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Aluminium Reinforced Metal Matrix Composites for Multiwall Carbon Nanotubes -A Review

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ABSTRACT: This review illustrates the production of multiwall carbon nanotubes by stir casting, squeeze casting, rheo casting and powder metallurgy MOCVD techniques. Stir casting technique employs the usage of commercial pure aluminium alloy. Squeeze and rheo casting techniques employ the addition of MWCNTs into A356 hypoeutectic aluminium-silicon alloy matrix with 0.5, 1, 1.5& 2 weight percentage as common for both. These grain dispersions are examined by scanning electron microscope (SEM). This process has facilitated a uniform dispersion of nanotubes within A356 aluminium alloy matrix as well as a refinement of grain size and the hardness property was found to be increased by 80% in stir casting. MOCVD technique employs plasma sintering of aluminium powder with MWCNTs of 0.1-1 wt. %. These are functionalised using H_2SO_4/HNO_3 and at 0.1% wt. of the MWCNTs the maximal increase in the hardness is observed.

KEYWORDS: Multi walled Carbon Nanotube, Stir casting, Squeeze casting, hardness, and strength.

I. INTRODUCTION

Single and multi-wall carbon nanotubes have created enormous expectations in the role of strengthening additives for metallic matrix and polymer composites because of their high strength and stiffness. The CNT were initially invented by Ijima in 1991 [1].Metal matrix/CNT composites have a set milestones in load bearing applications. Due to their high thermal conductivity and low coefficient of expansion they are extensively used in electronic packaging. A356 aluminium alloy is a casting alloy having aluminium, silicon, and magnesium as constituents and possess good strength and ductility as well as excellent casting characteristics, high corrosion resistance, and good fluidity. In traditional method there are two approaches of reinforcement phases to metal matrices: casting methods and powder metallurgy. Stir casting offers a simpler and less cost efficient, which has been applied commercially in the production of particle reinforcing metal matrix composites [2]. This provides opportunity to modify the structure and to optimise solubility and dispersion, allowing innovative applications in the fields of material science, electronics, chemical industries and so on.

Experiments have shown that CNTs have superior mechanical properties over carbon fibres, for example, the stiffness values up to 1000GPa, with an elastic modulus of CNTs as high as 1TPa, strength on the order of 100GPa, and thermal conductivity which ranges up to $6000 \text{Wm}^{-1} \text{ K}^{-1}$ [2 and 3]. The motto of this paper is to upsurge the study on properties of MWCNTs reinforcing Aluminium by stir casting, squeeze and rheo casting and MOCVD techniques.

Element	Si	Mg	Fe	Cu	Zn	Pb	Al
Percentage	6.6	0.30	0.27	0.020	0.01	0.022	Bal

Table 1: Chemical composition (wt. %) of A356 aluminium alloy



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It is evident from the table, the percentage of constituents present in the A356 aluminium alloy and thus the quantity of the alteration in the alloy for experimentation can be decided with the above reference. Powder metallurgy processing technique involves repeated cold welding, fracturing, and rewelding of particles using a high energy ball milling process. Itwasfoundthat the dispersionof MWCNT sinMMNCsis considered validating severalprocessing variables including pre-treatment of MWCNTs, type of milling, ball-to-powder weight ratio, milling speed, milling time, and process control agents [4 and 5]. By using a combination of rheo casting and squeeze casting techniques which includes the infiltration, mechanical stirring, and the pressure casting such asdie and squeeze casting stages nano composites are produced [2]. The influencing aswellas its strengthening mechanism are discussed further.

II. EXPERIMENTAL REVIEW

STIR CASTING

A. Material for experiment

Commercial purity aluminium ingots with Multi walled carbon nanotubes (MWCNTs) are used in the study. MWCNTs, are one dimensional multi pipe wall nano particles provided by Nano shell. Melting point is 3652-3697°C and the density ranges from 1~2gm/cc. The properties attributed with commercial purity aluminium ingots are 99.99% Aluminium balance, 2.70gm/cc density, with a melting temperature of 660.32°C which is considered for the experiments [7].

B. Purification of MWCNTs

The MWCNTs were made to disperse in a solvent (Ethanol) and were subjected to ultrasonic vibrations for 20 min and then poured to obtain pure MWCNTs. By using ultra sonic equipment the nanotubes dispersed are then dried completely by heating to its melting temperature. The dried MWCNTs can be used as reinforcement. It is done to remove the impurities present in the MWCNTs. This process is done for all the respective reinforcement weight percentages [7].

C. Preparation of composites

Aluminium samples were melted in the furnace upon addition of carbon nanotubes into melts and mixed with the help of stirring bar. MWCNTs of 0.5 wt.%, 1 wt.%, 1.5 wt.% and 2 wt.% of carbon nanotubes weight percentages was added into melt aluminium to get the composites [7]. The Aluminium /MWCNTs is poured into die and solidified.

D. Performance test of composites

The grain distribution of MWCNTs in matrix was observed by using Scanning electron microscope (SEM). Brinell hardness tester was considered to evaluate the hardness of the samples with various volume percentages [7].

E. Strength

Tensile test was carried out using universal testing machine (UTM). The samples were prepared as per the ASTM 8 standards and the specimen gauge length of 40mm, diameter 13mm and reduction in diameter is 5mm. Applying static loading conditions the test was conducted for yield strength & tensile strength [7].

RHEO AND SQUEEZE CASTING

A. Preparation of Al/MWCNT Metal Blocks Reinforcement using pre synthesis of CNTs

Carbonnanotubes (CNTs) were produced by electric arc discharge. The arc is generated between two electrodes using distilled water as a medium. The cathode and anode materials both were made up of graphite (99.99% pure). Tests were performed under AC arc discharge, 75A and 238V. BycompressingamixtureofMWCNTs,Al,andstearicacids in a steel mould for 15 minutes blocks were produced. Using Stearic acids as adhesive agents the ball milling process was performed in a rotary ballmillusing stainlesssteelballs withtheballtopowdervolumeratioof8:1 [2].Themixture was milledat a speed of 200rpmfor8hr.



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B. Process of melting A356 Aluminium Silicon Alloy

A356 aluminium silicon alloy which is hypoeutectic in nature was used in the experiment, Mgimproves the wettability of their enforcement in the matrix. A sample weighing 350g of the A356 Al alloy

was melted at 660°C in a graphite crucible using an electrical resistance furnace. Upon completion of melting the degassing was done using a solid dry (hexachloroethane) degasser and argon gas was introduced toprevent hydrogen

entrapment in the process, vthe alloy was allowed to cool to the semisolid temperature of 601°C judged using a K-type thermocouple. Aluminium/MWCNTs billets were put into the melt and stirring was continued for a minute in semisolid state. Aftercompleting the addition of aluminium/MWCNT, the process of agitation was stopped and the molten mixture was poured to a medium of preheated low carbon steel mould and immediately squeezed during solidification [2].

C. Experimental analysis of the sample

The analysis of the composite samples was done from the cast nano composites and they werecut by an auto-cutter for microstructural examinations using optical microscopy. Samples were ground under water using silicon carbide abrasive discs of increase in grit size up to 2000 grit silicon carbide papers. The intermediate and final states of MWCNTs in the pool of alloy matrices and Al-MWCNTs powder mixture were analysed using SEM equipped with energy dispersive X-ray element analysis system to ensure the existence of CNTs.Repeated trials for tensile and compression tests were carried out from each composite ingot and the tensile and respective values were averaged [2].

MOCVD TECHNIQUE (metal organic chemical vapour deposition)

A. Al-CNTs Powder Preparation

0.5% of CNTs was added to the Al powders and there are 3 different mixing methods, namely high energy and low energy ball millings, and polyester binder-assisting (PBA)techniques. Mechanical alloying was carried using a Germany planetary ball mill machine. A High energy ball milling for sample with Al-0.5 wt. % CNT powders were mixed by stirring and then transferred to jar. To avoid severe cold rolling, stearic acid was added to the mixture during the ball milling. Ball to powder weight ratio was 5:1 with a revolution speed of 200 RPM [6, 8, 9, & 10]. Low energy ball milling for the same sample and balls as used in high energy ball milling were initially loaded and

Low energy ball milling for the same sample and balls as used in high energy ball milling were initially loaded and then blended by a horizontal rolling machine and was rolled for 4h at 200rpm. Polyethylene glycol (PEG) in a form of flake with 20,000 molecular weight was hand mixed with CNTs in a mortar. Equipment used for mixing was Hake twin screw mixer. Size of powders milled for duration of 6 h and was found to be 48.4 nm. Extrudes of CNT-Al, is 56.6 nm which is slightly greater than the grain size of ball milled powders for better nanostructure [6].

B. Powder metallurgy technique

The aluminium powder (purity of 99.0%, grain size of 70μ m) and CNTs were mixedhomogeneously using hand grinding for 30min, and ethanol was added to avoid possible oxidation. Multiwall carbon nanotubes (MWCNTs) were treated within the concentrated nitric acid as reflux for 0-25 h in order to purify and disperse the tangled MWCNTs. The weight loss of MWCNTs increased as the reflux time increased. When the content of MWCNTs is less than 1.0 wt. %, MWCNTs can uniformly distribute on the surface of Al powders, but when the content of MWCNTs is 2.0 wt. %, MWCNTs entangle with each other on the surface of Al powders [6 and 11]. The technical methods employed in producing and mixing aluminium powder along with their morphology is explained in the below table 2.



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Mixing technique	Al powder morphology	CNT morphology	Dispersion effect		
Low energy ball milling	Round shape with severe change	Medium change	Dispersed within A1 powder though agglomerates still existed		
PBA Round shape		Negligible charge	Coated with A1 powder through agglomerates still existed		
High energy ball milling	Round shape with severe change	Severe damage	Effectively dispersed though insufficiently distributed		

Table 2: The summary of the MWCNTs dispersion results from the different mixing techniques

C. MOCVD methodology (Metal organic chemical vapour deposition)

The aluminium powder with 99.99% purity consisting of an average particle size of about 6.1 nm and pure MWCNTs having 2% of amorphous form of carbon and graphite produced by MOCVD method has their role to play in this study. The FMWCNTs and Al powder in ethanol using a joint ultrasonic treatment and intensive stirring to ensure a uniform FMWCNTs dispersion in the Al powder. The dried suspensions of 0 wt.%, 0.1 wt.%, 0.25 wt.%, 0.5 wt.%, 1 wt.% FMWCNTs were prepared. Further by using spark plasma sintering of powders at 600°C under 50 MPa applied stress for 20 min in a vacuum. Finally, cylindrical specimens of the Al-MWCNTs were fabricated [12].

III. EXPERIMENTAL RESULTS AND DISCUSSIONS

STIR CASTING

A. Microstructure

The multi walled carbon nanotubes were examined preliminarily by scanning electron microscope (SEM) and are shown in the figure (b)and then are examined by XRD. SEM images in figure (b) clearly depict that the particles are of carbon. XRD in fig (a) patterns depicts the multi walled carbon nanotubes images which reveals that the peak points are of carbon. The Casted composite sample reinforcement distribution over the matrix is uniform which can be observed in Fig a and Fig b [7 and 13].



Fig 1: (a) and (b) shows the analysis of XRD pattern and also the SEM image of the MWCNTs

B. Hardness

Hardness of the multiwall carbon nanotube reinforced with commercial purity aluminium composite processed by stir casting samples were determined by using Brinell hardness testing machine. Table.3 shows, hardness which increases with increase in Wt. % of MWCNTs [7].



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C. Strength Properties

The strength properties were evaluated by preparing the standard tensile test specimen as per the ASTM 8 standard. The effect of MWCNTs on Tensile and Young's modulus is shown in table 4, which indicates the tensile strength and young's modulus of casted MWCNT/Al composites increases as the weight percentage of MWCNTs increased [7].

Wt. % of MWCNTs	Hardness (BHN)	Composition	Experimental Density (g/cc)	Young's modulus (GPa)	Tensile strength (MPa)
Aluminium + 0 Wt. % of MWCNT	38	A1 + 0 Wt. %of MWCNT	2.70	70.12	123
Aluminium + 0.5 Wt. % of MWCNT	55	A1+0.5 Wt.%of MWCNT	2.58	74.53	147
Aluminium + 1 Wt. % of MWCNT	59	A1+1 Wt.%of MWCNT	2.61	79.5	151
Aluminium + 1.5 Wt. % of MWCNT	66	A1+1.5 Wt.%of MWCNT	2.63	83.72	152
Aluminium + 2 Wt. % of MWCNT	69	Al + 2 Wt. %of MWCNT	2.68	85.67	150



Wt. % of MWCNTs SQUEEZE AND RHEO CASTING

A. Microstructure

The X-ray diffraction pattern of the produced CNTs showed the strong and sharp reflection peak as in figure3. The presence of this peak in the XRD pattern of CNT indicates the concentric appearance of cylindrical nature graphene sheets present together and the nanotubes are multiwall in nature. The SEM images of aluminium powder shows the average particle size before and after being mixing with MWCNTs by ball milling at 200rpm in order to increase the homogeneity and dispersibility of MWCNTs in theme during stirring as indicated in the figure 2. Due to mechanical stirring and the addition of MWCNTs, the dendritic structure fragmented and the primary aluminium grains become moreuniformandsmaller than their corresponding of monolithic alloy samples and with increasing weight fraction of MWCNTs the grain refining also increases. The results in figure 3 indicate that MWCNTs have a major effect on the grain refining of A356Al-Sialloys. The effectiveness of squeezing is to break up agglomerates, help the redistribution of MWCNTs with also metal matrix and the formation of the dimples and cleavage areas are identified [2].



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Figure 2: (c), (d), (e), & (f) depicts the change in microstructural behaviour metal matrix along with MWCNTs further the dimples and cleavage areas are identified [2].

Figure 3: shows the graphical representation of XRD analysis with respect to microstructure by plotting Intensity v/s 20

B. Hardness and Strength factor

The hardness of cast samples has increased by the addition of MWCNTs. Itcanbeobservedfrom the figure 4 that the hardness values have slightly increased for 0.5 and 1% weight fractions, while they have increased for 1.5, 2, and 2.5wt% by a considerable value. Due to difference in the thermal expansion amongst matrix and room temperature the strengthening and hardening factor of the MWCNTs is observed to be increased. This behaviour may be also partly contribute to the decrease in the grain size of the A356 matrix of the composite samples by addition of MWCNTs and stirring action. After which CNTs, like other reinforcements strengthen the matrix by creation of a high density dislocation during cooling to room temperature due to the difference of the coefficients of thermal expansion between the CNTs and the matrix. It was observed by the graph in figure 4, that the density of the nanocomposites decreases with increasing weight percentages of MWCNTs. The reason for the decrease in the density is because of theadditionoflightweightandhighvolume CNTs compared to the matrix material, which increases the porosityofthenanocomposite samples. Figure 4 depicts the hardness variation al-356 alloy with MWCNT reinforcements [2 & 14].



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---- Hardness (RB)

Fig 4: Effect of MWCNTs different weight fractions on hardness of the A356 alloy.



Fig 5: Tensile properties of al-cast A356 alloy and A356/MWCNTs Nano composites.

The compressive strength of nano composites at optimal value is more than that of monolithic alloy due to the effect of grain size and the strength of a nano composite can primarily be attributed to grain refinement, presence of reasonably distributed MWCNTs, dislocation generation due to elastic modulus mismatch and coefficient of thermal expansion mismatch between the matrix and reinforcement phase. Presence of interfacial phases can act as medium to transfer stress from the matrix to the CNTs during the failure failure formation incompression testing and can result inweakening of the composites. The mechanical properties like yield strength, ultimate tensile strength and standard deviation of the same with respect to the standard deviation is mentioned in the fig 5 [2 & 15].



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POWDER METALLURGY AND MOCVD TECHNIQUE

A. Microstructure

Study of FMCNTs in Al-powder SEM images of the FMWCNTs dispersion in composites are shown below in figure 6 and 7. There are small pores of 300–700 nm and no definite boundaries between the particles in the Al/0 wt. % FMWCNTs specimen. Increase in the FMWCNTs amount leads more distinct and longer boundaries between particles indicating a significant contribution of nanotubes to the Al powder sintering process [12]. The variations altogether are represented in the figures 6, 7, 8 & 9 respectively.



Fig 6: a and b shows0.5 wt. % pure MWCNTsFig 7: c and d shows 0.5 wt. % FMWCNTs.



Fig 8: e and f shows Al/0 wt. % FMWCNTs and Al/0.1 wt. % FMWCNTs.

Fig 9: g and h shows Al/0.5 wt. % FMWCNTs and Al/1 wt. % FMWCNTs

B. Mechanical properties (hardness and strength)

Mechanical properties of the composites are represented in Table 5. A cross correlation is observed between microhardness and tensile strength values. Peak values were registered for 0.1 wt. % FMWCNTs amount, at which microhardness and tensile strength demonstrate an increase of 16% and 30% respectively, relative to Al/0% wt. % FMWCNTs samples. Strain in inversely proportional to the weight of FMCNTs. he specimen with the best tensile strength maintains high enough strain of about ~13%. Yield strength is within 85–101 MPa [12].



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Specimen	Property					
	Micro-hardness (MPa) (±1–2 MPa)	Tensile strength (MPa)	Yield strength (MPa)	Strain (%)		
A1+0%FMWCNTs (test specimen)	43	126	97	24		
A1+0.1% FMWCNTs	50	163	101	13		
A1+0.25% FMWCNTs	47	151	85	6		
A1+0.5% FMWCNTs	45	146	100	11		
A1+1% FMWCNTs	46	132	87	4		

Table 5: Mechanical properties of the FMWCNTs-Al metal matrix composites.

IV. CONCLUSION

This report has the discussions made by the implementation of three major methods of producing Aluminium metal matrix composites with MWCNT reinforcements. It can be seen that the reinforcement effect of MWCNTs to the commercial purity aluminium matrix is fairly effective. The hardness of the composites increases with increase in reinforcements.

- (i) A356aluminiumalloysreinforced with multiwall carbon nanotubes (MWCNTs) in wt. % of 0,0.5,1,1.5,2, and 2.5 wt% we resuccessfully produced and analysed by an approach using a combination of the ocasting and squeeze casting techniques.
- (ii) Uniformly dispersed MWCNTs into the melt resulted in good distribution and less agglomeration of CNTs in the matrices of the produced A356/MWCNTs nanocomposites which resulted in improved mechanical properties as the yield and ultimatetensilestrengths of the castings [2].
- (iii) The ultimate tensile strength and yield strength of 1.5wt% A356/MWCNTs nanocomposite are enhanced by 50% and 60%, respectively. Hardness measurement revealed also an improvement compared to the A356 monolithicalloy. As the weight fraction of MWCNTs increases, the hardness increases.
- (iv) Large amount of MWCNTs adversely affected the material strength due to agglomeration or poor wettability with the matrix. An effect of nanotubes amount on the SPS process of Al powder has been observed that addition of 0.1 wt. % FMWCNTs can lead to an increase of 16% and 30% of micro-hardness and tensile strength, respectively. Also, additional treatments are planned to study their contribution to enhancement of mechanical properties of composites [2].

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