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Effect of MWCNTs content on the characteristics of A356 nanocomposite

R.M. Rashad ^{a,*}, O.M. Awadallah ^b, A.S. Wifi ^b

^a Department of Mechanical Engineering, British University in Egypt,

- 11837 P.O. Box 43, Cairo, Egypt (On leave from Cairo University)
- ^b Department of Mechanical Design and Production, Cairo University, 12613 Giza, Egypt
- * Corresponding e-mail address: ragaie.hassan@gmail.com

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ABSTRACT

Purpose: The objective of the present paper is to study the effect of Multiwalled Carbon Nanotubes (MWCNTs) content on the mechanical properties of the A356 hypoeutectic aluminum- silicon based nanocomposite.

Design/methodology/approach: The semi-solid route stir casting technique is used for composite processing. MWCNTs and Aluminum powder are premixed by ball milling and green compacted to form small billets. Al-MWCNTs billets are added to the melt and incorporated by vigorous mechanical stirring. The mechanical and metallurgical properties of the produced composite are characterized by, scanning electron microscopy (SEM), optical microscopy, and tensile testing DIN 50125.

Findings: MWCNTs are successfully incorporated into the A356 melt up to 1.5 % weight fraction. SEM analysis revealed a uniform dispersion of MWCNTs with good interfacial bonding between the matrix and the MWCNTs. The ultimate tensile strength and elongation of the produced composite are increased by 34% and 250% respectively compared to their corresponding values of monolithic alloy.

Research limitations/implications: The research was carried out based on MWCNTs only with a range of percentage additions; it could be extended to single Walled CNTs and graphene sheets with different percentage additions. Stirring time and speed as well as heat treatment can also be applied as further study.

Practical implications: This work helps in introducing novel technique in dispersing Nano particulates in metal matrix composites. This could be good potential for new developed composites.

Originality/value: A novel approach for MWCNTs reinforcement addition technique is implemented. This technique results in a uniform dispersion of MWCNTs with good interfacial bonding between the matrix and the MWCNTs. **Keywords:** Composites; Casting; Multiwalled carbon nanotubes; Aluminium silicon alloys

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<u>1. Introduction</u>

Carbon nanotubes (CNTs) are nanostructures formed from rolled up graphene layers. Owing to their electronic, mechanical,

chemical, magnetic and optical properties carbon nanotubes are currently a subject of extensive scientific research. Two techniques are used for CNTs synthesis namely the carbon-arc process and the catalytic decomposition [1]. The main CNT applications areas are: Electron Technology (flat displays, FED)

Microelectronics (transistors FED, hypothetical quantum computers), Nanocomposites (reinforced with carbon nanotubes) and electrochemistry (energy storage, lithium ion batteries) [2]. For the past few years successive growth of the interests in the material composites can be noticed, mostly light metal based, which have found their applications in many industry branches, among others in the aircraft industry, automotive -, and armaments ones, as well as in electrical engineering and electronics [3]. Special attention is given to the metal composites reinforced by the ceramic particles. Metal Matrix Composites are exposed to the impact of high temperature, and successfully used as elements subjected to intensive wear and also elements in power transmission systems with low friction coefficient and high vibration damping capability [4]. Two main development directions of manufacturing metal matrix composite materials technology are observed: powder metallurgy and casting methods, with specific modification of the pressure infiltration of the porous, ceramic preforms with liquid metals alloys [3].

J.W. Kaczmar et al developed an EN AC-44200 aluminium based composite reinforced by alumina particles. Porous ceramic preforms were produced with 60%, 70%, 80% and 90 porosities and infiltrated by the molten alloy using squeeze casting technique at 100 MPa. The produced composite samples revealed low porosity and good bonding at the interface. Moreover, the Hardness, Bending strength, and compression strength increased with increasing volume content of alumina particles with optimal values at 40% vol. of alumina particles. However, the tensile strength and impact strength of the composites showed degradation with the increase in alumina content [5].

Qianqian Li et al. [6] successfully dispersed MWCNTs on Mg alloy chips, and then the chips are added to the melt with vigorous stirring. They found a significant improvement up to 36% in compressive yield strength and ultimate compressive strength only by addition of 0.1 wt% MWCNTs.

Xiaoshu Zeng et al. [7] developed a new approach by adding MWCNTs into a magnesium–aluminum alloy matrix. In this study, MWCNTs are added into molten alloys in the form of blocks consisting of the mixture of MWCNT and metal powders. The powder is mixed using the ball milling (at 100 rpm for 1h, 8h, and 16h) process to obtain a mixture with final compositions of the MWCNT blocks to achieve well-dispersed MWCNTs. The ball milled mixture then compressed in a steel mold. The optical microscopy and scanning electron microscopy revealed a uniform dispersion of CNTs within the magnesium alloy melt without evidence for any reaction between the CNTs and the metallic matrix. They observed a maximum tensile strength at 210.3 MPa and an elongation rate of 8.56%, which represents an increase of 30.8% and 124.1%, respectively.

Abbasipour et al. [8] produced CNTs reinforced - A356 (Aluminum silicon alloy) composite using stir casting and Compocasting routes. They have suggested a novel technique for CNTs addition which includes the injection of Ni-CNT coated aluminum particles instead of raw CNTs. This was achieved by deposition of CNTs on aluminum particles of less than 100 μ m in diameter using Ni-P electroless plating technique. They concluded that the new technique seems to be effective in overcoming the problems associated with poor wettability, agglomeration and gravity segregation of CNTs in the melt. In addition, CNTs can significantly refine both full liquid and semi-solid (0.3 solid fraction) cast microstructures of the A356 alloy.

From the above literature, it is evident that new trends are implemented for ceramic particles and CNTs dispersion and melt processing of Metal-Matrix Composites (MMCs). However, only few attempts are reported dealing with semi-solid processing of A356 aluminum silicon alloy. The objective of the present paper is to study the effect of MWCNTs content on the metallurgical and mechanical properties of A356 based nanocomposite using a modified stir casting technique which combines both the infiltration and mechanical stirring effects.

2. Experimental work

2.1. Synthesis and preparation of MWCNTs reinforcement

MWCNTs synthesized by arc discharge having an average diameter of 40nm and length of 3μ m are used as reinforcement for the A356 hypoeutectic aluminum silicon alloy. Special arc discharge method was implemented in order to obtain high quality MWCNTs with high aspect ratio which revealed a high surface area to volume ratio, this surface area to volume ratio may facilitate the incorporation and wettability of MWCNTs by the molten metal [9]. Fig. 1 shows SEM micrograph for the purified MWCNTs that are used during the current work.

Sample of purified MWCNTs is mixed with pure Aluminum powder (avg. size 100 μ m) with a weight ratio of 1:6 respectively. Stearic acid is then added as process control agent. The MWCNTs are dispersed with the Aluminum powder by ball milling at 200 rpm for one hour and the mixture is then compacted at 90 MPa for 10 minutes to produce MWCNTs-Al porous preforms shown in Fig. 2a. The prepared blocks with length *L*: 52 mm, width *W*: 28 mm, and thickness *T*: 9 mm are cut into small billets that are introduced into the melt by vigorous stirring (Fig. 2b).

Pure Magnesium is added to the melt with a 0.5% weight fraction in order to improve the wettability of the matrix alloy because it serves as a surfactant [12].

2.2. Reinforcement addition, composite processing and characterization

Stirring process is performed using four blades stirrer at 500 rpm for 1 minute in semi-solid state (590-600°C) after the pre-infiltration of the preform by the molten metal for 30 seconds. Argon gas is purged to prevent hydrogen entrapment during melt processing. Fig. 3 shows a schematic diagram for the apparatus used for composite preparation. Samples with four different weight fractions (0.5%, 1.0%, 1.5%, and 2.0%) are processed and poured into a preheated cylindrical metallic mould.

Al-MWCNTs powder mixture is investigated using Scanning Electron Microscopy (QUANTA FEG 250) in order to check the de-agglomeration of the MWCNTs after ball milling. In addition, the fractured and etched surfaces for selected composite samples are investigated using Field Emission Scanning Electron Microscopy (FESEM-LEO Supra 55) and Energy Dispersive X-ray analysis (EDX) to check the integrity of MWNCTs dispersion in the metal matrix. Furthermore, tension test (samples according to DIN 50125) and microstructure examination are carried out.



Fig. 1. SEM image for MWCNTs used in the current study



Fig. 2. a) The green compacted powder in the form of rectangular block, b) the small billets of reinforcement

3. Results and discussions

3.1. Scanning electron microscopy analysis of ball milled powder

The SEM investigation of the ball milled mixture revealed good dispersion occurred with little damage could be noticed for the structure of MWNCTs. Fig. 4a shows the large Al particles after ball milling. The MWCNTs deposited on the Al particles as shown in Fig. 4b. Some bends and distortions could be observed on the MWCNTs due to the effect of ball milling, while there is no evidence for the occurrence of cold welding for the matrix powder. The Aluminum - MWCNTs mixed particles also are observed to become larger than the initial particle size, with average size of 250 μ m.



Fig. 3. The apparatus used for preparation of the Metal Matrix Nanocomposite: (1) Power and temperature control unit, (2) Supporting frame,(3) Thermocouple, (4) Electric motor, (5) Four blades Stirrer, (6) Argon inlet, (7) Al-MWCNTs billets open, (8) Crucible, (9) Heating coil, H: molten metal height, D: diameter of crucible



b)





Fig. 4. (a) Aluminum particles mixed with MWCNTs after ball milling (b) High magnification FESEM micrograph depicts the dispersion of MWCNTs over the Al powder

3.2. Scanning electron microscopy analysis of the MWCNTS reinforced-A356 based nanocomposite

Figs. 5 and 6 (a, b) show FESEM micrographs for the etched surface of samples reinforced with 1.0 and 1.5wt% MWCNTs respectively. The SEM images revealed a successful incorporation and homogenous dispersion of MWCNTs in the matrix alloy. Moreover, the SEM images imply an indication for efficient bonding between the MWCNTs and matrix alloy. These images indicates that samples reinforced with 1% and 1.5 wt% MWCNTs exhibited good wetting and infiltration by the molten matrix alloy.

It is expected that wetting between matrix alloy and MWCNTs will be poor which leads to some difficulties to introduce relatively high amounts of CNTs into the alloy because the surface tension of MWCNTs is 100 - 200 mN/m, while that of molten aluminum silicon alloy is approximately 800 mN/m [10]. However, the interfacial reaction and formation of aluminum carbide Al_4C_3 is found to reduce the contact angle to 45° [11], consequently the formation of interfacial carbides favors the wetting and infiltration of the liquid metal into the MWCNTs porous preform [12].

a)



Fig. 5. FESEM images showing composite with 1.0 wt% **MWCNTs**

In case of low silicon alloys, the formation of aluminum carbide is preferred over silicon carbide.

Fig. 7 depicts FESEM images with different magnifications for the samples reinforced with 2.0 wt% MWCNTs. The SEM study shows positions of agglomerations of MWCNTs. In addition, there is no homogenous distribution observed for the investigated samples. This behaviour may be attributed to the flotation and un-recovery for some parts of the compacted blocks; this may due to the relatively large amount of MWCNTs which strongly affected the metal infiltration into the compacted block. Consequently, samples reinforced with 2 wt% MWCNTs experienced difficulties during infiltration of liquid metal into the MWCNTs, thus small pieces of micro-sized MWCNTs clusters are left and lead to degradation for composite properties.

a)



Fig. 6. FESEM images showing composite with 1.5 wt% **MWCNTs**

As shown in Fig. 8, the EDX analysis for 1.0% and 1.5 wt% reinforced samples confirm the existence of MWCNTs. In case of 1.5 wt % sample, the high carbon peak suggests the formation of aluminum carbide, which enhances the interfacial bonding and improve the mechanical properties of the composite as discussed above.

Further investigation will be done using Transmission Electron Microscopy (TEM) study to ensure the formation of aluminum carbide. The oxygen peak may be attributed to porosity formation and aluminum oxide inclusions introduced during melt processing.



Fig. 7. FESEM images showing composite with 2.0 wt% MWCNTs \backslash



Fig. 8. EDX analysis for composite samples (a) 1.0 wt% MWCNTs (b) 1.5 wt% MWCNTs

3.3. Microstructure observation of nano-composite

Fig. 9a shows the optical microstructure of the base alloy A356 processed at the same conditions of the composite. The dendritic microstructure could be easily traced as a result of the casting conditions because there is no reinforcement added in order to modify the primary aluminum grain size.

Fig. 9b indicates the microstructures of 1.0 wt% MWCNTs reinforced composite. Due to mechanical stirring and addition of MWCNTs, the dendritic structure fragmented and the primary aluminum grains become more uniform and smaller than their corresponding of monolithic samples. The overall morphology changed with the incorporation of the MWCNTs which is obvious not only for the primary Aluminum, but also for the eutectic Silicon phase that forms globular structure in contrast to the coarse grains of the base matrix.



Fig. 9. Optical micrographs of the (a) A356 monolithic alloy (b) 1.0 wt% MWCNTs reinforced composite

3.4. Mechanical properties of nanocomposite

Fig. 10 indicates that the tensile strength increased with increasing the MWCNTs weight fraction. The tensile strength increases from 155.4 MPa for the monolithic alloy to an average value of 201.2 MPa for 1.5wt% MWCNTs reinforced composite (the maximum value is 208 MPa). The increased tensile strength is due to increasing the dislocation density in the alloy around the MWCNTs as a result of thermal expansion mismatch between the MWCNTs and the matrix material [13].

However, increasing the amount of MWCNTs above 1.5% is found to deteriorate the tensile strength of the composite to a value of 170.4 MPa for 2.0 wt% MWCNTs reinforced composite. The lower value of strength for the 2.0 wt% reinforced composite may be attributed to the difficulties of MWCNTs billets infiltration during stirring, which results in agglomerated and un-recovered MWCNTs clusters.

Another possibility for composite strength degradation is the high possibility for porosity formation and oxide inclusions introduced during melt processing. This result is confirmed by SEM and EDX analysis as explained previously.



Fig. 10. Effect of the MWCNTs different weight fractions on the tensile strength of the A356 alloy processed in semi-solid state

The addition of MWCNTs leads to improvement of the composite ductility as shown in Fig. 11. The elongation percentage increased from 1.5% for the monolithic alloy to a maximum value of 5.3% for 1.50 wt% MWCNTs reinforced composite. The simultaneous increase of composite ductility with tensile strength is attributed to the slip mode transition produced by the presence of MWCNTs which depends on the MWCNTs/alloy interaction [14]. Because MWCNTs represent very fine precipitates, the plastic deformation changed from dislocation reinforcement shearing to dislocation reinforcement bypassing. But due to the high strength of CNTs, the MWCNTs impede the dislocation motion and collapse them around the MWCNTs/A356 interface [15]. The activation of such cross slip modes is responsible for the increased ductility. The results obtained during the current study are similar to other researcher's work on Magnesium and Aluminum alloys [16,17]. Further TEM investigations will be done in the future to precisely detect the mechanism responsible for the simultaneous increase in tensile strength and ductility.

The SEM micrograph for the fracture surface of 1.5 wt% MWCNTs reinforced sample (Fig. 12) shows nano-voids coalescence revealing a dimpled surface as a sign for ductile fracture. The occurrence of nano-dimples suggests a strong interfacial bonding between the matrix and reinforcement since the crack cannot propagate neither through the high strength MWNCNTs nor the interface. Consequently, the only possible way for the crack is to propagate through the weaker matrix, but the uniform dispersion of the MWCNTs prohibits the void coalescence and growth. This fracture mechanism may be responsible for the increased ductility of the nanocomposite.



Fig. 11. Effect of MWCNTs different weight fractions on the elongation % of the A356 alloy processed in semi-solid state



Fig. 12. Fracture surface of 1.5 wt% MWCNTs reinforced sample showing a dimpled surface

4. Concluding remarks

The present study introduced a novel processing approach for MWCNT/A356 Nanocomposite using a modified stir casting technique. In this approach MWCNTs and Aluminum powder are premixed by ball milling and green compacted to form small billets. Al-MWCNTs billets are added to the melt and incorporated by vigorous mechanical stirring. The SEM study proved the successful incorporation of MWCNTs in the matrix alloy with good dispersion up to 1.5%Wt.

In addition, Tension testing revealed a simultaneous increase in the ultimate tensile strength and elongation percentage with optimal MWCNTs weight fraction of 1.5 % under the present experimental conditions. Moreover, the microstructure observations imply grain refining for the prepared nanocomposite samples.

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