Microstructure Analysis of the Carbon Nano Tubes-Aluminum Composite with different Manufacturing Conditions

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Abstract: ***The interest in carbon nano tubes (CNTs) as reinforcements for aluminum (Al) has been growing considerably. Efforts have been largely focused on investigating their contribution to the enhancement of the mechanical performance of the composites. The uniform dispersion of CNTs in the Al matrix has been identified as being critical to the pursuit of enhanced properties. Stir Casting method as a mechanical dispersion technique has proved its potential. In this work, we use Stir casting method to disperse up to 4 wt. % CNT in an Al matrix. The effect of CNT content on the Physical properties of the composites like SEM, XRD was investigated. The improvement of physical properties for composites of Al – CNT has been compared with pure aluminum [1100].***

Keywords: Scanning electron microscope, XRD, Carbon Nano tubes, Aluminum.

1. Introduction

Among discontinuous metal matrix composites, stir casting is generally accepted as a particularly promising route, currently practiced commercially. Its advantages lie in its simplicity, flexibility and applicability to large quantity production. It is also attractive because, in principle, it allows a conventional metal processing route to be used, and hence minimizes the final cost of the product. This liquid metallurgy technique is the most economical of all the available routes for metal matrix composite production (Surappa, 1997), and allows very large sized components to be fabricated. The cost of preparing composites material using a casting method is about one-third to half that of competitive methods, and for high volume production, it is projected that the cost will fall to one-tenth (Jolla, 1988). In general, the solidification synthesis of metal matrix composites involves producing a melt of the selected matrix material followed by the introduction of a reinforcement material into the melt, obtaining a suitable dispersion.

The next step is the solidification of the melt containing suspended dispersions under selected conditions to obtain the desired distribution of the dispersed phase in the cast matrix. In preparing metal matrix composites by the stir casting method, there are several factors that need considerable attention, including the difficulty of achieving a uniform distribution of the Reinforcement material, weldability between the two main substances, porosity in the cast metal matrix composites, and chemical reactions between the reinforcement material and the matrix alloy. In order to achieve the optimum properties of the metal matrix composite, the distribution of the reinforcement material in the matrix alloy must be uniform, and the wet ability or bonding between these substances should be optimized. The literature review reveals that the major problem was to get

homogenous dispersion of the ceramic particles by using low cost conventional equipment for commercial applications. In the present work, a modest attempt have been made dispersion of Multi wall carbon nanotubes (MWNT) particles in Al matrix fabricated with the help ofMelt stirring equipment. Composition of Al – MWNT, Above composition done under method of stir casting. An effort has been made to establish a relationship between Scanning electron microscope and X-Ray diffraction for Al – MWNT.

1.1 Fabrication of the Amcs

There are many processes viable to fabricate AMCs; they can be classified as solid state, liquid-state and deposition processes.

In ***solid-state processes***, the most spread method is *powder metallurgy* PM; it is usually used for high melting point matrices and avoids segregation effects and brittle reaction product formation prone to occur in liquid state processes. This method permits to obtain discontinuously particle reinforced AMCs with the highest mechanical properties. These AMCs are used for military applications but remain limited for large scale productions.

In ***liquid-state processes***, one can distinguish the infiltration processes where the reinforcements form a pre form which is infiltrated by the alloy melt with pressure applied by a piston or by an inert gas (*gas pressure infiltration* GPI) and without pressure . In the last case, one can distinguish (a) The reactive infiltration processes using the wetting between reinforcement and melt obtained by reactive atmosphere, elevated temperature, alloy modification or reinforcement coating (*reactive infiltration*) and (b) the dispersion processes, such as ***stir-casting***, where the reinforcements are particles stirred into the liquid alloy. Process parameters and alloys are to be adjusted to avoid reaction with particles.

*In **deposition processes***, droplets of molten metal are sprayed together with the reinforcing phase and collected on a substrate where the metal solidification is completed. This technique has the main advantage that the matrix microstructure exhibits very fine grain sizes and low segregation, but has several drawbacks: the technique can only be used with discontinuous reinforcements, the costs are high, and the products are limited to the simple shapes that by obtained by extrusion, rolling or forging.

1.2 Carbon Nanotubes

Carbon nano tubes (CNTs; also known as bucky tubes) are allotropes of carbon with a cylindrical nanostructure. Nano tubes have been constructed with length-to-diameter ratio of up to 132,000,000:1(Wang, 2009), which is significantly larger than any other material. These cylindrical carbon molecules have novel properties which make them potentially useful in many applications in nanotechnology, electronics, optics, and other fields of materials science, as well as potential uses in architectural fields. They may also have applications in the construction of body armor. They exhibit extraordinary strength and unique electrical properties, and are efficient thermal conductors .

Single-walled nano tubes are an important variety of carbon nano tube because they exhibit electric properties that are not shared by the multi-walled carbon nano tube (MWNT) variants. In particular, their band gap can vary from zero to about 2 eV and their electrical conductivity can show metallic or semiconducting behavior, whereas MWNTs are zero-gap metals. Single-walled nanotubes are the most likely candidate for miniaturizing electronics beyond the micro electromechanical scale currently used in electronics. The most basic building block of these systems is the electric wire, and SWNTs can be excellent conductors (Mintmire, 1992).

Table 1: Chemical composition of multi wall carbon nano tubes

Components	*Contents (%)*
C	99.76
Al	0.03
Cl	0.09
S	0.12

The composition of MWCNT contains 99.76% of carbon and 0.12% of sulfur and remaining Al and Cl. The objective of the present studied those properties of CNT-Alumina nanocomposites. In this study, Carbon nanotube-alumina (CNT-Al2O3) nanocomposites have been synthesized by direct growth of CNTs on alumina by chemical vapor deposition (CVD) and the as-grown nanocomposites were densified by spark plasma sintering (SPS). The thermal properties of carbon nanotube-alumina composites have been investigated as a function of CNT content.

The research work reveals tensile fracture and thermal conductivity characterization of toughened epoxy/CNT nanocomposites. Rubber toughened epoxy/CNT nanocomposites were manufactured at different weight percents between 0 and 1% of multiwall carbon nanotube (MWNT) using a high intensity ultrasonic liquid processor with a titanium probe. Mechanical properties of manufactured dog bone samples were measured in tension and the results indicated a maximum of 23% increase in the elastic modulus at 0.6% by weight of MWNT. However, the fracture strength showed a maximum decrease of about 11% as a function of increasing MWNT loading. Scanning Electron Microscopy (SEM) images from the neat samples revealed a distinct circular pit at the top left edge of the specimen with an overall tearing deformation causing the fracture paths. Comparatively, all nanocomposite samples on an average seemed to show a prominent brittle fracture with little or no evidence of circular pit formation [2].

Georg Broza,et al., (2009) has proposed in their journal of Nanocomposites of poly(vinyl chloride) with carbon nanotubes (CNT).. The nanocomposites of PVC with multi-walled carbon nanotubes and single wall carbon nanotubes were prepared in THF solution, followed by film casting. The scanning electron microscopy allowed confirming a homogeneous distribution of the CNT's in the PVC matrix. Depending on the CNT's concentration, changes of sorption in methylene chloride as well as of the characteristic temperature of PVC transformation, determined by means of the DSC measurements, were found. The electrical measurements indicated an increase of the conductivity with growing CNT's content in the PVC matrix [3].

Anshu Sharma,et al., (2009) has proposed in their journal of Aligned CNT/Polymer nanocomposite membranes for hydrogen separation. CNT/Polymer nanocomposites have been fabricated by dispersing (0.1%) weight fraction of SWNT and MWNT in polycarbonate matrix separately using benzene as a solvent. Alignment has been performed by inducing DC electric field (500 V/cm). X-ray diffraction measurements have been performed to confirmation of SWNT, MWNT and their presence in PC matrix. Gas permeability has been found to be increased in aligned CNT/polymer nanocomposites comparison to random dispersed CNT/polymer nanocomposites [4].

The available research article deals with effects of CNT alignment on electrical conductivity and mechanical properties of SWNT/epoxy nanocomposites. In this study, the single-walled carbon nanotubes (SWNTs) filled nanocomposite SWNT/epoxy resin composite with good uniformity, dispersion and alignment of SWNTs and with different SWNTs concentrations were produced by solution casting technique. Subsequently, the semidried mixture was stretched repeatedly along one direction at a large draw-ratio of 50 for 100 times at ambient atmosphere manually to achieve a good alignment and to promote dispersion of SWNTs in the composite matrix. Composite showed higher electrical conductivities and mechanical properties such as the Young's modulus and tensile strength along the stretched direction than perpendicular to it, and the electrical property of composite rise with the increase of SWNT concentration [5].

Fawad Inam, et al., (2010) has proposed in their journal of the sintering and grain growth behavior of ceramic–carbon nanotube Nanocomposites. The sintering and grain growth behavior of alumina + 2, 3.5 and 5 wt. % carbon nanotubes (CNTs) and alumina + 2 wt. % carbon black nanocomposites prepared by Spark Plasma Sintering (SPS) were studied. The addition of CNTs to ceramics produces a large reduction in the sintering temperature required for their complete densification and a significant grain size refinement by a previously unreported mechanism. The CNTs form a strong entangled network around the grains, which constrains the normal and abnormal grain growth. An alumina/alumina + 2 wt. % CNT/alumina laminate structure was prepared to demonstrate directly the large grain-growth retardation effect of CNTs. These effects open up the possibility of using CNTs as a sintering aid to control the sintering behavior and microstructures of ceramics in bulk, laminate and functionally gradient (FGM) form [6].

3. Materials and Methods

3.1 Material Used

The metals identified for the present study are
- Aluminum [1100] (Al).
- Carbon nanotubes (MWNTs).

The components present over in melt stirring machine are Furnace, heating element, sample crucible, mechanical stirrer, argon inlet, argon outlet.

The block copolymer disperbyk-2150 was first dissolved in ethanol in a small beaker. Then MWCNTs were added to the as-prepared solution. This mixture was put at room temperature into an ultrasonic bath for 15 min. Then it was stirred for 30 min at 250 rpm. After adding Al chip the suspension was further stirred at 250 rpm inside a fume cupboard to evaporate ethanol and homogenize the mixture.

After the mixture was dried, the MWCNT coated chips were placed in a cylinder sample crucible. This crucible was placed into an oven and heated up to 650 c under an inert gas atmosphere to avoid oxidation. When the Al chips were molten, the liquid was mechanically stirred at 370rpm for 30min to further disperse MWCNTs. After stirring, the molten MWCNT/Al composite was poured into a mould. The cooled sample was machined to cylinder shaped specimens for subsequent tests. Reference sample were made using exactly the same procedure but from pure Al.

3.2 Scanning Electron Microscope

The scanning electron microscope (SEM) is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity. The Quanta series from FEI is the advanced, flexible solution for current and future diagnostics applications. Featuring three imaging modes – high-vacuum, low-vacuum and ESEMTM it accommodates the widest range of samples of any SEM system. It is engineered to provide maximum data – imaging and microanalysis – from all specimens, with or without preparation. The new 2nd generation of Quanta's feature improved image resolution and contrast thanks to a new imaging engine that also accommodates remote control.

3.3 X-RAY Diffraction

The principle of X ray diffraction.XRD is a technique that is widely used in the nano-technology. Applications range from phase identification, quantification and determination of crystallite and particle size, all on nano-scale level. We will explain typical advantages using XRD in this technology over other techniques such as the non-destructive nature, averaging of properties, proper statistics and fast measurements. Also we present Small Angle X-ray Scattering, used to determine nano-sized particles, that is now available on a standard laboratory-scale diffract meter.

Crystallite size can also cause peak broadening. The well known Scherrer equation explains peak broadening in terms of incident beam divergence which makes it possible to satisfy the Bragg condition for non-adjacent diffraction planes. Once instrument effects have been excluded, the crystallite size is easily calculated as a function of peak width (specified as the full width at half maximum peak intensity (FWHM)), peak position and wavelength

4. Result and Discussions

4.1 SEM Analysis for Al – CNT Composition

The scanning electron microscope (SEM) is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity.

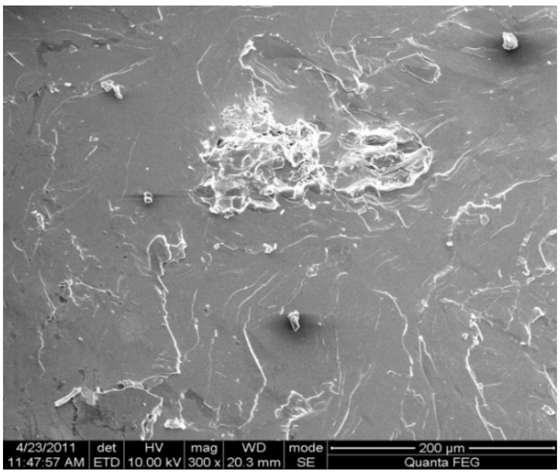

Figure 1: SEM Image for Al (1100)

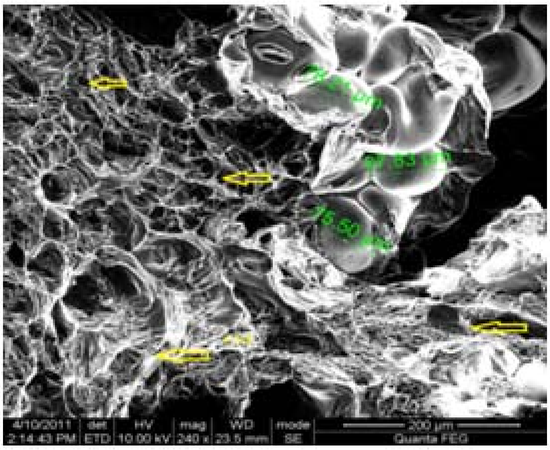

Figure 2: SEM image for Al -2%CNT

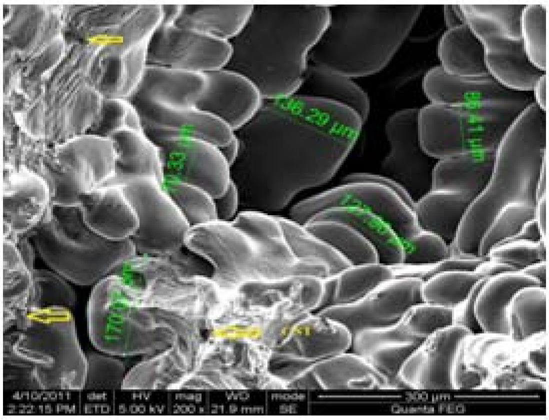

Figure 3: SEM image for Al-3%CNT

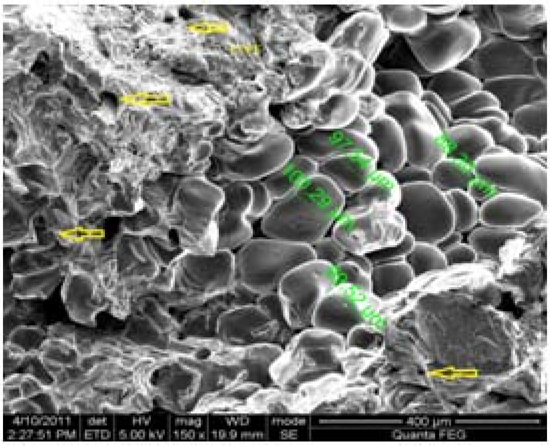

Figure 4: SEM Image for Al-4%CNT

The work of fracture (WOF) results show that pure Al has a lower fracture resistance as compared to the nano composites with up to 4 wt% CNTs added. It is due to the improved yield strength and ductility of the nanocomposites. Al reinforced with 4 wt% CNTs has a more optimum combination of strength and ductility compared to the other two nanocomposites, as it requires the maximum amount of energy to fracture. The WOF decreases with increasing addition of CNTs, a possible reason could be due to the increase in clustering effect with higher amount of CNTs which will inevitably lead to porosity.

Fig.4 Shows ductile Fracture surface analysis of Al – CNT shows the presence of individual strands of CNTs on the surface of Al. Fig 2, 3 Shows that CNTs are found to be quite well dispersed, with clusters occasionally observed.

4.2 XRD Test

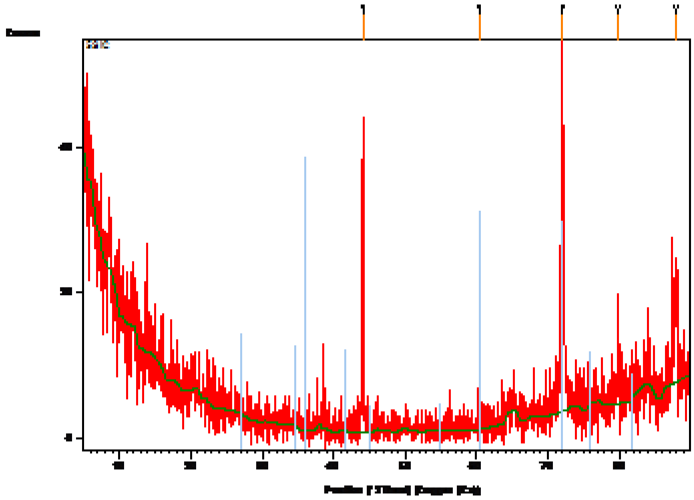

Figure 5: XRD image for Pure Al

Position (2θ)	*Height [cts]*	*FWHM Left [°2Th.]*	*d-spacing [Å]*	*Rel. Int. [%]*
44.052790	37.480220	0.240000	2.05395	84.83
60.321210	5.005113	0.180000	1.53316	11.33
72.033490	44.181960	0.480000	1.30998	100.00
79.853870	9.557991	0.360000	1.20020	21.63
88.013340	8.357759	0.300000	1.10876	18.92

Table 2: Indexed Peak Parameters for Pure Al

From table 2. The indexed peaks represents the XRD analysis of the nanocomposites without Carbon nano tube presents. The highest peak position of without CNT Nanocomposites is 44.181960 counts and the peak angle lays at 72.03° and d spacing of 1.30998.

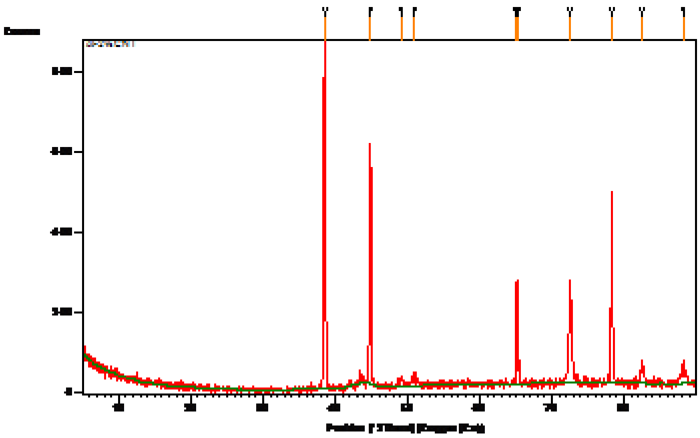

Figure 6: XRD Image for Al – 2%CNT

Position (2θ)	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
38.541320	875.350300	0.300000	2.33402	100.00
44.801870	564.713100	0.420000	2.02133	64.51
49.015940	20.914730	0.720000	1.85695	2.39
50.939190	23.639430	0.960000	1.79126	2.70
65.004350	241.668500	0.240000	1.43356	27.61

Table 3: Indexed Peak Parameters for Al – 2%CNT

From table 3, The indexed peaks represents the XRD analysis of the nanocomposites with Carbon nano tube presents. The highest peak position of with CNT Nanocomposites is 875.35030 counts and the peak angle lays at 38.54° and d spacing of 2.33402.

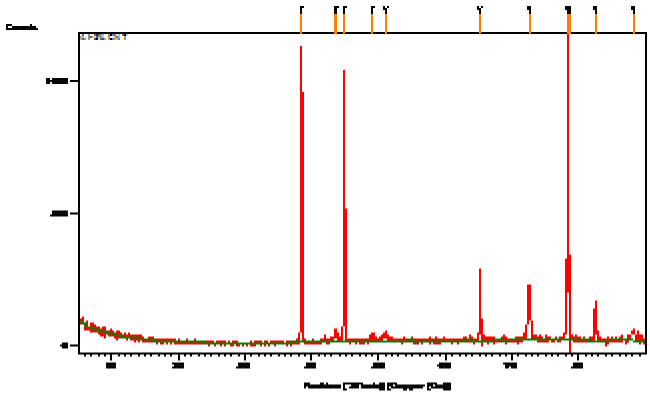

Figure 7: XRD Image for Al – 3%CNT

Position (2θ)	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
38.546970	1084.53800	0.300000	2.33369	95.50
43.584880	27.495980	0.720000	2.07491	2.42
44.794570	943.079500	0.240000	2.02164	83.05
49.079380	19.028580	0.840000	1.85470	1.68
50.966630	22.475650	0.840000	1.79036	1.98

Table 4: Indexed Peak Parameters for Al – 3%CNT

From table 4, The indexed peaks represents the XRD analysis of the nanocomposites with Carbon nano tube presents. The highest peak position of with CNT Nanocomposites is 1135.621000counts and the peak angle lays at 78.24° and d spacing of 1.22077.

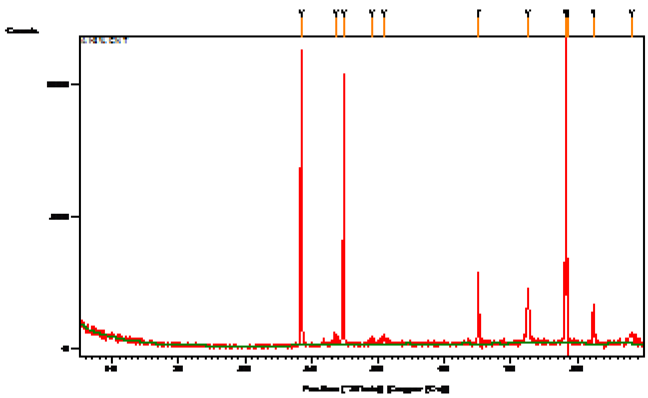

Figure 8: XRD Image for Al – 4%CNT

Position (2θ)	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
38.589320	1194.39700	0.240000	2.33123	100.00
43.618790	23.577330	0.720000	2.07337	1.97
44.831690	724.471700	0.240000	2.02005	60.66
65.158800	162.507600	0.300000	1.43054	13.61
72.546950	227.576900	0.480000	1.30197	19.05
78.286970	460.893500	0.180000	1.22025	38.59
88.201970	36.313470	0.720000	1.10687	3.04

Table 5: Indexed Peak Parameters for Al – 4%CNT

From table 5, The indexed peaks represents the XRD analysis of the nanocomposites with Carbon nano tube presents. The highest peak position of with CNT Nanocomposites is 1194.397000counts and the peak angle lays at 38.58° and d spacing of 2.33123.

4. Conclusion

In the present work reinforcement of CNT in light Aluminum using melt stirring method. Al with 2%, 3% and 4% CNT were prepared. The physical properties of composites were investigated. The Improvement of Physical properties has been compared with pure Al. The experimental study reveals following conclusions.

The result of study suggests that with increase in compositions MWNTs, with Al shows increase in Physical properties has been observed.

Scanning Electron Microscope

SEM analysis on the fractured surface of tensile specimens for the cases of composition indicates that MWNTs were still remained with no morphological change and promoted their Physical properties.

X-Ray Diffraction

The addition of MWNTs with Al, increases the impact resistance of the reinforced Al by reducing the cracks and voids in the crystal lattice which was observed in the XRD analysis.

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