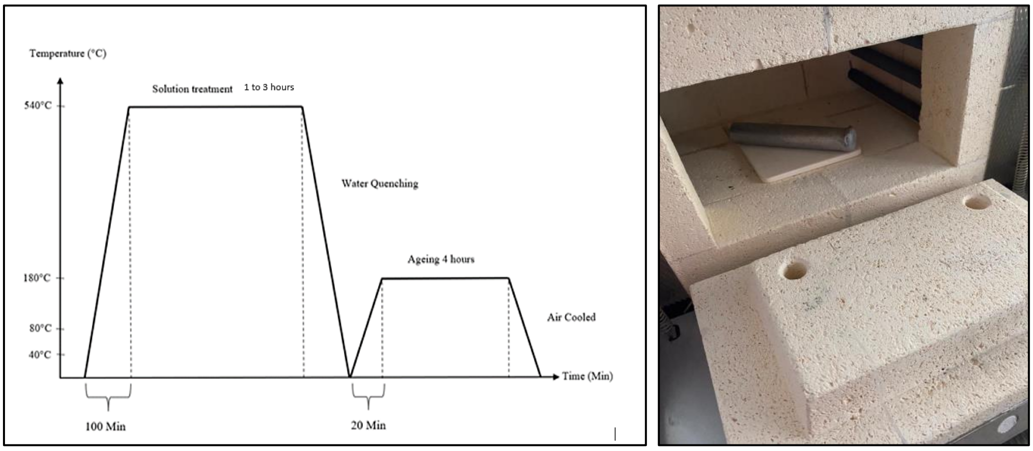

Fig. 2. (a) Schematic diagram of solution and ageing treatment, (b) specimen after heat treatment process

Fig. 3. Dog-bone shape

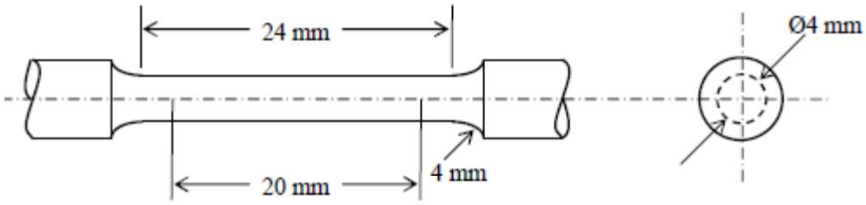

Fig. 4. Tensile test sample ASTM E8M [13]

3. Results

3.1 Microstructure analysis

Figures 5-7 depict the silicon forming as a solid in the eutectic phase of hypoeutectic or nearly eutectic aluminium-silicon alloys. The silicon within the Al+Si eutectic might manifest in diverse forms or alterations that can be strategically employed in a specific region. The Si-eutectic phase can be observed in the microstructure as black patches and is characterized by its needle- or plate-like morphology. During the solidification process of the aluminium-silicon alloy, a eutectic reaction occurs, forming regions with a high concentration of silicon. The Si-eutectic phase is formed through the eutectic reaction, occurring at a specific composition and temperature. The alloy's microstructure incorporates Si-eutectic, hence enhancing the alloy's strength [14-15]. Microstructural evolution exhibits a noticeable variation when the graphene content and the duration of heat treatment increase. The presence of the Si-eutectic phase becomes more frequent. This finding suggests that the 0.9 wt% GNPs reinforced aluminium composite with 180 minutes of heat treatment possesses the highest mechanical characteristics due to the greater presence of Si-eutectic phases.

On the other hand, the microstructure of the α-Al phase, which is the main aluminium matrix, becomes more globular with the increase of GNPs content. This phase is distinguished by the equiaxed or dendritic appearance of the aluminium grains that make up its structure. Adding GNPs significantly changes how the α-Al grains look. The larger α-Al becomes with increasing GNPs content from 0.3 to 0.9wt%. It indicates that the dendritic type of microstructure had been turned into the rosette type microstructure. The microstructure becomes closely packed, there are fewer gaps between boundaries, and the presence of porosity will be reduced [16]. The grain refinement will enhance the strength and plasticity of Al alloy [17]. When the aluminium atoms organize into a crystalline structure during the alloy's solidification, the α -Al phase is created. The alloy exhibits high ductility and heat conductivity due to the presence of the α -Al phase. The distribution and shape of the Si-eutectic and α -Al phases can change in the microstructure based on several variables, including the alloy composition, the heat treatment procedures, and the cooling rate during solidification. The Si-eutectic phase may show a network-like structure across the α -Al matrix in some samples where it is more evenly distributed. The Si-eutectic phase may display more pronounced characteristics in other samples, such as discrete particles or clusters.

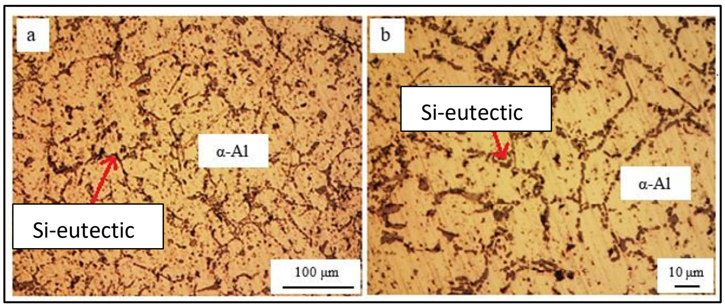

Fig. 5. (a) Microstructure of Aluminium A356 and (b) heat-treated Composite of A356 (graphene added) for specimen 1

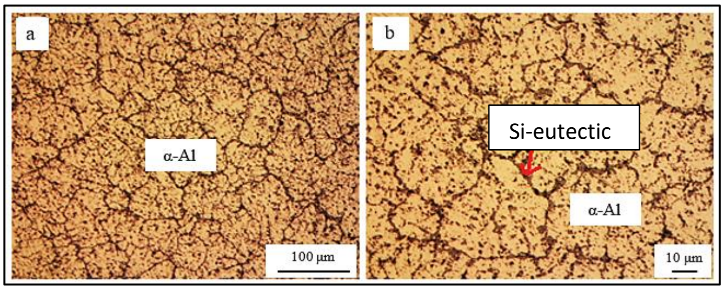

Fig. 6. (a) Microstructure of Aluminium A356 and (b) heat-treated Composite of A356 (graphene added) for specimen 4

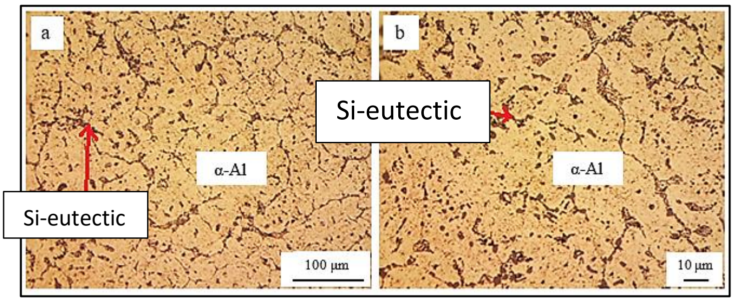

Fig. 7. (a) Microstructure of Aluminium A356 and (b) heat-treated Composite of A356 (graphene added) for specimen 7

The existence and distribution of Si-eutectic and α-Al phases, among other microstructural features of Aluminium A356, are essential in defining the alloy's mechanical qualities and performance. The interaction between these phases impacts important characteristics, including strength, ductility, and thermal conductivity. Modifying the material's qualities to satisfy certain application needs is feasible by comprehending and manipulating the microstructure. Si-eutectic and α -Al phases are combined in the microstructure of aluminium alloy A356. The Si-eutectic phase provides strength and hardness, whereas ductility and thermal conductivity are brought about by the α-Al phase. The overall characteristics of the alloy might change depending on the distribution and shape of these phases. Understanding the microstructure is crucial for improving Aluminium A356's performance and ensuring it is suitable for various technical applications.

FESEM-EDX analysis was conducted to prove the existence of particular phases in the material. β -Al5FeSi and - π Al8Si6Mg3Fe were found in the samples, as shown in Figure 8. These phases could be recognized and characterized from the rich information on the composite microstructure analyzed from the FESEM images. Additionally, EDX analysis was carried out to get information on the elemental makeup. Iron (Fe), silicon (Si), magnesium (Mg), and aluminium (Al), which are the main ingredients in the creation of the indicated phases, were found in the samples, according to the EDX analysis. A deeper knowledge of the properties and prospective uses of the composite material was made possible by the combination of FESEM and EDX analysis, which offered insightful information on the microstructural characteristics and elemental composition of the Aluminium A356 samples.

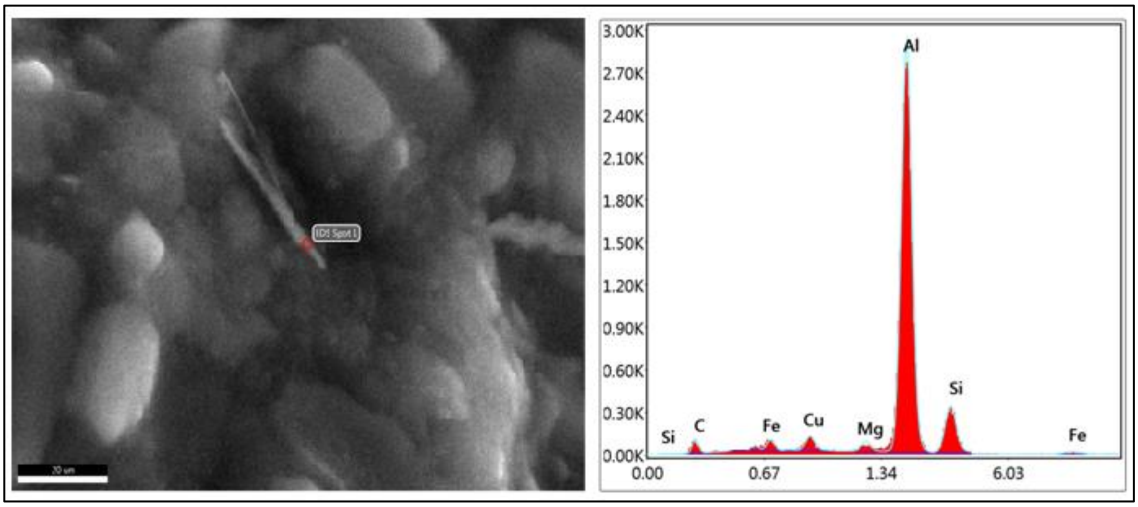

Fig. 8. FESEM-EDX: phase β-Al$_5$FeSi and π-Al$_8$Si$_6$Mg$_3$Fe of specimen 1

The aluminum matrix and properties of carbon (graphene nanoplatelets) are well-understood thanks to X-ray diffraction (XRD) investigations. The results of specimens (1–6) are illustrated in Figure 9. The diffraction of X-rays by the carbon atoms in the composite material is shown as peaks in the XRD pattern at varied angles. Counts are used to express peak intensity, representing the diffraction signal's relative strength. The presence of carbon peaks in all specimens (1–9) from the XRD data suggests that the graphene nanoplatelets (GNP) were successfully incorporated into the aluminum matrix composite. The peaks' varying intensities show the amount and distribution of carbon in the composite throughout the various angles. When carbon peaks show up at certain angles in the XRD data, it shows that the graphene nanoplatelets were spread out well in the aluminum matrix. Peak intensity represents the homogeneity and concentration of the carbon phase. Significant carbon peaks are seen in the Al-specimens at various angles, demonstrating strong graphene nanoplatelet dispersion.

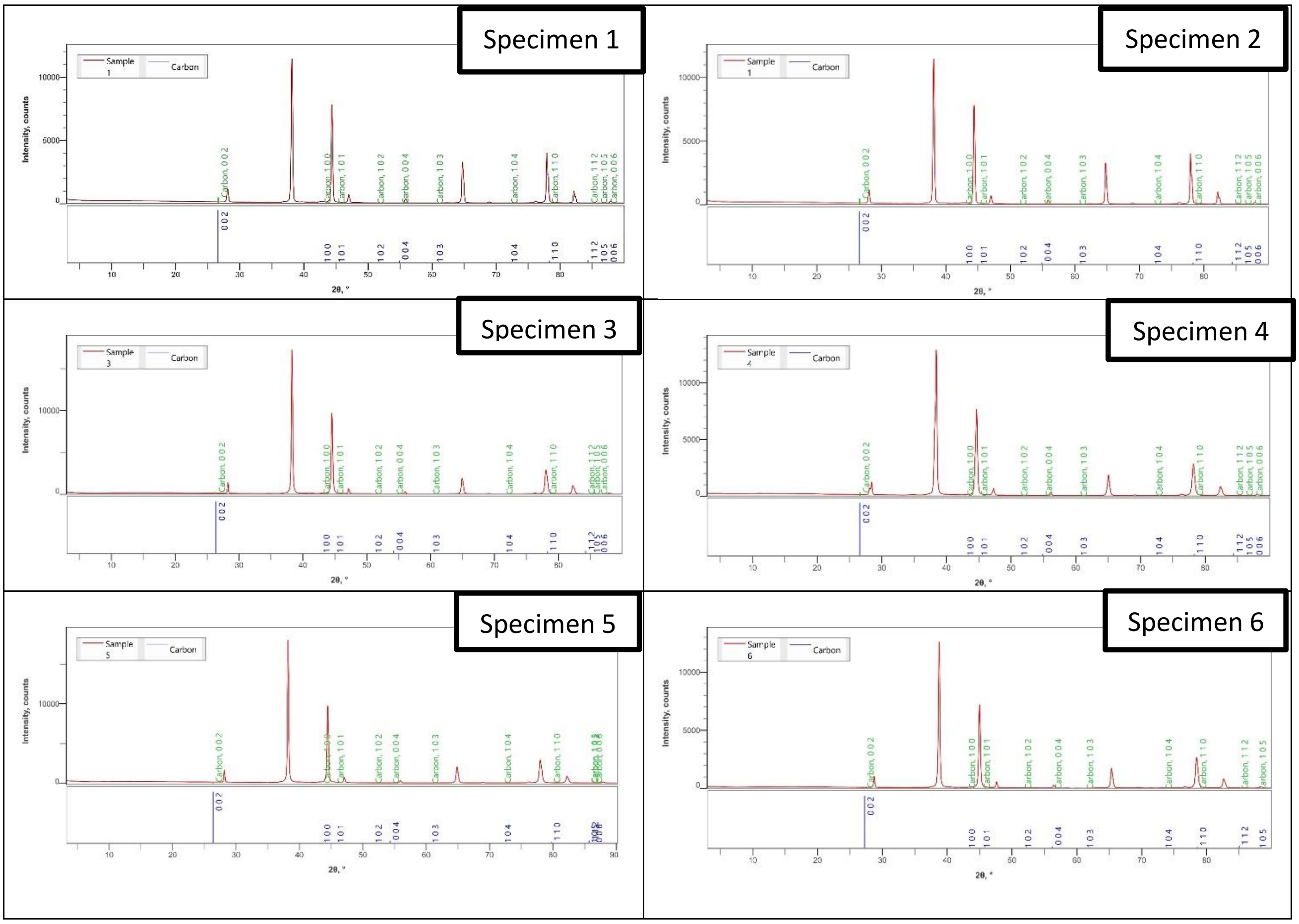

Fig. 9. XRD results of all specimens

3.2. Mechanical Properties

The results of tensile testing from Universal Tensile Machine for each specimen have been summarized in Table 4 based on Taguchi Method using Minitab 17 program software. This shows the connection between the quantity of graphene nanoplatelets (GNP), the length of the heat treatment, and the specimens' ensuing mechanical qualities. The mechanical characteristics significantly improve as the GNPs contents increases. Starting with the specimens with 0.3%, the ultimate tensile strength, yield strength, and elongation to fracture stay constant during the various heat treatment periods. This implies that the duration of the heat treatment has little effect on the mechanical characteristics of this specific GNP concentration. However, an intriguing pattern becomes apparent when looking at the samples with a 0.6% GNP concentration.

The highest stress that a material can endure before it cracks under strain is known as the ultimate tensile strength (UTS). We can see that the UTS values between the various specimens vary by examining the data. Regardless of the heat treatment length, the UTS for the specimens with 0.3% GNP concentration (Specimens 1, 2, and 3) remained constant at 170 MPa. This indicates that, at least throughout the range of heat treatment durations studied, the inclusion of 0.3% GNP had no discernible impact on the material's resistance to fracture under strain. However, the specimens with 0.6% GNP concentration (Specimens 4, 5, and 6) show a rise in UTS as the length of the heat treatment rises. For treatments lasting 180 minutes, the UTS increases from 240 MPa for a 60-minute treatment to 240 MPa. This implies that a material with better tensile strength may endure greater pressures before fracture when combining higher GNP content and longer heat treatment time. Similarly, the 0.9% GNP-containing specimens (Specimens 7, 8, and 9) display a constant UTS of 250

MPa throughout a range of heat treatment times. This shows that the material's tensile strength has already been optimized by obtaining a GNP content of 0.9% and a 60-minute heat treatment.

In contrast, any increases in heat treatment time do not produce appreciable gains. The yield strength values in the given data are fairly stable for all specimens and heat treatment times. The yield strength is always 150 MPa for the specimens with 0.3% and 0.6% GNP concentration. The yield strength of specimens containing 0.9% GNP is somewhat greater, ranging from 210 to 230 MPa. These findings imply that the GNP content, as opposed to the length of the heat treatment, has a greater influence on the yield strength.

The elongation to fracture for specimens containing 0.3% GNP remained constant at 3% during various heat treatment times. In a similar vein, specimens containing 0.6% GNP show a constant elongation to fracture of 5.2–5.5%. Specimens with 0.9% GNP concentration show a consistent elongation to fracture of 6% throughout the various heat treatment times. The outcomes of the tensile testing show some intriguing patterns and ideas. First, the evidence shows a considerable effect of GNP on the material's ultimate tensile strength and yield strength. Tensile strength typically increases with GNP concentration, but yield strength responds more complexly.

Additionally, the effect of the heat treatment period on mechanical characteristics changes depending on the GNP concentration. The ultimate tensile strength, yield strength, or elongation to fracture for the specimens with 0.3% GNP concentration do not appear to be appreciably impacted by the length of the heat treatment. Longer heat treatment times, however, enhance the final tensile strength of the specimens with 0.6% GNP concentration. As the heat treatment time is increased, the mechanical characteristics of the specimens with 0.9% GNP concentration, however, exhibit little to no change, indicating that the material has already hit its maximum potential for that GNP level. These results underline how crucial it is to optimize the mechanical characteristics of materials by considering both GNP content and heat treatment time. Additionally, it highlights the requirement for additional research and analysis to identify the ideal mix of GNP content and heat treatment for producing the requisite mechanical qualities.

Table 4
Mechanical properties of the Composites

Specimen	GNP (%)	Heat treatment (min)	Ultimate Tensile Strength (MPa)	Yield Strength (MPa)	Elongation to fracture (%)
1	0.3	60	170	150	3
2		120	170	150	3
3		180	170	150	3
4	0.6	60	240	200	5.2
5		120	240	210	5.2
6		180	240	210	5.5
7	0.9	60	250	210	6
8		120	250	230	6
9		180	250	250	6

4. Conclusions

The study focused on investigating the effects of graphene nanoplatelets (GNP) on an aluminium matrix composite's mechanical characteristics and microstructure. The successful integration of GNP in the composite was confirmed through X-ray diffraction (XRD) analysis. Compression and tensile tests were conducted to evaluate the material's mechanical properties. Key findings include that the concentration of GNP significantly influenced the composite's tensile strength, yield strength, and ductility. The optimal GNP concentration improved resistance to external stresses and deformation, while excessive GNP content increased brittleness.

The specimens with various concentrations of graphene nanoplatelets (GNP) underwent tensile testing, which revealed critical details about their mechanical characteristics. The findings showed that the addition of GNP affected the materials' tensile strength, yield strength, and ductility. Compared to the 0.9% GNP specimen, the 0.3% GNP and 0.6% GNP specimens had greater tensile and yield strengths. This shows that a GNP concentration that is just right can improve the material's resistance to external stresses and deformation. The 0.3% GNP and 0.6% GNP specimens also showed greater ductility, demonstrating their ability to withstand substantial plastic deformation before failing. The 0.9% GNP specimen, however, showed decreased ductility, which suggested greater brittleness.

The study emphasizes the importance of carefully selecting GNP concentrations to achieve desired mechanical characteristics. The research offers valuable insights for enhancing GNP-reinforced composites, which could benefit aerospace, automotive, and structural engineering. However, further investigation is needed to understand the precise impacts of GNP on various traits and to compare the results with existing literature. In conclusion, the study provides a preliminary understanding of the relationship between GNP content, microstructure, and mechanical characteristics in aluminium matrix composites. Future research should focus on detailed microstructural analysis, optimizing GNP parameters, and a wider range of mechanical testing to develop high-performance composite materials for specific applications.

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