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Effect of Graphene Addition on Microstructure and Mechanical Properties of Cast Aluminium Alloy Composite

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ABSTRACT

Aluminium-reinforced metal matrix composites (Al-MMCs) are highly desirable because of their low density, excellent corrosion resistance, high thermal and electrical conductivity, high strength, and stiffness. This research intends to improve the mechanical properties of Al-MMCs for lightweight automotive and aerospace applications. A Taguchi method with two parameters and four levels was used to determine the optimum amounts of graphene nanoplatelets (GNPs) (0.1, 0.25, 0.5, and 0.6 wt%) and stirring duration (1, 2, 4, and 8 minutes). GNPs were added to the molten aluminium alloys through the stir-casting process to enhance the mechanical properties of the Al-MMCs. The results revealed that the Al-MMCs with 0.25 wt% GNPs and 8 minutes of stirring time produced the highest value of hardness and tensile properties. Under these parameters, the microstructure changed from dendritic to rosette, which had a tremendous effect on the improvement of the mechanical properties. The Ultimate Tensile Strength (UTS) and Yield Strength (YS) of the samples were improved after T6 heat treatment with a value of 210.26 MPa and 145.31 MPa, respectively, and the hardness was 116 HV while the elongation to fracture was 4.89%. The results showed that the high GNP concentration improved the mechanical properties of the Al-MMCs.

1. Introduction

Aluminium-reinforced Metal Matrix Composites (Al-MMCs) are novel lightweight materials with remarkable mechanical qualities, rendering them well-suited for many industries, including aerospace, automotive, biomaterials, and others [1-3]. Nevertheless, further investigation is required to fully explore its features, including ductility, corrosion, and creep. Adding strengthening

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nanoparticles like titanium, nickel, graphene, carbon nanotubes, and silicon carbide is essential for changing the matrix's properties and making it more robust, stiffer, and resistant to wear [4]. Consequently, these reinforcements enhance specific strength, stiffness, damping characteristics, and wear resistance compared to conventional engineering materials [5]. As a result, Al-MMCs demonstrate enormous potential as a viable substitute for traditional alloys such as iron, steel, and aluminium [6]. Non-homogeneous distribution, inadequate creep resistance at high temperatures, and limited corrosion resistance have been recognised as challenges. This highlights the necessity for additional research to enhance the properties of aluminium-based composites, enabling their use in a broader range of applications.

The mechanical stirring technique used in the previous study [7-9] had positively impacted the reinforcement process. However, the lack of control over some factors has led to grain size inconsistencies during the stirring process. A study by Lei *et al.,* [10] highlighted that the smaller grain sizes typically increase the cast material's strength, hardness, and toughness due to the Hall-Petch phenomena, which asserts that reducing grain sizes improves the mechanical properties of the materials by increasing its resistance to dislocation [11].

Research and development have made numerous attempts to improve the Al-MMCs properties with a favourable combination of strength and ductility. A few studies have demonstrated that adding graphene as reinforcement elements can enhance the strength of the matrix. However, graphene tends to float on top of the molten alloy, which leads to increased agglomeration during casting [12]. The shearing action of the rotating blades caused the particles to deagglomerate and homogenise [13]. Several parameters have been utilised to improve the mechanical robustness of graphene-reinforced aluminium alloy. The intricate nature of the technologies utilised in the casting process implies that even minor alterations in the chemical elements can impact the outcome and result in faulty castings. Therefore, optimising process variables improves casting quality, productivity, and product quality [14].

A few studies have found that adding the Graphene Nanoplatelets (GNPs) as the reinforcement can improve the mechanical properties of the composite. The optimum casting parameters must be utilised to produce the composite with higher mechanical capability. The current study involved fabricating Al-MMCs reinforced with graphene using mechanical stir-casting. The Taguchi method was employed to optimise the strength of the MMCs by considering the GNPs content and stirring times. Subsequently, an analysis was conducted to assess each specimen's mechanical properties and microstructural characteristics.

2. Methodology

2.1 Taguchi Method

Orthogonal arrays were used in this study to examine every design component while minimising experiments carefully. Tables 1 and 2 summarise the Taguchi method's parameters and design. This work produced Al-MMCs with different GNPs weight percentages (wt%) and stirring times. The stir-casting parameters are presented in Table 1.

Table 1
Condition parameter

Parameter	Level 1	Level 2	Level 3	Level 4
GNPs content (wt%)	0.1	0.25	0.5	0.6
Stirring time (min)	1	2	4	8

Table 2
L16 orthogonal array

No of run	GNPs content (wt%)	Stirring time (min)
1	0.10	1
2	0.10	2
3	0.10	4
4	0.10	8
5	0.25	1
6	0.25	2
7	0.25	4
8	0.25	8
9	0.50	1
10	0.50	2
11	0.50	4
12	0.50	8
13	0.60	1
14	0.60	2
15	0.60	4
16	0.60	8

2.2 Experimental Procedure

The alloy used for this process was a commercial A356 aluminium alloy in the form of an ingot. Its chemical composition by weight percentage (Si = 7.38%, Mg = 0.26%, Cu = 0.0463%, Fe = 0.25%, Mn = 0.190%, Zn = 0.0068%, and Ti = 0.0411%) was obtained using the spectrometry technique. The GNPs were covered with aluminium foil and reheated to eliminate moisture. GNPs were injected into the molten base with a steel plunger and swirled with a three-blade propeller and WiseStir HT120AX. When the molten temperature decreases to 650°C, stirring will continue at 450 rpm. Finally, the molten liquid was poured into a billet mould to produce a composite feedstock. The casting method used is illustrated in Figure 1.

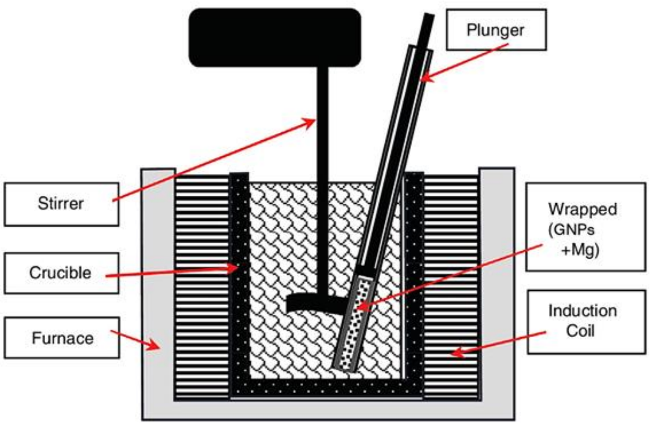

Fig. 1. Mechanical stir casting [15]

After the casting process, the billet was subjected to T6 heat treatment and material characterisation. The Al-MMCs underwent characterisation utilising several testing and analysis techniques, including X-ray Diffraction (XRD) and mechanical testing. The microstructure distribution of GNPs in the composites was examined using a Nikon Optical Microscopy (OM) and a Field Emission Scanning Electron Microscope (FESEM) by ZEISS Sigma 500, Germany. The microstructure analysis was conducted after grinding the samples with different grit sizes (400, 600, 800, and 1200), polishing with diamond solutions of varying particle sizes (3 µm and 1 µm), and etching with Keller's reagent solution. Tensile tests were performed using the 100 kN universal testing machine at a 5 mm/sec speed. Subsequently, the samples were machined into the dog bone shape according to the ASTM E8M standard for each group, as depicted in Figures 2 and 3. A total of two samples were utilised in this experiment to provide dependable data analysis.

Fig. 2. Dog bone sample

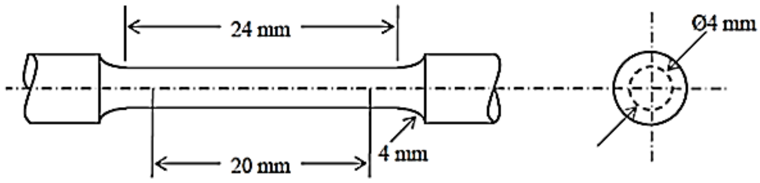

Fig. 3. ASTM E8M-04

3. Results

3.1 XRD Analysis

Figure 4 illustrates the XRD patterns of A356 alloys and GNP-A356 composites. The presence of aluminum peaks at [1,1,1], [2,0,0], [2,2,0], [3,1,1], and [2,2,2] indicates that the crystal structure of aluminum is face-centered cubic. The A356 alloy and composite samples exhibited prominent aluminium peaks. However, no pronounced peak was observed for GNPs. Furthermore, the absence of GNP peaks in all composite samples can be ascribed to the low GNP concentration. Carbon (C) particles were seen at an angle of 43.61° [1,0,1] in the GNPs-A356 composites. The XRD test also showed that the as-cast sample had small diffraction peaks of Al_4C_3 at angles of 43.61° and 40.10°, which were in line with the [0,0,12] crystal plane. In addition, other studies have reported the presence of the compound Al_4C_3 in graphene-reinforced aluminum composites [16]. Al_4C_3 facilitates effective load transfer between the aluminum matrix and GNPs, thereby impeding dislocation migration [17]. It is to be noted that a carbide phase can decrease the surface tension between carbon and aluminium, increasing carbon's capacity to spread and adhere to aluminium and strengthening the hardness of the composite [18].

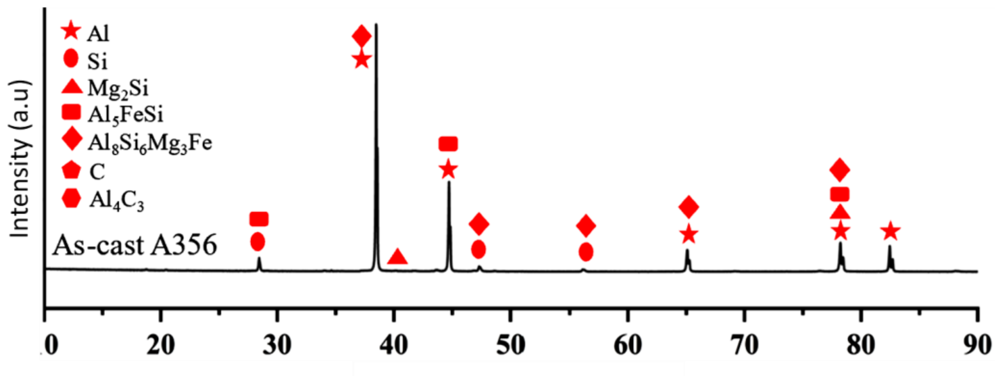

Fig. 4. Intermetallic phase found in XRD

3.2 Microstructure Analysis
3.2.1 Optical microscopy analysis

Figures 5 to 8 illustrate the microstructure of Al-MMCs with different GNPs contents. The molten alloys were stirred for 8 minutes before pouring into the mould. Based on the samples, it is manifest that the dispersion of alloy components is separated near the eutectic phase, although it is nearly uniform within the matrix grains. The grains of the as-cast Al-MMCs alloy were composed of rosette and near globular structure. The microstructures of the as-cast composites display prominent, semi-spherical grains surrounded by smaller ones. During the solidification process in the crucible, large α-Al grains are initially formed, allowing sufficient time to increase in size. The addition of GNPs has a substantial impact on the appearance of α-Al grains. As α-Al increases, it transforms from a dendritic microstructure to a rosette microstructure [19]. The microstructure becomes closely packed, leading to reduced interstitial spaces and lower porosity, which enhances the mechanical properties of the Al-MMCs.

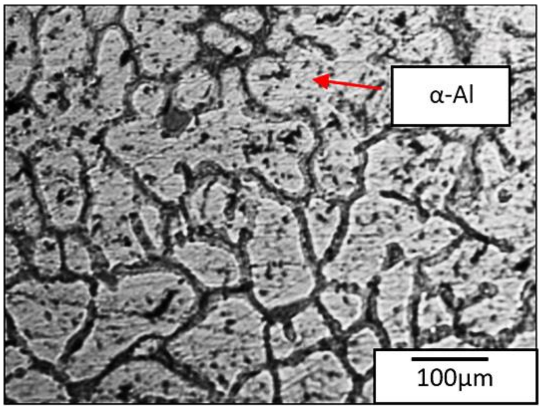

Fig. 5. Sample 4 (0.1 wt% GNPs, 8 minutes casting duration)

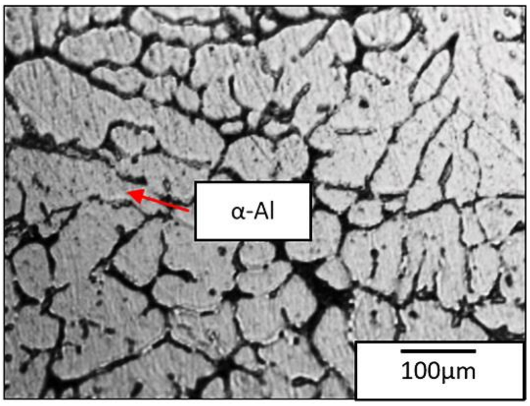

Fig. 6. Sample 8 (0.25 wt% GNPs, 8 minutes casting duration)

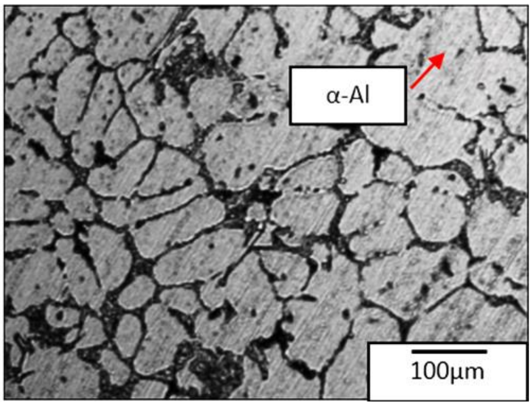

Fig. 7. Sample 12 (0.5 wt% GNPs, 8 minutes casting duration)

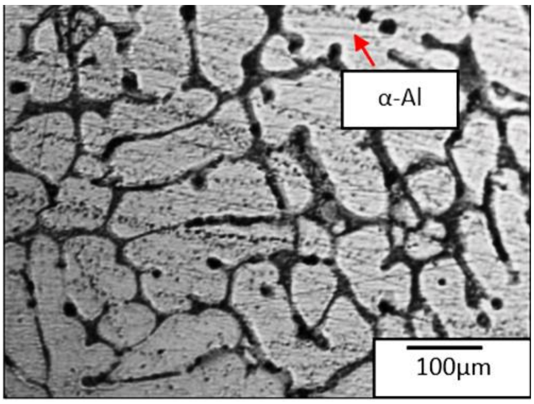

Fig. 8. Sample 16 (0.6 wt% GNPs, 8 minutes casting duration)

3.2.2 Field emission scanning electron microscope (FESEM) analysis

Four samples of Al-MMCs with various GNP contents were characterised using high-magnification FESEM, as shown in Figure 9. Three intermetallic phases were identified using FESEM: β -$AlFe_5Si$, Mg_2Si, and $Al_8FeMg_3Si_6$. The presence of a Fe-rich needle-like intermetallic compound was detected in the microstructure of as-cast A356. Because of its low diffusion coefficient, Fe is known to improve the thermal stability of aluminum alloys. Nonetheless, an uncontrolled Fe-rich intermetallic compound can degrade the hardness of the material [20]. Adding GNPs helps to control the nucleation of the Fe-rich due to limited growing space and converts the harmful Fe-rich into a less detrimental form. There was also the π-phase observation where π-Al8FeMg3Si6 existed in a compacted morphology and was less detrimental than the β-Al_5FeSi. Nevertheless, the precipitation of Mg_2Si in small formations alongside the silicon particles is homogeneously distributed throughout the composition. This formation helped to improve the composite's hardness properties [21].

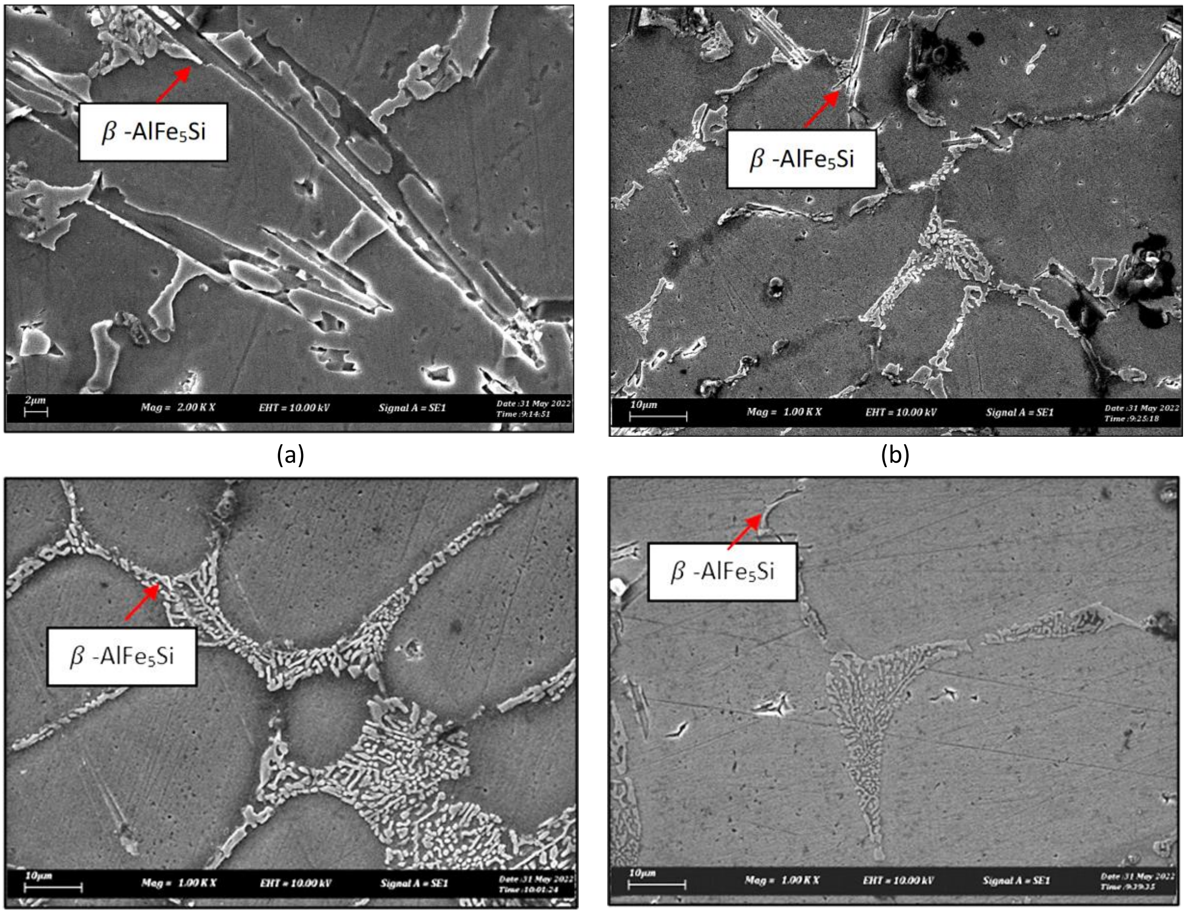

Fig. 9. FESEM results (a) Sample 4 (b) Sample 8 (c) Sample 12 (d) Sample 16

3.3 Mechanical Testing

3.3.1 Hardness test

The Vickers Hardness testing was conducted on all samples as tabulated in the Table 3. The hardness measurement was taken five times in different positions, and the data average was calculated to obtain reliable results. The bar graph shown in Figure 10 portrays the average hardness testing value, which visually displays the results of the samples. The composite with the highest hardness value was recorded as high as 116.09 HV, labelled as sample 8, and was prepared with a parameter of 0.25 wt GNPs and a stirring time of 8 minutes. Subsequently, samples 7 and 6 were measured to have hardness values of 114.93 HV and 104.25 HV, respectively. These results demonstrate a significant alteration in the raw aluminium alloy's mechanical properties, specifically hardness. The considerable disparity in hardness values is attributed to the parameter variation employed during the mechanical stir-casting process. The microstructures gathered from field emission scanning electron microscope and optical microscopy have shown the main factors affecting the composites' hardness. Under optimal conditions, the dendritic arms in the microstructure of optical microscopy undergo fragmentation and evolve into a rosette-like shape, which is evenly distributed during the stirring process. Note that the formation of spherical microstructures leads to dense packing between barriers, thereby preventing porosity. The incorporation of β -$AlFe_5Si$, Mg_2Si, and $Al_8FeMg_3Si_6$ enhanced the mechanical properties of the casting samples.

Table 3
Vickers hardness reading

Sample no	Hardness, HV					
	1	2	3	4	5	Average
1	84.32	83.82	82.23	83.96	82.33	83.33
2	83.78	84.54	83.10	83.99	82.97	83.68
3	86.60	86.23	86.43	85.91	85.70	86.17
4	86.57	85.49	86.90	86.69	87.11	86.55
5	98.34	98.31	97.89	97.40	97.83	97.95
6	98.11	99.80	97.32	100.11	98.12	98.69
7	98.11	99.81	97.32	102.11	100.12	99.49
8	98.53	99.12	98.98	106.38	100.42	100.69
9	78.30	77.65	78.35	78.27	78.30	78.17
10	77.95	78.12	78.19	78.64	77.98	78.18
11	79.12	79.19	79.21	78.93	79.03	79.10
12	81.20	80.98	80.61	80.80	80.90	80.90
13	80.71	80.76	80.71	80.68	80.72	80.72
14	87.70	87.72	86.79	86.81	87.59	87.32
15	89.40	89.42	89.41	89.27	88.98	89.30
16	90.43	89.92	91.20	91.18	89.85	90.52

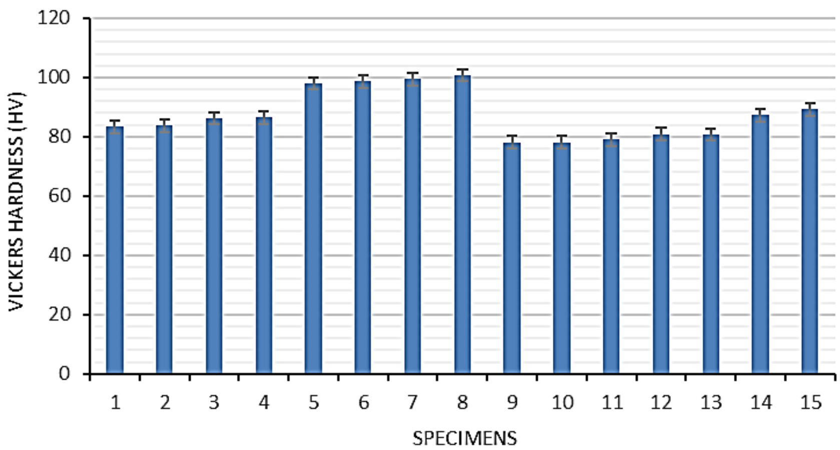

Fig. 10. Average hardness of Al-MMCs sample

3.3.2 Tensile properties

Table 4 shows a tensile testing result for the Al-MMCs samples. The tensile test was done twice for every sample to obtain reliable results. Sample number 8 had the highest Yield Strength (YS) with a value of 145.22 MPa and ultimate tensile strength (UTS) of 210.26 MPa, while the elongation was 4.89%. The study revealed that 0.25 wt% of GNPs and 8 minutes of stirring time produce higher UTS than other parameters, followed by sample number 7 with 0.25 wt% of GNPs and 4 minutes of stirring time. The pattern of the results indicated that the higher content of GNPs gives superior mechanical properties as long as it does not exceed 0.25 wt% and has a longer stirring duration.

Table 4
Tensile testing

Run no	Tensile								
	YS (MPa)			UTS (MPa)			Elongation (%)		
	1	2	Average	1	2	Average	1	2	Average
1	99.81	101.38	100.60	169.92	172.30	171.11	3.67	3.44	3.56
2	102.32	103.20	102.76	178.22	177.09	177.66	3.98	3.21	3.60
3	114.36	113.50	113.93	187.09	186.88	186.99	3.96	3.60	3.78
4	117.33	118.01	117.67	199.00	188.41	193.71	4.11	4.65	4.38
5	125.91	125.36	125.64	198.37	199.33	198.85	4.66	4.73	4.70
6	132.27	139.30	135.79	211.70	196.92	204.31	4.73	4.77	4.75
7	142.84	141.08	141.96	209.87	205.98	207.93	4.75	4.83	4.79
8	145.12	145.31	145.22	216.72	203.80	210.26	4.84	4.93	4.89
9	78.63	85.65	82.14	165.78	168.09	166.94	3.21	2.99	3.10
10	79.89	86.32	83.11	167.90	161.13	164.52	3.41	3.11	3.26
11	87.36	85.84	86.60	169.37	163.75	166.56	3.29	3.25	3.27
12	112.39	97.72	105.06	174.89	173.56	174.23	3.82	3.73	3.78
13	98.30	97.36	97.83	169.87	170.78	170.33	3.51	3.49	3.50
14	97.94	98.81	98.38	175.70	169.53	172.62	3.44	3.58	3.51
15	120.33	121.40	120.87	194.22	190.67	192.45	4.63	4.89	4.76
16	123.67	123.74	123.71	201.38	185.29	193.34	4.74	4.82	4.78

4. Conclusions

The study aimed to investigate the impact of GNPs on the mechanical characteristics and microstructure of the Al-MMCs. The microstructural investigation revealed that the concentration of GNPs significantly influenced the samples' tensile properties. A higher concentration of GNPs enhanced resistance to external stresses and deformation, while an excessive quantity of GNPs led to greater brittleness caused by agglomeration. Nevertheless, the duration of stirring is of utmost importance in disrupting the clumping together of GNPs throughout the stirring process. The Al-MMCs had a rosette-shaped particle microstructure with tighter packing, smaller gaps between the particles, and fewer holes. Sample 8 has the highest UTS of 210.26 MPa, YS of 145.22 MPa, and elongation to fracture of 4.89%, while the hardness is 116.09 HV. Based on the data, it can be concluded that sample 8, which utilised 0.25 wt% of GNPs and a stirring period of 8 minutes, was the most favourable parameter for this experiment.

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