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# Fabrication of Functionally Graded Carbon Nanotube-Reinforced Aluminium Matrix Laminate by Mechanical Powder Metallurgy Technique - Part I

**Udupa G*, Rao SS and Gangadharan KV**

*Department of Mechanical Engineering, National Institute of Technology, Karnataka, Surathakal, India*

## Abstract

Advances in material science have led to innovation of new materials having different properties. Functionally Graded Laminates (FGL) belong to this trend. In this work, an attempt has been made to fabricate Functionally Graded Carbon Nano Tube (CNT) reinforced Aluminium matrix laminates by an innovative powder metallurgy based cold compaction technique. The FGL composites have been prepared by incorporating CNT particles to aluminium metal matrix in different weight fractions, ranging from 0.1 to 0.5 wt%. The mechanical properties of these FGL composites have been studied in detail for assessing the effectiveness of the adopted fabrication technique. This method is an effective way to change the properties of the materials as required in different directions. The gradient layers here exhibited different microstructures and variation in hardness from one end to the other. Absence of Aluminium Carbide in the FGL composites is an important finding of this work highlighting the chemical stability of CNT in Al-matrix. An increase in the hardness by 129% was observed in the case of 0.5 wt.% of CNT in the formed laminates. Each layer demonstrated good adhesion after sintering process with no serious pores or microcracks.

**Keywords:** CNT; FGM; FGCM; FGL; Composite; Aerospace

## Introduction

CNT is an effective reinforcement material in the case of composite material developments, owing to its good physical and chemical properties [1]. Incorporation of CNT to any alloy leads to controlled changes in its properties. Layers of composite laid up one over another form laminated composite, known as Functionally Graded Laminates [2-4]. Different kinds of FGL material have been developed with variety of reinforcement and matrix materials for variety of applications like defence, airbus, space technology, etc. In the case of FGL, layered stepwise gradation formed delivers a smooth variation in property like hardness and microstructure, from one end to the other end of the material [5-7]. Aluminium alloys are widely used in various applications like aerospace, automotive as well as chemical industries due to their low density. Variety of processing routes are available to get the FGL out of which PM is the most simple and cost effective [8-9]. To the best of authors' knowledge, the current experimental data on Aluminium Multi Walled CNTs (Al-MWCNTs) are still very much limited. Hence, the aim of this paper is to develop CNT reinforced Al metal matrix FGL and compare the mechanical properties, microstructure variation, with different weight percentages (0.1,0.2,0.3,0.4,0.5) of chemically treated MWCNT, to the monolithic 99.96% pure aluminum fabricated by cold compaction [10]. Microstructure was observed using optical microscope, after these specimens were polished to the level of mirror finish. The microhardness test was carried out in a CSMTM microhardness tester using a Berkovich indenter under load control along the radial crosssection of circular samples. The indentation load was set to 3 N and the load-penetrations responses were recorded.

## Concept of FGM and FGL

In the case of conventional FGM, mismatch of the gradation in relative direction led to damage of the component. This can be avoided by incorporating techniques of laminates. In the case of FGL, gradation is achieved by forming very thin layers of laminates as shown in Figure 1. Bonding of laminates at the interface is achieved by applying proper gum or pressing under pressure [11-13]. Creation of voids and clustering of materials can be avoided through this technique. Mixing of reinforcement material is done separately for each layer of laminates, maintaining proper gradation over the direction. Various theories for the strengthening and stiffening of Al-MWCNTs FGM are also explored. Process involved here is more simple and initial expenditure is very cost effective [14,15].

## Experimental Procedure

The MWCNTs used in the present study were synthesized via chemical vapor deposition technique (Chengdu Co. Ltd, China). The MWCNTs are 20-30 nm in diameter and 10-30 μm in length. The Al powders (Acros, purity 99%, 200 mesh, Code: 300935001) were irregular in shape with a flake morphology. MWCNT-Al powder mixtures were prepared by a mixing process that involved an intensive sonication in ethanol. The solution was ultrasonicated and heated to a temperature of 550°C until most of the ethanol had evaporated [16].

Homogeneously well dispersed CNT-Al composite powders containing different weight percent CNT were prepared by a planetary ball milling process. Methanol was added as a process control agent (PCA) in order to minimize cold welding of the Al particles. The mixtures of 0.1% to 0.5% weight fraction MWCNTs and Al powders were placed in 300 mL mixing jars containing 25 stainless steel milling balls of 10 mm in diameter (giving an initial ball-to-powder weight ratio (BPR)=10:1) [17-19]. The mechanical alloying was done at 250 r/min speed for various time durations for pure Al and MWCNT-Al composites. Mixing time for mechanical alloying was chosen to be 15 h for homogeneous mixing. Pure Al was also investigated for comparison purposes. Different nano-composite compositions were

***Corresponding author:** Udupa G, Department of Mechanical Engineering, National Institute of Technology, Karnataka, Surathakal, India, Tel: 0824 247 4000; E-mail: hiudupa@gmail.com

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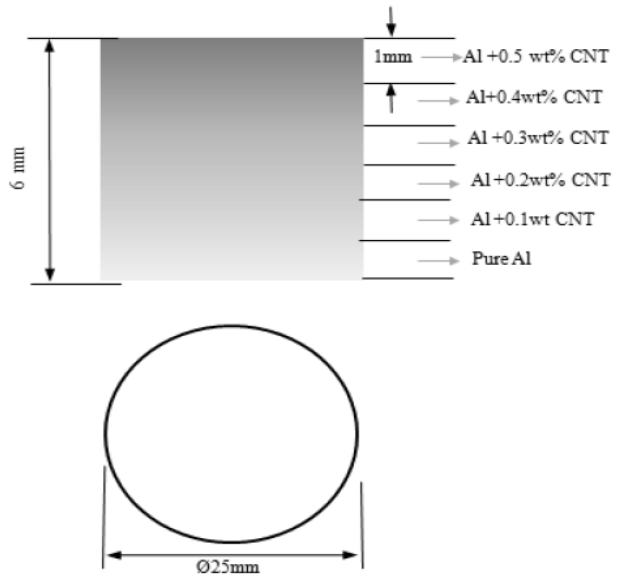


**Figure 1:** Schematic diagram of FGCM component.

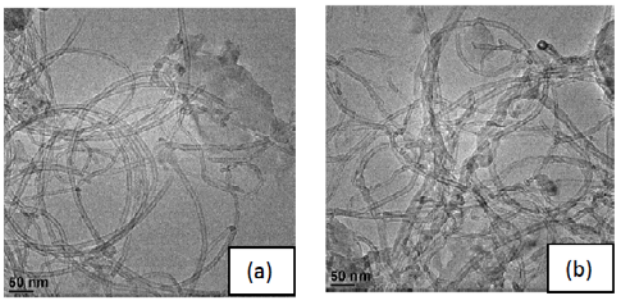


**Figure 2:** TEM images of (a) Al - 0.5wt.% CNT mixture (b) Al - 1.0wt.% CNT mixture.

studied, through MWCNTs additions of 0.1 wt.%, 0.2 wt.%, 0.3 wt.%, 0.4 wt.%, and 0.5 wt.% to the Al-matrix [20]. The ball-milled powder mixtures were compacted in a cylindrical-diameter compaction dye. Based on the relationship between green density and compaction pressure of the Al alloys, the compaction pressure of the samples was chosen to be 12 KN for pure Al. The samples were sintered in a vacuum furnace [21]. FGL was prepared by the same process but the Al-CNT composite powders were assembled in a layered structure inside a 25 mm diameter die, with compositions ranging from pure Al to composite containing 0.5 wt.% CNT, followed by cold-pressing (UTM Testing machines, 400 KN) for 60 sec under an uniaxial pressure of 220 KN. Compacted samples were sintered for 3 hrs at 550°C under vacuum with a heating rate of 50°C/min. The pure Al reference sample was not milled; it was only consolidated and sintered at the same conditions [22-24]. The starting materials were characterized by field-emission scanning electron microscopy (FE-SEM) (using a Hitachi model SE 4160) to examine the dispersion of the CNTs within the Al matrix and to characterize the composites, morphology, and size of raw materials. The microstructural observations were carried out with an optical microscope (leica Co. DM 4000 M) and a scanning electron microscope (Camscan model MV2300) equipped with energy dispersive spectrometry (EDS model Oxford). The microhardness test was carried out in a CSMTM microhardness tester using a Berkovich indenter under load control. The indentation load was set to 3 N and the load-penetrations responses were recorded. XRD studies were carried out on JEOL-JDX-8P diffractometer using copper K$\alpha$ radiation with nickel filter. 30 KV tube voltage and 20 mA tube current are chosen. A scan speed of 0.25 degree per minute was employed over an angular $2\theta$ range of 20-80°. XRD analysis has been carried out on radial cross section of prepared FGL.

## Results and Discussion

### Microstructural observation and properties test

Circular samples of 25 mm diameter were sectioned along the radial direction, anthe samples for microstructural observation and properties test were taken at different positions (x-axis) from the external circumference of the cylindrical specimen. The microscopic photograph of the cross-section of FGLC sample is shown in Figure 3, in which variation of CNT reinforcement varies from pure Al to 0.5 wt.% CNT from one end to other end of the specimen. The variation of microstructure is shown in Figure 5. As the reinforcement increased from 0.1 wt.% to 0.5 wt.% of CNT in the Aluminium matrix, the grain size continuously decreased from the value of 127 μm to 53.4 μm as shown in Table 1 which in turn results in variation of the hardness of the

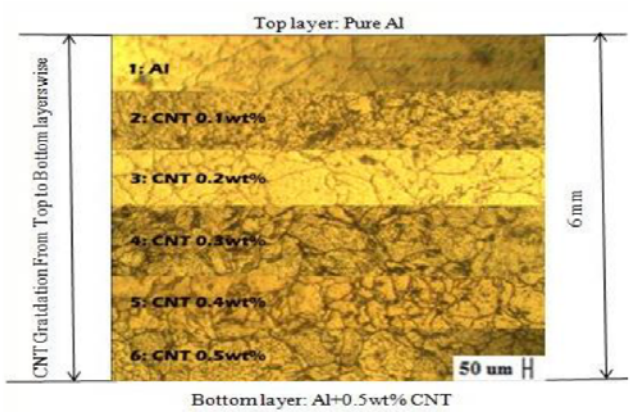


**Figure 3:** FGCM of Al with CNT reinforcement macro structure with different layers.

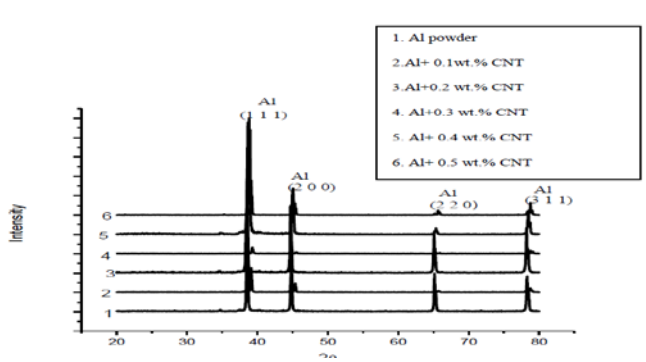


**Figure 4:** XRD patterns of Al and CNT reinforced Al composites.

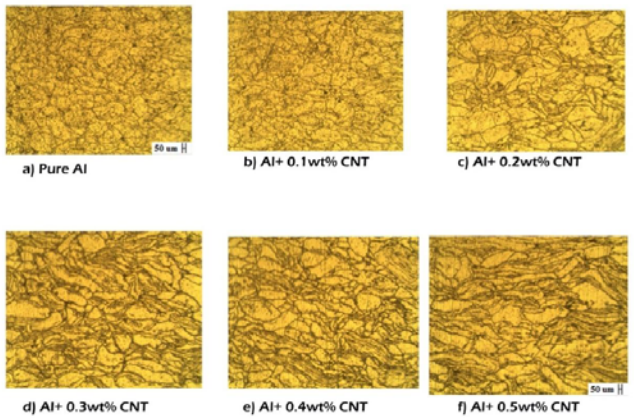


**Figure 5:** Microstructure with 40X magnification of 0.1,0.2,0.3,0.4,0.5wt.% CNT reinforcement with Al.

| CNT wt% | Theoretical density (gm/cm³) | Measured density (gm/cm³) | Relative density |
|---|---|---|---|
| 0 | 2.7 | 2.537 | 93.9 |
| 0.1 | 2.695 | 2.479 | 91.9 |
| 0.2 | 2.691 | 2.454 | 91.1 |
| 0.3 | 2.685 | 2.448 | 91.1 |
| 0.4 | 2.681 | 2.445 | 91.1 |
| 0.5 | 2.675 | 2.421 | 90.5 |

**Table 1:** Density FGL at differemt weight fraction of CNT.

specimen from one end to other end. This is observed with the Optical Microscope images, shown in Figure 4. The mechanical properties of the CNT-Al composites are mainly affected by four factors, namely: matrix strength, interface reaction, dispersion of CNTs and damage of CNTs [24-28].

The elongation of the CNT-Al composites is mainly affected by the following factors: ductility of matrix, dispersion of CNTs, and damage of CNTs. Good dispersion of CNTs can increase the elongation of the composites. As the ball-milling time is increased beyond 15 hrs, the improvement of dispersion uniformity was not obvious, but the damage of CNTs and the incorporation of impurity increases [29-31]. It led to a decrease in the ductility as revealed in the fracture toughness test. It was found that the mechanical properties of the composites are closely related to the interfacial bonding status [32,33]. Highly-magnified SEM images of the prepared specimen showed that for the composite with 0.3 to 0.4 wt.% CNT reinforcement, lots of entangled CNTs are shown in Figure 6, where as for the reinforcement of 0.5 wt.%, no entangled CNTs were found (Figure 7). This is mainly due to the uniform distribution of CNT in the matrix materials (Figure 4). Increasing the ball-milling time results in more uniform dispersion of the CNTs and tighter bonding between the CNTs and matrix Al (Figure 5). As a result, ultimate tensile strength and yield strength of the CNT-Al composites increased as the ball-milling time increased. The mechanical strength of composite interms of Ultimate tensile strength and Yield strength improved when compared with matrix material Al only in the ball milling process [34-36]. This is because the strengthening of the composites by CNTs was improved first with the good dispersion of the CNTs. However, the improvement of dispersion uniformity was not obvious after 15 h ball-milling. Stretching the ball-milling further led to more serious damage to the CNTs. Even the interface reaction between the damaged CNTs and Al matrix, results in reduced mechanical properties of the CNT-Al composites [37-39]. The densities of the compact Al and Al-MWCNT composites were measured by the Archimedes principle with deionized water as the immersion medium and the values are listed in Table 2. The density of sintered samples decreased with increasing MWCNTs weight percentage. This expected result is as per the rule of mixtures given that the density of MWCNTs is less than that of Al. Similar trend is also reported in the literature [34] for CNT reinforced MMCs. The role of porous MWCNTs in controlling the densification is an important issue with the results obtained .The microstructure of the specimen showed large good dispersion of CNT in Al. Homogeneous bridging of MWCNTs in the matrix was evident. The microstructure indicated some preferential alignment of MWCNTs into the matrix material [39,40].

The density of nanocomposite decreases with increasing mass

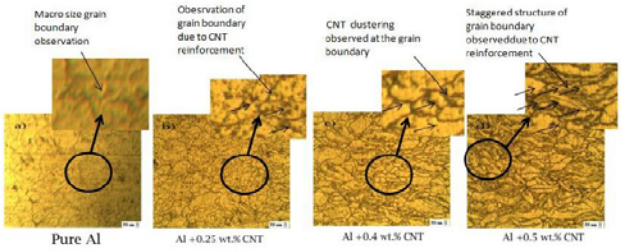


**Figure 6:** Variaton of grain boundary after CNT reinforcement.

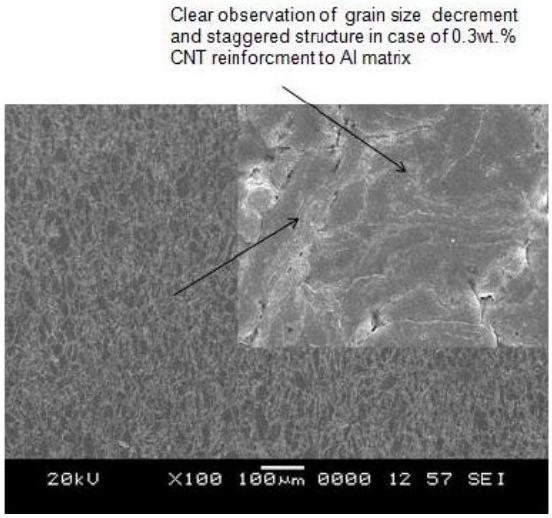


**Figure 7:** SEM imgae of Al+0.3wt% CNT.

| CNT wt % | MicrO hardness (HV) |
|---|---|
| 0 | 31 |
| 0.1 | 38 |
| 0.2 | 44 |
| 0.3 | 53 |
| 0.4 | 64 |
| 0.5 | 71 |

**Table 2:** Micro hardness values.

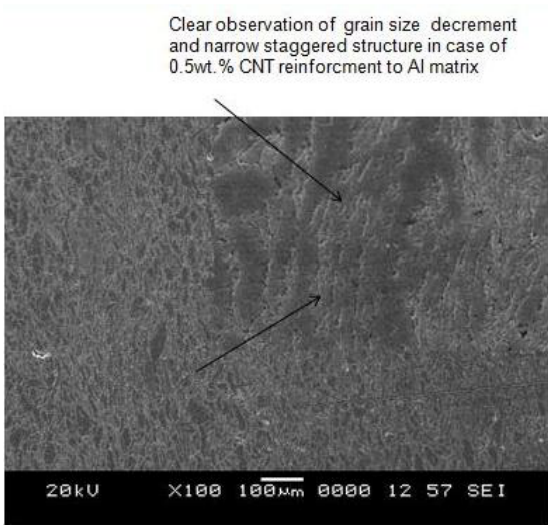


**Figure 8**: SEM imgae of Al+0.5wt% CNT.

| Sl.No | Layers Content | Grain Size No (ASTM) | Actual Grain size (µm) |
|---|---|---|---|
| 1 | Pure Al | 3 | 127 |
| 2 | Al +0.1 wt% | 3.5 | 106.8 |
| 3 | Al +0.2 wt% | 4 | 89.8 |
| 4 | Al +0.3 wt% | 4.5 | 75.5 |
| 5 | Al +0.4 wt% | 5 | 63.5 |
| 6 | Al +0.5 wt% | 5.5 | 53.4 |

**Table 3:** Grain size as per ASTM E112-10.

fraction of CNTs, and Al/CNTs nanocomposite with higher mass fractions of CNTs exhibited higher porosities by the hollow structure of CNTs. The hardness of the composite increases with increasing weight fraction of CNTs from 0 to 0.5% and decreases with more CNTs added. The introduction of CNTs in the Al matrix results in a decrease of the density of composite, whereas the hardness of composite increases by about 129% at the best reinforcement condition of 0.5 wt.% CNTs. This is due to the staggered structure of grain boundaries due to CNT clustering as observed in optical microscopic image in Figure 4. The variation of grain boundary size and structure are clearly observed in Figure 5 [41,42].

## XRD Analysis and Vickers microhardness for each layers of FGCL sample

Results obtained from XRD analysis of FGL composites are shown in Figure 8. There is no evidence of aluminum peaks shift, which indicates no solid solution formation. However, shortening of aluminum characteristic reflections is observed as the MWCNTs content increases. This phenomenon is observed in the case of 0.3 wt.% of CNTs. The shortening could be attributed to absorption effects. It is important to notice the absence of aluminium carbide ($Al_4C_3$) in the prepared FGL composites. Under similar processing conditions of the composites, have reported similar results by observing the interfacial structure between CNT and Al using TEM. This validates the chemical stability of carbon nanotube in Al matrix and MWCNT has not reacted with the base material. CNTs have also been identified as suitable reinforcement in Al based composites due to their chemical stability. Previously, it has been reported that formation of aluminium carbide ($Al_4C_3$) was found in all MWCNTs concentrations. This is mainly due to the presence of amorphous carbon in nanotubes which reacts with aluminum during the process. Formation of amorphous phase at the Al–MWCNTs interface reacts with Al due to the presence of defects along this amorphous layer (structure), and the high temperature during sintering process. The measured average micro Vickers hardness values in HV (taken from four different sampling points) for pure Al and Al-MWCNT composite layers are listed in Table 3. The results show that the hardness values of Al-MWCNTs increase as MWCNTs weight percentage increases. Quantitatively, the increase in hardness is very significant: about 129% increase in hardness was observed when MWCNTs weight percentage was 0.5 wt.% [43-46]. Figure 4 shows optical microstructures of circular sample taken at different positions from the external circumference. CNT was used a reinforcement element with Al the base material. The CNTs are dispersed uniformly in the aluminum matrix and only a few voids can be observed. As seen from Figure 4a-4f, the cross-section of the sample along the radial direction of the cylinder is divided into six reinforced zones. These zones are segregated as pure Al zone and others are CNT reinforced zones, with variation of 0.1 wt.% to 0.5 wt.% of CNT. Layer wise variation of CNT reinforcement and homogeneous distribution of MWCNTs in the Al matrix has been achieved with simple PM processing technique [47-50]. The dispersion of the CNTs in Al matrix is very important. High energy ball mill with 15 hrs of running time is more suitable to get uniform distribution of CNT in matrix. TEM images of Al-0.5 wt.% MWCNT and Al-1.0 wt.% MWCNT are shown in Figure 2. Observations revealed CNT distribution was homogeneous and improved the composite properties.

## Conclusion

Aluminium matrix reinforced with 0.1,0.2,0.3,0.4 and 0.5 wt.% of MWCNTs in layered structure were manufactured successfully using cold compaction followed by sintering through powder metallurgy techniques. The hardness attained across different layers ranged from 31 HV on one side to 71 HV on the other end, thus showing an improvement in the hardness by 129% along the direction linearly. Also, as the weight fraction of CNT increased, the relative density of the composite decreased. Grain size also decreased from the value of 127 µm to 53.4 µm as the weight fraction of CNT increased. Agglomeration of CNT on the grain boundaries resulted in the staggered structure for the Al matrix, which leads to reduction in the grain size and improvement in the hardness of the developed material. A good dispersion of CNT in Aluminium matrix was observed upto 15 hrs of ball milling time.

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