

Effect of micro raphite particles on the microstructure and mechanical behavior of aluminium 6061 (Al-Mg-Si) alloy composites developed by novel two step casting technique

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1. Introduction

Abstract

In the present research, the effect of micron sized graphite addition on the microstructure and tensile failure of Al6061 alloy has been studied. The Al6061 alloy metal matrix composites reinforced with 6, 9 and 12 varying weight percentages of graphite particles were fabricated by novel two step stir cast route which helps in improving the wettability of Al6061 alloy matrix with graphite particles. The synthesized composites were subjected to microstructural studies, density, hardness and tensile properties testing. Microstructural characterizations of obtained samples were carried out by SEM microscopy, EDS and XRD patterns. The occurrence of graphite particles were confirmed by the XRD patterns. The density and hardness of metal composites have been decreased with adding of graphite reinforcement, while the ultimate tensile strength and yield strengths were improved with the addition of graphite particles. There was increase in the elongation of the Al6061 alloy composite after the incorporation of the reinforcement. Various fracture mechanisms were observed in the Al6061-graphite composites using SEM.

A composite material is a macroscopic arrangement of two or more different materials, with a comprehensible interface between them [1-3]. Composites are utilized for their auxiliary behaviors, as well as for thermal, tribological and several applications [4,5]. Recent materials are normally improved to accomplish a specific parity of properties for a given scope of uses.

Metal composites have risen as a vital course of resources for basic structural and wear appliances, basically because of their better weight-strength to cost proportions when contrasted with the monolithic materials [6-8]. Among MMCs Al metal composites are being reflected as a gathering of new impelled materials for its light weight, high explicit modulus, low coefficient of thermal extension and great wear properties [9,10].

The exclusive combinations of properties by Al and its alloys make it one of the versatile, cost-effective, and attractive metallic resources for a broad variety of uses from soft, highly elastic wrapping foil to the wide engineering applications [11,12]. Aluminum alloys are second just to steels in being used as auxiliary structural metals.

Several researchers have investigated the various properties of ceramic particles reinforced metal matrix composites. Many investigations were made to evaluate the tensile behavior of particulates reinforced AMCs. Ultimate tensile strength of aluminium 6061 and Al₂O₃ composites were evaluated by Bhaskar and co-authors [13]. UTS of 6061 Al with 20% Al₂O₃ composites was found around 310 MPa. Qiyao [14] has reported the improved tensile strength and decreased elongation of A356 with SiC composites. Tensile strength of Al alloy with 6 wt% graphite content was studied by Poonam and co-authors [15], reported improved YS and UTS due to presence of graphite particulates. Ramesh and co-authors [16] have conducted the experiments on Al6061 with graphite composites, revealed improved UTS and ductility due to presence of graphite particles up to 4 wt%. Recently, Jaswinder singh [17] evaluated the strength of Al2024/SiC/red mud composites, Nishant and co-authors [18] have experimented mechanical behavior of AA7075 with B4C and rice husk composites, and tensile behavior of 6061 aluminium reinforced with Al₂O₃ particulates were studied by Bharath and co-authors [19].

In the literature, the investigations made on graphite particles with varying particle sizes upto 100 $\mu m.$ In the present research an attempt

has been made to study the Al6061 alloy with 100 µm to125 µm sized graphite particles reinforced composites. Also, for the preparation of metal based composites Palanikumar *et al.* [20] adopted conventional stir casting process. Al6061 alloy with 15 wt% of SiC and 4 wt% of graphite particles reinforced composites were studied for influence of drilling parameter on thrust force. In the current investigations Al6061 alloy with 6, 9 and 12 weight percentages of graphite reinforced composites were synthesized by novel two step stir casting technique. Since the density of graphite particles is 2.20 g·cm⁻³ which are too lighter than the matrix, it is difficult to wet these particles in the molten Al6061 alloy. This novel two step stir process helps in improving the wettability between the base matrix and reinforcement particles. The improved bonding between the matrix and graphite particles directly impacts the behavior of prepared composites.

Al6061 is a mixture of metal with lower density and moderate strength, but lacks in high strength with more ductility. In order to improve negative aspect this alloy is reinforced with micron sized graphite particles. The behavior of aluminium 6061 alloy reinforced with graphite is synthesized and the microstructure, physical, hardness and tensile properties are evaluated.

2. Experimental details

2.1 Materials and composites preparation

Metal composites with 6, 9 and 12 weight percentages of graphite particulates with 100 μ m to 125 μ m size were produced by stir method of casting. Aluminum 6061 alloy (Fenfee Metallurgical Pvt. Ltd., Bangalore) is chosen as matrix due to its greater casting properties, strength, formability, heat treatment nature, good corrosion resistance, machinability and wide applications in several sectors etc. while graphite particles with a size of 100 μ m to 125 μ m were utilized as reinforcements (Figure 1). The chemical configuration of the Al6061 alloy utilized in the present studies is represented in Table 1.

Graphite particles of range between 100 μ m to 125 μ m (Supplied by Bioaid and Scientific Industries, Bangalore, Karnataka, India) are used for the present study. The SEM analysis of graphite particulates are carried by using SEM, at BMSCE, Bangalore, Karnataka, India. The composition of graphite particles is shown in the Table 2 and Table 3 contains the various properties of graphite.

The estimated amount of Al6061 alloy ingots are placed in the furnace for melting. The melt was heated to 750°C. By the utilization of thermocouple the melt temperature is measured. Once the melt reaches to 750°C degassers (solid hexachloroethane-C2Cl6) are introduced into the melt. Vortex was created by using the chromium steel stirrer, which was coated with zirconia. The impeller speed was maintained at 300 rpm and the depth of immersion of the impeller is 60% of the height of the molten matrix material. Once the vortex is made then the preheated graphite particles in steps of two stages are introduced into the melt by a persistent feed rate, which includes distributing the whole weight mixture of reinforcements in two equivalent weights. At every stage vigorous stirring carried out before and after incorporation of alumina particles to prevent particle clustering and to ensure that the micro particles are homogeneously dispersed in the melt. With continuous stirring, the molten metal is discharged into preheated cast iron die. The Al6061-graphite composite specimens with 15 mm

diameter and 120 mm length are obtained after casting as shown in Figure 2 The Al6061 with 6 to 12 weight percentages of graphite composites are prepared by same procedure. The major reason to choose the graphite particulates in step of 6, 9 and 12 wt% is due to its density. The density of graphite is 2.20 g·cc⁻¹, which is lesser than the base matrix (2.70 g·cc⁻¹). The higher weight percentages of graphite creates the more volume fraction of the graphite which causes clustering or de-bonding.



Figure 1. SEM micro-photograph of $100 \,\mu m$ to $125 \,\mu m$ sized graphite utilized in the present study.

Table 1. Al6061 alloy chemical composition.

Element	Al6061	Al6061	
(wt%)	(as per ASTM standard)	(actual)	
Mg	0.8 - 1.2	0.89	
Si	0.4 - 0.8	0.64	
Fe	Max. 0.7	0.23	
Cu	0.15 - 0.40	0.17	
Ti	Max 0.15	0.10	
Cr	0.04 - 0.35	0.07	
Zn	Max. 0.25	0.03	
Mn	Max. 0.15	0.07	
V	Max. 0.05	0.01	
Al	Balance	Balance	

Table 2. Chemical composition of graphite particles.

Element	Composition (wt%)	
Carbon	99.9	
Others	0.01	

Table 3. Properties of graphite particles.

Element	Composition (wt%)	
Density (g·cc ⁻¹)	2.20	
Melting point (°C)	2,900	
Poisson's Ratio	0.14	
Modulus of Elasticity (GPa)	10-15	
Hardness (HV)	1.7 Moh's	
Compressive Strength (MPa)	110	
CTE ($\mu m \cdot m^{-1} \cdot {}^{\circ}C$)	9.6	
Thermal conductivity (W·mk ⁻¹)	86	



Figure 2. Prepared Al6061 alloy graphite composites.

2.2 Testing of Composites

The size, shape and distribution of graphite particles present in Al6061 compound composites are done utilizing SEM instrument (TESCAN VEGA 3 LMU, Czech Republic). The machine is connected with JED 2300 examination software programme for EDX investigation. For SEM, specimens are cut to get 15 mm diameter and 5 mm height. The cut samples are made flat surface using belt grinder. Then the samples are polished on a series of silicon carbide emery papers with grit size of 300 to 1000. Finishing is carried out by hand on micro cloth by fine cerium oxide. Produced samples are etched to reveal the proper granular structure using Keller's reagent. The etching solution consists of 95 mL of H₂O, nearly 2.5 mL HNO₃, also 1.5 mL HCl and 1 mL HF. After etching, the samples are washed and thoroughly dried.

X-ray diffraction studies are conducted on Al6061 alloy composites in order to recognize different phases of Al alloy matrix composites. For XRD studies Al6061 with 6 and 12 weight percentages of graphite composites were selected. For this purpose prepared composite samples are cut to 15 mm diameter and 2 mm height in size specimens and same polishing method is carried as in the case of SEM. XRD studies are carried out by a PANALYTICAL XRD using Cu-K alpha radiation. The 2θ range is selected such that all the intense peaks of the material phases predictable are covered.

Variation in density of Al6061 alloy particulate composites on solidification is a measure of shrinkage of castings, the level of nonmetallic inclusions present and porosity. In the case of AMMCs with graphite particulates as reinforcements, there will be this variation in density, thus giving some idea of the reinforcement particles, their weight fraction and porosity. Taking this view in mind, specimens of varying dimensions were cut from different locations of as-cast alloy and composites for density measurements. ASTM D792 [21] procedure is used for density evaluation, circular pieces of size 12 mm in dia., and 30 mm in length are used with an allowance of ± 0.15 mm. Refined water at room temperature is utilized as the drenching fluid and the mass is estimated utilizing digital balance with a 0.001 g resolution. Experimentally, the composite density is acquired by displacement methods utilizing a physical offset with measuring kit according to the ASTM: D792-66 details. Further, theoretical densities were estimated by using standard rule of mixture formula.

To measure the hardness Vickers hardness tester (Model: Olympus) having a load range of 25 g to 1000 g is used. The ASTM E384 [22] standard is used for testing. Loads are applied by dead weight. A precision diamond indenter on the material is used and a load of 100 g is applied over the specimen of diameter 15 mm and 10 mm length for a period of 30 s. Then, the indentation is measured microscopically and the applied load is used to estimate the value in terms of Vickers hardness number (VHN). Three readings have been recorded on each sample at various locations and mean value has been noted.

The VHN is calculated by below Equation 1.

$$VHN = 1.854 \, P/d^2 \tag{1}$$

Where, P is indicating the load in kg and average length of the diagonals is indicated by d.

By the utilization of computerized universal testing machine (UTM) of Instron make, with 60 kN capacity with least count of 4 N, tensile tests are carried out. The tensile specimens having a dimension of 9 mm gauge dia. and gauge length of 45 mm respectively as per ASTM E8 [23] standard as shown Figure 3. All the experiments are done at a rate of 0.1 mm·min⁻¹ in a displacement mode. Three tests are carried out and the best results are considered. Tensile behavior of the matrix and its composites viz., UTS, YS and ductility are evaluated. After the test, fracture surfaces are presented for microstructural studies using SEM to understand the fracture mechanism.



Figure 3. Tensile test specimen as per ASTM standard.

3. Results and discussion

3.1 Microstructural Analysis

Figute 4(a-d) shows the SEM micrographs of Al6061 alloy and graphite reinforced composites. Figure 4(a) shows the SEM micrograph of as-cast Al6061 alloy. It confirms that there is less porosity and grins are visible properly. Figure 4(b-d) shows the SEM images of Al6061 with 6 to 12 weight percentages of graphite. It is observed that in the two stage reinforcement mixing stir casting method of Al6061 alloy composites; the SEM micrographs of Al6061 alloy with 6, 9 and 12 weight percentages of graphite composites revealed the uniform distribution of graphite particles in the matrix. There is no agglomeration of particles in 6 and 9 weight percentages of graphite composites as seen in Figure 4(b) and Figure 4(c). Further, little agglomeration happened in Al6061 with 12 weight percentage of Gr composites due to lesser density of graphite, which increases the quantity of reinforcement by volume.

EDS analysis is also carried on the 6 and 12 weight percentages of graphite reinforced specimens which clearly showed the presence of C (carbon) peaks confirming the presence of graphite as shown in Figure 5(a-b). Figure 5(a) shows the elemental analysis of Al6061 with 6 weight percentage of graphite composite using EDS which confirm the elements like C, Mg, Si, Mn, Fe, Cu, Ti and Zn in Al alloy matrix composite. Figure 5(b) shows the elemental analysis of Al6061 with 12 weight percentage of graphite composite using EDS which confirm the elements like C, Mg, Si, Mn, Fe, Cu and Ti in Al alloy matrix composite. The distribution of Gr particles in Al6061 alloy composite is confirmed by the presence of Gr in the form of carbon (C).

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Figure 4. (a-d) SEM micrographs of (a) Al6061 alloy (b) Al6061-6 wt% graphite (c) Al6061-9 wt% graphite (d) Al6061-12 wt% of graphite composites.



Figure 5. EDS spectrum of (a) Al6061-6 wt% graphite (c) Al6061-12 wt% graphite composite.



Figure 6. X-ray diffractograms of Al6061-6 wt% graphite (b) Al6061-12 wt% graphite composite.

The XRD is carried out in a θ -2 θ diffractometer Panalytical using Cu K α radiation, with 35 kV of voltage and 50 mA current. In Figure 6(a-b) the peak observed for the Al6061 with graphite composite is at 27°, 42° and 60° approximately, similar observations were made by Ram Prabhu and co-authors [24].

3.2 Density Measurements

Figure 7 shows the theoretical and experimental values of densities of the Al 6061-graphite particulate composites for various weight percentages of graphite particles. The theoretical density of as-cast Al6061 alloy is 2.7 g·cm⁻³ and corresponding experimental density is 2.66 g·cm⁻³. Al6061 alloy with 6, 9 and 12 wt% graphite reinforced composites showed 2.6 g·cm⁻³, 2.65 g·cm⁻³ and 2.63 g·cm⁻³ theoretical densities respectively. Experimental densities of Al6061 with 6 to 12 weight percentages of graphite composites are 2.59 g·cm⁻³, 2.52 g·cm⁻³ and 2.48 g·cm⁻³ respectively, which are in line with theoretical densities. The density of graphite particles is lesser than the Al6061 alloy, which results in decreased density of composites. Similar results were found by Shanthanikethan and co-authors [25] in Al6061-graphite and rice husk composites

3.3 Hardness Measurements

From Figure 8 as the graphite percentage increases for 6 to 12 weight percentages, the hardness of the composites decreases. The hardness of Al6061 alloy is 93 VHN and for 6, 9 and 12 weight percentages of graphite reinforced composites are 88 VHN, 82 VHN and 74 VHN respectively. The abatement in hardness of composites is not out of the ordinary since graphite, being a delicate dispersiod, does not contribute decidedly to the hardness of composites. As the addition of graphite increases the ductility [26], is an effective solid lubricant, renders the material more easily deformable with respect to the indenter of the hardness tester. Such a monotonic reduction in the hardness of the composite as graphite might be added to improve the other mechanical properties. Seah [27] concluded that the MMC hardness decreases with graphite particles presence.

3.4 Tensile properties

The plot of UTS and YS for Al6061 with 6, 9 and 12 wt% of micro graphite in metal lattice composite has been introduced in Figure 9.

From the test it is found that the ultimate and yield strength of base Al6061 alloy is 141 MPa and 126.5 MPa respectively. The UTS of Al6061 with 6, 9 and 12 weight percentages of graphite are 184.0 MPa, 191.1 MPa and 194.4 MPa respectively. The yield strength of Al6061 with 6 weight percentage graphite is 159.0 MPa, with 9 weight percentage of graphite it is 174.5 MPa and with 12 weight percentage of graphite composite it is 174.8 MPa. The Al6061 and 12 weight percentage of graphite reinforced composites shown 37.8% improvements in the UTS and 38.2% improvements in the YS as compared to base alloy. Similar increase in UTS and YS values were reported by Nikhil et al. [28] in graphite reinforced metal matrix composites. Veereshkumar and co-authors [29] studied the ultimate tensile strength of Al6061 alloy with varying weight percentage of WC and 4 wt% of constant graphite particles reinforced composites. The improvement in the ultimate strength is due to fact that the combined effect of WC and graphite particles which acts as a barrier for the load transfer.



Figure 7. Variation in density of Al6061-graphite particulate composites with increasing graphite particle content.



Figure 8. Variation in hardness of Al6061-Gr particulate composites with increasing graphite particle content.



Figure 9. Variation in ultimate and yield strength of Al6061-graphite particulate composites with increasing graphite particle content.

From Figure 9 as the graphite content increases from 6 to 12 weight percentages, the UTS and YS of the composite material increases. These outcomes are as per those obtained via Seah *et al.* [30] who detailed comparable discoveries in graphite strengthened composites. This enhancement in UTS and YS is because of Al6061 alloy strengthening that may have happened because of grain strengthening after a decrease in composite grain measure, and the created high dislocation density in the Al6061 framework. Further, from the plot it is noted that graphite reinforced composites shown rapid improvements in the ultimate and yield strength for 6 weight percentage of graphite composites shown slight improvements in UTS and YS over 6 weight percentage of reinforced composites. This is mainly due to lesser density of graphite, as weight percentage of graphite increases quantity of graphite content will be more, which creates more clustering and pores. Mohanvel *et al.*

[31] studied the impact of 4 to 12 wt% of graphite particles on the mechanical behavior of Al6351 alloy fabricated by stir casting process. In the present Figure 9 the enhancement in strength is mainly due to the impact of novel two step stir cast process. In this process the required amount of graphite particles were added to the molten metal in two steps. This will helps in improving the wettability, which further enhances the properties of the synthesized composites.

Figure 10 is a graph indicating the effect of Gr content on the ductility of Al6061 with 6, 9 and 12 weight percentages of Gr composites. As the graphite percentage increases 6 to 12 weight percentages, the ductility of the composite material increased. In fact, as the graphite percentage is increased from 6 to 12 weight percentages the ductility improved by about 47.9%, these results are in line with the researcher Turkan *et al.* [32] who has investigated the properties of Al-Gr materials. Since graphite is an effective lubricant, causes the movement in the grains along slip planes.

3.5 Tensile fractography

The fracture surfaces of Al6061 base, Al6061 with 6, 9 and 12 weight percentages of graphite composites have been studied as shown in Figure 11. The surface of monolithic Al alloy sample indicates a fibrous mode of fracture (Figure 11(a)). A portion of the damages saw on the fractured surface are found far from profound voids, which uncover the more prominent flexibility of matrix material. Figure 11(b-d) contains the surfaces fractured during the test of Al6061 with 6, 9 and 12 weight percentages of graphite composites. The addition of soft graphite particles increases the ductility of composites. SEM micrograph of composites indicates the ductile fracture. Further, fracture surfaces of graphite reinforced composites shown enlarged grains along the slip planes during tensile test, Figure 11(d) indicates the movement of grains along the planes and contributes in increasing percentage elongation.



Figure 10. Variation in ductility of Al6061-Gr composites with increasing graphite particle content.



Figure 11. (a-d) Tensile fracture surfaces SEM micrographs of (a) Al6061 (b) Al6061-6 wt% graphite (c) Al6061-9 wt% graphite (d) Al6061-12 wt% graphite composites.

4. Conclusions

The Al6061 alloy with graphite particles MMCs are effectively produced by stir cast route with different weight percentages of reinforcement. The produced composites (Al6061-6, 9, 12 weight percentages of graphite) are effectively synthesized via stir casting with two-stage additions of reinforcement. The uniform dispersion of graphite particles from the SEM two stage additions of alumina particles in Al6061 matrix has resulted in uniform dispersion of reinforcing particles as obvious from SEM studies. The EDS study showed the existence of graphite particles in produced composites and the XRD patterns of Al6061-6 and 12 weight percentages of graphite composites were analyzed. XRD investigations validate the existence of graphite phases in the Al6061 alloy matrix. After the inclusion of graphite particles in Al6061 matrix alloy, the theoretical and experimental densities of Al6061-6, 9, 12 weight percentages of graphite composites have decreased. The micro hardness of graphite reinforced composites decreased, while UTS and YS of graphite reinforced composites have increased with slight increase in the ductility. Fractographic analysis of tensile fractured surfaces using SEM has indicated the various fracture mechanisms of base alloy Al6061 and produced composites.

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