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Mechanical properties and microstructure of A356 alloy reinforced AIN/MWCNT/graphite/AI composites fabricated by stir casting

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Abstract

In this experimental research work, the metal matrix composites of A356 alloy were prepared by reinforcing different shaped particles Aluminium Nitride (AlN), Multi-Walled Carbon Nano Tube (MWCNT) and graphite (Gr). The main objective of this research work was to investigate the influence of different shaped reinforcement in the microstructure and mechanical behaviour of composites. The reinforcements (AlN & MWCNT) were added as 0.5, 0.75, 1 and 2 vol% and the graphite was maintained in 0.5 vol%. The composites were fabricated using stir casting followed by ultrasonic vibration treatment. The tensile, compression and Brinell hardness test were carried out by following ASTM standards. The characterization of reinforcements, castings and fractured surfaces was performed using Optical Microscope (OM) and Scanning Electron Microscope (SEM). The mechanical tests and characterization results revealed that there was a significant influence of morphology of reinforcements on mechanical properties of the materials. Particle strengthening and grain refinement strengthening were found to be operative in the composites. The SEM micrograph of fractured surface of composites with MWCNT exhibited the crack bridging effect as strengthening mechanism apart from particle strengthening. The porosity and cluster of reinforcements were observed in the composites with MWCNT more than 1 vol% of and AlN 0.75 vol%.

1. Introduction

A356 is a hypoeutectic aluminum alloy used in automotive, aircraft, flow and structural components. The mechanical and surface properties of A356 alloy are enhanced by fabricating Aluminum Metal Matrix Composites (AMMC's) with ceramic reinforcements. In several research works, researchers have frequently experimented Alumina (Al₂O₃) [1, 2], Silicon Carbide (SiC) [3, 4], Boron Carbide (B₄C), Carbon nanotube (CNT)[5, 6] and Graphite (Gr) as reinforcements in A356 alloy matrix. The AMMC's based on A356 have often exhibited superior mechanical properties due to the refinement of Secondary Dendrite Arm Spacing (SDAS) of alpha Al dendrites and eutectic Si phase in the matrix [5]. Wang et al [7] have reported the enhancement of mechanical properties with morphological changes of eutectic Si phase in the matrix from needle like structure to globular structure after T6 treatment of composites. The changes in the microstructure of A356 alloy matrix have generally occurred as a result of manufacturing processes and post treatment of materials. The commonly used manufacturing processes to fabricate AMMC's are identified as stir casting, compo casting [2], squeeze casting [8], in liquid route processing and hot iso static pressing, cold upsetting in solid phase fabrication process. The process parameters in various manufacturing processes are controlled in order to avoid the agglomeration of reinforcement particles in the matrix. It has been reported in the literatures [8, 9] that the tendency of agglomeration of reinforcement is reduced considerably in the compo casting and squeeze casting processes. Yuan et al [8] have reported that 400 MPa of squeeze casting pressure and 3 min ultrasonic vibration

Table 1. Chemical Composition (Wt%) of A356 alloy.

Element	Si	Mg	Ti	Cu	Zn	Fe	Mn	Al
Wt%	6.89	0.26	0.010	0.008	0.002	0.080	0.004	Balance

treatment on composite melt reduced the size of the SDAS and distributed the nano SiC particles uniformly in the matrix.

Yang et al [10] have expressed that the refinement of alpha aluminum crystals led to the improvement of strength and ductility of the composites. Ghandvar et al [9] have explained that the reinforced SiC particles are found in the inter-dendritic locations after solidification. They have also expressed that the eutectic Si phase are refined and few of the Si phase are nucleated on the SiC particles which reduced the toughness of the material. Cetin et al [11] have reported that SDAS of A356 reduced at higher solidification rate. They have also noted that the tendency of clustering increases when the size of the SDAS is found to be lesser than the average size of reinforcement particle. The near rapid solidification and thermo mechanical treatment of composite melt are reported to reduce the size of the eutectic Si to nano scale and enhanced the properties. The ultrasonic vibration treatment and T6 treatment of composites have changed the long columnar dendrite of alpha alumina into spherical shape and refined it [12]. In situ manufacturing techniques are established as a novel and efficient technique to synthesis AMMC's. Ma et al [13] have produced Al₃Ti inter metallic compound in A356 with T6 treatment. The T6 treatment has enhanced the reaction between Al₃Ti and eutectic Si in base alloy and enriched the presence of Si in the composite. Xu et al [14] have manufactured alumina (Al₂O₃) in A356 at 850 °C using Cobalt Oxide (Co₂O₃) through in situ technique. The nucleated Al₂O₃ particles are observed in the nanoscale and refined the needle like Si into platelet shape. Hosseinzadeh et al [15] have produced Al₂O₃ using the precursors V₂O₅, Al and C through microwave sintering process and subsequent stir casting technique. The ultrasonic sound waves have changed the morphology of *in situ* synthesized rod like Al₃Ti compound into blocky shape [10].

Friction Stir Process (FSP) is another promising solid-state technique to fabricate AMMC's. A356/B₄C composites are manufactured through FSP technique and reported to offer high ductility due to grain refinement and orientation during thermo mechanical process [16]. Choi *et al* [17] have reported that the morphology of Si changed to spherical shape in stir zone of FSPed material and improved the mechanical properties. Friction stir process after *in situ* synthesis of A356/Al₃Ti composites has refined the size of Al₃Ti compound and reduced the porosity of the composites [18]. Strontium (Sr) [13], Scandium (Sc) [19], Lanthanum (La) [20] and Titanium (Ti) are identified as efficient matrix grain refinement agents during *in situ* synthesis process. The addition of small amount of above said elements has reduced the size of SDAS and modified the Si phase in the matrix and enhanced the tensile strength, yield strength and ductility. Large surface to volume ratio of CNT has made it difficult to disperse in A356 matrix melt [6]. Shayan *et al* [5] have fabricated A356/MWCNT composites through two stage casting process. The casted composites have undergone for accumulative roll bonding in order to avoid agglomeration of CNT. In another work, Carbon nanotubes areelectro less plated with Ni-P and Al powder in the perspective of improving the wettability and distribution of CNT in the matrix [21]. Ayyar and Chawla [22] have expressed that the morphology and orientation of reinforcement particles play significant role in affecting the crack growth in composite materials.

Out of extensive literature survey, it is identified that the effect of the combined morphology of reinforcement in A356 alloy has not been sufficiently explored. The AlN is rarely used as reinforcement in A356 alloy matrix. As the AlN is a block-shaped nitride form of major constituent in the base matrix, the wettability of the reinforcement can be improved in the matrix. The MWCNT is a tubular form of carbon and observed to improve the mechanical properties in various matrices. The main objective of this research work is to investigate the influence of multi-walled carbon nanotube (MWCNT) and Aluminum Nitride (AlN) on microstructure and mechanical properties of A356 alloy. In order to enhance the wettability of the reinforcements, the reinforcements are mixed with Al metal powder and introduced into the A356 melt.

2. Materials and methods

A356 alloy rods were purchased from commercial suppliers and its chemical composition was given in table 1. The melting point of A356 alloy was noted to be 610 °C and density was known as 2.67 g/cc. Aluminum nitride (AlN) and Multi-Walled Carbon Nanotube (MWCNT) with purity greater than 98% were received from Sigma Aldrich. Aluminum and graphite powders were purchased with greater than 99% purity from local suppliers in Chennai, India.



Figure 1. SEM Images of (a) Al metal powder (b) AlN (c) MWCNT (d) Graphite.

The micrographs of particles used as reinforcement are shown in figure 1. Figure 1(a) represented irregular shaped Al particles with smooth surface and average size around 10 μ m. Figure 1(b) showed the block shaped AlN particles of Aluminum Nitride (AlN) of average size around 2 μ m. The diameter of multi-walled carbon nanotube was identified in the range of 10 to 20 nm as represented in figure 1(c). The graphite grains were noted with plate like shape and average size of grains were noted in the range of 10 to 20 μ m as displayed in figure 1(d). The reinforcements were mixed in the required proportions using poly vinyl alcohol and water as plasticizer at 70 °C through manual stirring process. The mixture was then heated to 120°C in the perspective of removing moisture content [23].

The composites were manufactured using ultrasonic assisted stir casting equipment. A356 rods were melted in the furnace and the temperature of the melt was maintained at 800 °C. The melt was agitated using stainless steel stirrer at 500 rpm for a period of 3 min and the reinforcements were introduced into the melt. The ultrasonic vibration was offered during the addition of reinforcement. The melt of composite was then poured into the die which was preheated to 250 °C.

The content of MWCNT in the composites was optimized in order to ensure its improved performance in hybrid composites. The details of compositions used for optimizing the content of MWCNT are shown in table 2. The SEM images of mixed particles are shown in figure 2. The micrographs of mixed reinforcements showed well-mixed particles of different shapes explained in figure 1. The prepared composites were machined to the required dimension as per ASTM E8/E8M standard in order to conduct tensile test. The tensile test was carried out on the machine made by Auto Instruments with 5-ton capacity. The crosshead speed of the machine was set as 1 mm min⁻¹.

The composites with MWCNT and AlN were prepared based on the composition given in table 3. The SEM images of the mixed reinforcements are shown in figure 3 and represented the presence of particles. The Energy Dispersive Spectroscope (EDS) results of the mixed reinforcements are shown in figure 4. The spectroscopy results confirmed the presence of elements in mixed particles. Apart from tensile test, the Compression test and Brinell hardness test were also carried out on all the prepared composites by following ASTM E9-89a and ASTM

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Figure 2. SEM images of mixed reinforcements (a) MWCNT-Gr C1 (b) MWCNT-Gr C2 (c) MWCNT-Gr C3 (d) MWCNT-Gr C4.

Composites code	Al metal powder (Vol %)	AlN (Vol %)	MWCNT (Vol %)	Graphite (Vol %)	A356 (Vol %)
MWCNT-Gr C1	1		0.5	0.5	98
MWCNT-Gr C2	1.25	_	0.75	0.5	97.5
MWCNT-Gr C3	1.5	_	1	0.5	97
MWCNT-Gr C4	2.5	—	2	0.5	95

Table 2. Composition of Composites with MWCNT.

Table 3. Composition of Composites with MWCNT and AlN.

Al metal powder (Vol %)	AlN (Vol %)	MWCNT (Vol %)	Graphite (Vol %)	A356 (Vol %)
2	0.5	1	0.5	96
2.25	0.75	1	0.5	95.5
2.5	1	1	0.5	95
3.5	2	1	0.5	93
	Al metal powder (Vol %) 2 2.25 2.5 3.5	Al metal powder (Vol %) AlN (Vol %) 2 0.5 2.25 0.75 2.5 1 3.5 2	Al metal powder (Vol %) AlN (Vol %) MWCNT (Vol %) 2 0.5 1 2.25 0.75 1 2.5 1 1 3.5 2 1	Al metal powder (Vol %)AlN (Vol %)MWCNT (Vol %)Graphite (Vol %)20.510.52.250.7510.52.5110.53.5210.5

E10-15 standards respectively. The composite test specimens were machined to the dimension of 13 mm diameter and 25 mm length. The crosshead speed was maintained as 1 mm min⁻¹ during compression test. The tensile and compression tests of each composite were conducted on three samples and average of the results was considered for the analysis. The load of 500 kg was applied for the dwell period of 15 s in Brinell hardness test. The diameter of the steel ball indenter was 10 mm. The hardness values were measured along the axis of the composites at five different locations and the average of results were plotted.

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Figure 3. SEM images of reinforcement mixture of (a) AlN-MWCNT-Gr C5 (b) AlN-MWCNT-Gr C6 (c) AlN-MWCNT-Gr C7 (d) AlN-MWCNT-Gr C8.

3. Results and discussion

3.1. Microstructure

Optical Micrographs of prepared composites are shown in figure 5. The dark lines in figure 5(a), represented the grain boundary and presence of eutectic Si in the grain structure. In figures 5(b)–(d), showed the presence of MWCNT in the grain boundary. The amount of MWCNT can be witnessed from increasing darkness of the grain boundary. The nanotubes were found to segregate in the grain boundary. When the volume of MWCNT was greater than 1%, the tendency of cluster was identified in the matrix as shown in figure 5(e). The micrographs of composites with AlN and MWCNT are shown in figures 5(f)–(i).

The AlN particles are observed to be distributed uniformly in the matrix as shown in figures 5(f) and (g). When the volume of AlN increased more than 0.75%, the agglomeration occurred as shown in figures 5(h) and (i). Few clusters of AlN particles was found along the interface between MWCNT and the matrix. Pores were also noted in the composites which possessed clusters of reinforcement. The XRD result of prepared composite sample with MWCNT and AlN was shown in figure 6 and it confirms the presence of added reinforcements. The deleterious compound formation was not observed in the XRD result of composite.

The tensile fractured surfaces of composites with MWCNT are shown in figure 7. The micrographs in figure 7(a) showed the presence of dimples and plastic deformation in MWCNT-Gr C1. The crack bridging was found to be operative in the failure of the composites as the fractured MWCNT was noted in the micrograph of figure 7(b). The tear ridges were intensely observed in the grain boundary of composite MWCNT-Gr C3 as shown in figure 7(c). This could be attributed to the resistance offered by the MWCNT which were uniformly distributed in the grain boundary. The fracture occurred in composites MWCNT-Gr C1—C4 was found be



ductile in nature. The pores were noted in the fractured surface of the composites MWCNT-Gr C4. These might have formed during the process due to agglomeration MWCNT in the grain boundary as explained earlier. These pores could have further supported the coalescence of cracks and accelerated the propagation of cracks much faster than other composites. Few slip bands were also noted in the composite MWCNT-Gr C4 as shown in figure 7(d). The mixed mode of ductile and brittle fracture was observed in the composite MWCNT-Gr C4.

The fractured surfaces of composites AlN-MWCNT-Gr C5—C8 are shown in figures 8(a)–(d). The grain refinement was noted in the composites with AlN and MWCNT. The refinement of dendrite arms was observed to increase with AlN content up to 0.75 vol%. The tear ridges were also noted in the fractured surface of composite AlN-MWCNT-Gr C6. This indicated that the MWCNT and AlN in the matrix offered the combined resistance for fracture. The agglomeration of AlN in composites AlN-MWCNT-Gr C7 and C8, around MWCNT, reduced the uniform refinement of matrix dendrites. The refinement at specific spots can be witnessed from micrographs of figures 8(c) and (d).

3.2. Effect of AlN and MWCNT on mechanical properties

The results of tensile test of composites with MWCNT are shown in figure 9. The ultimate tensile strength of the composites increased with increasing fraction of MWCNT up to 1 vol%. A decrement in strength was noted when the volume percentage was raised to 1.5. Hence, the optimum volume percentage of MWCNT in A356 matrix was identified as 1 vol%.

The tensile and compressive properties of the composites are shown in figure 10. The shape of the reinforcements played a vital role in the failure of composites. The tubular nature of MWCNT enhanced the tensile and compressive strength of the composites. The tendency of agglomeration is naturally high in case of MWCNT in the matrix melt. On account of this clustering nature, the ultrasonic vibration followed by stirring process was carried out in the fabrication process. The process parameters still need to be optimized in such a way that the flocculation could be avoided when the vol% increased more than a limit. The combination of AlN and MWCNT in the matrix improved the mechanical properties more than composites with MWCNT alone. The blocky AlN particles offered resistance for the propagation of cracks nucleated in the grain boundary. The path followed by the crack was extended with the presence of different shaped reinforcement particles. Frequent changes in crack path was anticipated and observed so. The blocky nature of AlN particles changed the crack



Figure 5. Optical Microscope images of as-cast (a) A356 alloy (b)MWCNT-Gr C1 (c) MWCNT-Gr C2 (d) MWCNT-Gr C3 (e) MWCNT-Gr C4 (f) AlN-MWCNT-Gr C5 (g) AlN-MWCNT-Gr C6 (h) AlN-MWCNT-Gr C7 (i) AlN-MWCNT-Gr C7 (j) AlN-MWCNT-Gr C8.

path frequently than spherical particles. The particle strengthening was intensely offered the resistance up to 0.75 vol% of AlN along with 1 vol% MWCNT. The increasing volume content of AlN more than the above said limit led to the clustering of particles, which reduced the effect of particle strengthening.







Figure 8. SEM images of fractured surfaces of (a) AlN-MWCNT-Gr C5 (b) AlN-MWCNT-Gr C6 (c) AlN-MWCNT-Gr C7 (d) AlN-MWCNT-Gr C8.



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3.3. Ductility and porosity

The ductility of the composites is shown in figure 11. The ductility of the composites mostly reported to decrease with increasing tensile strength. The ductility of the composites was integrally increased with tensile strength. The effect of agglomeration also affected the ductility as shown in figure 11. This may be attributed to the rapid propagation of cracks due to the reduced crack path. The influence of porosity on the tensile and compressive strength is shown in figures 12 and 13. The porosity was found to be very low in the above said optimum combination of volume fraction of AlN and MWCNT. The peak value was noted in AlN-MWCNT-Gr C8 composite, which could be predicted with the agglomeration of AlN around MWCNT and insufficient control on fabrication process parameters like stirring speed, sonication time and depth of sonication, which need to be explored further for the composites. Figure 12 represented the tensile strength of the composites increased with decreasing content of porosity. As shown in figures 12 and 13, the porosity results of C4, C7 and C8 composites represented the increasing value of porosity cluster). Even though porosity increased in composites C7 and C8, it exhibited improved strength than other composites with MWCNT alone. It explained the fact that the particle strengthening in the composites with AlN and MWCNT was active even with the particle porosity clustering effect. The retainment of strength in such composites showed the effect of combined reinforcement





with blocky and tubular nature. The compressive strength of composites C7 and C8 was affected lesser than tensile strength as shown in figure 13. This could be predicted with the closure of pores during the application of compressive force. The rupture of composites C4, C7 and C8 was still influenced by porosity.

The hardness of the composites is displayed in figure 14. The homogeneous distribution of the particles enhanced the hardness of the composites. The composites C4, C7 and C8 exhibited a decreament in hardness values. This might be because of the clustering effect of reinforcement in the matrix. Because of improper distribution of reinforcement, the potential of hard reinforcement was not observed in the results and the bulk hardness of the composite materials was reduced.

3.4. Strengthening mechanism

The schematic representation of failure of composites is shown in figure 15. The composites with MWCNT along were identified with particle strengthening and bridging effect of MWCNT in the matrix. The major strengthening mechanisms observed in the developed composites with AlN and MWCNT were particle strengthening and strengthening due to grain refinement. The deflection and bifurcation of crack path was observed to be active in the composites which enhanced the energy required for plastic deformation and





strength of the material. The similar energy enhancement and plastic deformation was observed through different mechanism in the composites with only MWCNT that is crack bridging effect due to the breakage of MWCNT. The clustering was identified as prime factor to degrade the properties by reducing the particle strengthening and inducing the porosity around the clusters.

4. Conclusion

The composites with different shape reinforcements AlN (blocky) and MWCNT (Tube) were fabricated using stir casting followed by ultrasonic Vibration treatment. The composites were tested for their mechanical properties and the following conclusions were arrived.

- 1. A significant influence of morphology of different shaped reinforcement was identified in the composites and superior mechanical properties were reported.
- 2. The particle strengthening effect of composites was much pronounced in the composites with combined shape reinforcements in A356 alloy matrix. Grain refinement was observed when AlN was added to A356 matrix.
- 3. The deflection and bifurcation of crack were observed to be active in the composites due to the blocky and tubular reinforcement in the matrix.
- 4. The tensile strength and ductility of the composites increased integrally as the grain refinement enhanced the plastic deformation of the composites. The ductile fracture was observed in the composites with AlN and MWCNT. The mixed mode of ductile and brittle fracture was observed in composites with agglomeration of AlN in the composites.
- 5. The composites with MWCNT alone were found with bridging of crack with broken MWCNT apart from particle strengthening.
- 6. The optimum vol% of reinforcements was identified as 1% for MWCNT and 0.75% for AlN. The particleporosity cluster was identified when the vol% of reinforcement increased more than above said limits. The process parameters need to be optimized to further avoid the agglomeration of particles in the volumes higher than these values.

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Declaration of conflict of interest

The Authors declare that there is no conflict of interest.

Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

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