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Tensile and fracture behavior of single and dual matrix aluminum-carbon nanotube composites

Ehab Salama

THE AMERICAN UNIVERSITY IN CAIRO

SCHOOL OF SCIENCES AND ENGINEERING

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BY

Ehab Ismail Fathy Salama

B.Sc. Industrial Engineering, 2007

A thesis submitted in partial fulfillment of the requirements for the degree of

Master of Science in Mechanical Engineering

Under the supervision of:

Dr. Amal Esawi

Professor, Department of Mechanical Engineering

The American university in Cairo

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Abstract

Research on Al-CNT composites is in strong demand due to their high specific properties, and their potential applications in many advanced areas like in automotive and aerospace industries.

In the current study, tensile and fracture properties of aluminum-multiwall carbon nanotube composites (Al-CNT) were investigated. A 99.7% pure AlPOCO® aluminum powder having an average particle size of 75 microns, in addition to 70-90% pure Elicarb® multi-walled carbon nanotubes having 10-12 nm average diameter were utilized in the synthesis of single and dual matrix multiwall carbon nanotube reinforced aluminum composites. Single matrix Al-CNT composite powders with 1, 2, 2.5, 5 wt.% CNT fraction were synthesized using the high energy ball milling (HEBM) of Al and CNT powders for 1 hour at 400 rpm. Dual matrix Al-CNT composites of 1, and 2.5 wt.% CNT loadings were synthesized from 1:1 mixtures of single matrix Al-2 and 5 wt.% CNT composite powders, respectively, and unmilled aluminum powders using HEBM for 1 hour at 400 rpm.

Composite powders of different compositions were consolidated using conventional powder metallurgy processes; this included cold compaction, hot compaction, sintering, and hot extrusion processes in order to obtain high density compacts of the Al-CNT composites that are appropriate for different mechanical testing procedures. Several mechanical testing and characterization methods were applied to closely explore the mechanical properties and structural features of the Al-CNT composites. This included mechanical tension, and Elastic plane-strain fracture toughness tests as well as scanning electron microscopy, x-ray diffraction, Nanoindentation, and Raman spectroscopy.

Improvements in composite properties by tailoring the synthesis parameters as well as structural related information revealed by different testing and characterization methods are reported later in this study. It was concluded that the addition of CNT to the Aluminum matrix had a positive impact on the material strength with a corresponding loss in ductility. The study also showed that the dual matrix principle could positively retain some of the material ductility when employing the right milling conditions and mixing ratios. On the other hand, no significant influences of CNT on the elastic plane strain fracture toughness of aluminum was

observed. Instead, transition of the material fracture behavior to a less ductile manner was observed.

Acknowledgments

Actually no words could express how grateful I am at this moment to all people who contributed to this work even with a small piece of advice. First of all, I would like to deeply thank my outstanding advisor Dr. Amal Esawi for her precious contributions to this work. Actually this work wouldn't have appeared in a complete form except for her scientific and technical inputs, personal encouragement, endless effort, persistence, and continuous support during different phases of this study.

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Dedication

I sincerely dedicate this work to the soul of Egyptian revolutionaries who have spent their lives seeking justice and freedom.

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List of abbreviations

Al	**Aluminum**
BPR	**Ball to powder weight ratio**
CNT	**Carbon nanotubes**
CVD	**Chemical vapor deposition**
EDM	**Electric discharge machining**
FESEM	**Field emission scanning electron microscopy**
HIP	**Hot isostatic pressing**
HPT	**High pressure torsion**
MA	**Mechanical alloying**
MM	**Mechanical milling**
MMC	**Metal matrix composites**
MWCNT	**Multi-walled carbon nanotubes**
OM	**Optical microscopy**
PCA	**Process control agent**
Raman	**Raman spectroscopy**
SEM	**Scanning electron microscopy**
SPE	**Spark plasma extrusion**
SPS	**Spark plasma sintering**
SWCNT	**Single-walled carbon nanotubes**
TEM	**Transmission electron microscopy**
XRD	**X-ray diffraction**

Chapter One

General introduction, objectives, and scope of the thesis

Composite materials are receiving strong interest due to their outstanding combination of properties. They are engineered to serve specific applications that require superior properties to those of conventional materials. A composite material is a mixture of two or more phases that are chemically distinguishable and separated by a distinctive interface providing properties that individual constituents cannot achieve. Generations of composites have been developed over the past decades. Understanding of the structure-property relationships is very important. Different types and geometries of reinforcements are utilized to tailor the composite properties to suit diverse applications.

The discovery of CNTs has shifted the materials universe to higher performance levels. Their outstanding set of mechanical, electrical, thermal, chemical, and biological properties in addition to the high controllability of these properties have tremendous impacts in different fields of applications. Structural composites, super conductors, electron field emitters, artificial tissue engineering, electronics packaging, aerospace industries, bio-sensors, DNA sequencing, atomic field microscopy, hydrogen storage, and many more areas are greatly benefiting from the exceptional properties of CNTs. Further information about the fabrication methods and structural characteristics of CNTs are available in Appendix A.

Structural composites utilizing CNT reinforcements have been intensively investigated. Although not as heavily studied as CNT-reinforced polymer composites, CNT-reinforced aluminum composites are receiving increasing attention due to their light weight, high specific properties, ease of processing and versatility of applications. Several processing techniques are being developed to overcome the processing difficulties of Al-CNT composites.

Powder metallurgy is a powder fabrication method involving consolidation of metallic particles using the appropriate pressure and temperature combinations. The technique is very useful in producing nonporous products having properties equivalent to the fully dense original material. It has high potential of producing net shape and near-net shape components with high densities. Several powder processes are employing the powder metallurgy technique such as: die pressing, hot extrusion, spark plasma sintering (SPS), spark plasma extrusion (SPE), high pressure torsion (HPT), and hot isostatic pressing (HIP). More detailed

information about powder metallurgy and its related processing techniques are available in Appendix A.

Mechanical alloying (MA) as defined by (Suryanarayana, 2001) is a solid state powder synthesis technique involving continuous welding, fracturing, and re-welding of powder particles in a high energy ball mill. Considering the variety of methods and equipment designed to serve the MA concept, the process has a huge potential to synthesize a large variety of alloys and compounds starting from blended elemental powders. Non equilibrium compositions could be synthesized by using the right constituent proportions. High fractions of reinforcements could be used in composite synthesis. The MA technique has been reported to effectively disperse CNTs within the aluminum matrix. The MA technique also provides a severe refinement effect that makes it possible to synthesize nano-structured materials (Suryanarayana, 2001). More details about the mechanical alloying technique are given in Appendix A.

Recent investigations on Al-CNT composites have reported improvements in the composite mechanical properties in terms of strength. However, this is typically associated with a loss in material ductility that poses a critical limitation on their applications. The current study is a continuation of previous work conducted within the same research team on the synthesis and mechanical properties of CNT-reinforced aluminum composites. This study aims to provide improved processing procedures and parameters that maximize the advantage from the outstanding properties of CNTs. The current work focuses on the successful dispersion of CNTs within the aluminum matrix using the mechanical milling process. Tackling the loss of ductility issue is attempted and the dual matrix structural design is investigated.

The current study has three main objectives:

1. Provide a processing roadmap for synthesizing Al-CNT composites of enhanced mechanical and structural properties using the mechanical milling technique. This is achieved by examining the effect of an improved set of processing conditions for the synthesis of Al-CNT composites on the resulting structural and mechanical properties of the composite.

2. Investigate the effectiveness of the dual matrix composite structural design on retaining some of the Al-CNT composite ductility while as much as possible keeping its high strength, e.g. to reach optimum strength and ductility tradeoffs.

3. Investigate the potential of CNTs as a toughening material for aluminum based composites processed by the mechanical milling process.

Chapter two of this thesis "Literature review" covers the main literature about synthesis and characterization of Al-CNT composites. This is presented in terms of conventional as well as more elaborate synthesis techniques and the achieved enhancements in mechanical properties of the composites. An analysis of the Al-CNT strengthening mechanisms and interfacial interactions is also presented in Chapter Two. Chapter Three "Materials and Experimental procedure" covers the details of the processing, characterization, and testing techniques involved in the current investigation. Chapter Four of this thesis "Results and discussions" presents the results obtained from the current investigation for both single and dual matrix Al-CNT composite. This includes their mechanical properties as well as the outputs of different characterization techniques involved. Chapter Six "Conclusions" presents the conclusions drawn from the current study of Al-CNT composites followed by recommendations for future progress in that field. Additional background information pertaining to the mechanical milling process, CNTs, fracture toughness measurement and all characterization techniques utilized in the current study are presented in Appendix A.

Chapter Two

Literature review

2.1. Synthesis of Al- CNT composites

Since the discovery of CNTs in 1991, a window to new technological areas has been opened. CNTs exhibited a set of extraordinary mechanical, electrical, thermal, optical, and catalytic properties which created their high potential of application in many fields of science and technology. These outstanding properties in fact are a reflection of the near-perfect nano-sized tubular structure of CNTs. For instance CNTs exhibit very high Young's modulus of 600 to 1100 GPa and very high tensile strength of 35 to 110 GPa which have centered them in the focus of material scientists.(Bakshi and Agarwal, 2011)

One of the major applications of CNTs was in the reinforcement of composite materials which led to extensions in the conventional performance limits of materials.(Cha et al., 2005) Researchers have realized the outstanding advantage of utilizing CNTs in reinforcing polymeric materials, which have then extended to metallic and ceramic materials. It was then that when principal investigators became concerned with the development of suitable processing methods that would result in a uniform distribution of CNTs in different metallic or ceramic matrices. The Superior mechanical properties of CNTs, in terms of strength and stiffness, as well as their extraordinary nano-scale tubular structure have stimulated their use as reinforcements in structural materials. Aluminum gathered special attention as a possible matrix material due to its light weight (e.g. high specific properties) and high process-ability. Several processing techniques were investigated ranging from conventional melt processing and powder metallurgy methods to advanced molecular level mixing techniques. Several powder metallurgy consolidation methods were investigated starting from conventional powder compaction, extrusion, and rolling to the most advanced spark plasma sintering (SPS), spark plasma extrusion (SPE), and high pressure torsion (HPT) processes.

Several published articles have highlighted the critical issues that limit the advantage from the superior mechanical properties of CNTs in composites.(Agarwal et al., 2011, Esawi and Morsi, 2007, Esawi et al., 2009, Esawi et al., 2010, Esawi et al., 2011) The first challenge is the deagglomeration and uniform distribution of individual CNTs within the matrix material while retaining their near-perfect structure. The second challenge is concerned with the

orientation of CNTs within the composite structure and their effect on the resulting mechanical properties. The third is concerned with the interfacial bonding between CNTs and the matrix material.(Bakshi and Agarwal, 2011) Poor interfacial bonding between CNTs and matrix material has reported.(Choi et al., 2009)

2.1.1. Effect of CNT morphology and diameter on the synthesis and properties of Al-CNT composites

(Esawi et al., 2011) have studied the effect of CNT morphology and diameter on the processing and properties of Al-CNT composites. The authors demonstrated a comparison between the influences of two different morphologies and diameters of CNTs on the processing and properties of Al-CNT composites. The authors employed large diameter CNTs with straight, and stiff morphologies against 3.5 times-smaller diameter CNTs having bent, and entangled morphologies. A powder metallurgy based synthesis technique was employed in this study. The authors utilized a relatively harsh mechanical milling process in the dispersion of CNTs within the aluminum matrix. This was followed by a cold compaction and hot extrusion processes reaching a final dense composite part that is suitable for consecutive testing and characterization methods. The authors indicated that CNTs morphology and diameter significantly affect both of the processing and properties of Al-CNT composites.

Small diameter bent and entangled CNTs are found difficult to disperse in the structure especially with high CNT fractions due to their large tendency of agglomeration. They are found to exhibit high interfacial contact areas with the matrix which boost carbides formation at low CNT fractions. They were also reported to minimize cold welding between aluminum matrix particles during milling and to be highly susceptible to interactions with the formed aluminum carbides, nano-size oxide particles, and carbon nano-rods existing in the matrix structure.

On the contrary, large diameter straight and stiff CNTs were easier to disperse even with high CNT fractions without significant carbides intervention. They are found to retain their tubular structures intact after processing which give rise to enhancements in the composite mechanical properties. The authors illustrated that variation in the mechanical properties of Al-CNT composites incorporating the same volume fraction of these CNT is a direct consequence for their morphological and diameter dissimilarities.

2.1.2. Conventional Powder metallurgical methods

(Choi et al., 2008) investigated the effectiveness of CNTs as reinforcements for the aluminum matrix, this included initial washing out of amorphous-carbon particles from the CNT charge using a mixture of acids, followed by synthesis of pure aluminum and Al-4 vol.% CNT composites using conventional mechanical milling, powder compaction, and hot extrusion processes. The authors reported an enhancement in composite strength and Young's modulus of the composite. The authors reported a modulus of 104 GPa for Al-4 vol.% CNT compared to 70 GPa for processed aluminum and attributed this enhancement to the combined effect of reinforcement, that were uniformly dispersed and aligned as observed by TEM images, and the structural refinement associated with processing. They also reported a good match with the results obtained by the rule of mixtures of discontinuous fiber composites.

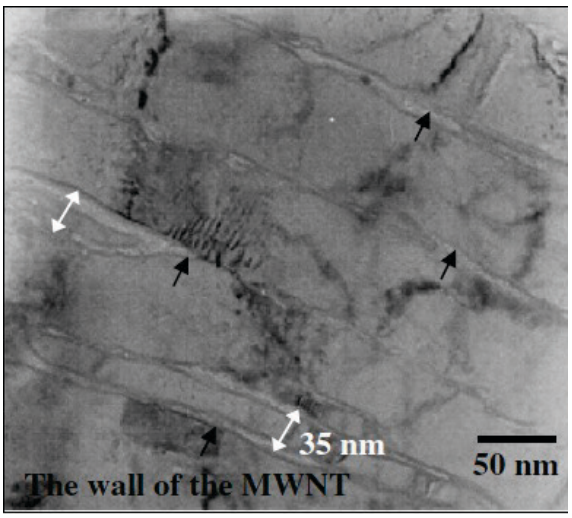

Figure 1 TEM images of the hot extruded Al-4 vol.% CNT showing individually dispersed CNTs (arrows).(Choi et al., 2008)

2.1.3. Synthesis of Al-CNT composites using the mechanical milling process

(Choi et al., 2009) utilized the mechanical milling technique to successfully disperse individual CNTs within aluminum powders. The technique was reported to considerably eliminate the formation of interfacial compounds in the bulk scale when combined with the appropriate powder consolidation method and thus maintain the structural order of the CNTs. (Choi et al., 2009) produced four different compositions of the Al-CNT composite (1.5, 3, 4.5, and 6 vol.% CNT) in addition to pure aluminum to investigate the impact on the mechanical

properties of the composite. Their procedure included an initial purification treatment of the CNTs using mixtures of acids to eliminate impurities and amorphous carbon particles. They used an attritor type ball mill rotating at 500 rpm for a milling period of 6 hours at which CNTs were completely embedded within the aluminum particles as confirmed by the SEM images. Stainless steel vial and balls of 5 mm in diameter were used to produce the composite powders. They used a ball-to-powder weight ratio of 15:1 and 1 wt.% stearic acid as the process control agent in an inert atmosphere. They provided control over the milling process temperature using a circulating water system. A hot rolling consolidation process was used to produce a fully dense composite. Canned milled composite powders were hot-rolled at 480 C for 27 rolling passes with an overall reduction in area of 12%. Thin sheet specimens were obtained on which several testing and characterization methods were performed.

(Choi et al., 2009) demonstrated that mechanical milling by the conditions mentioned above causes infiltration of some aluminum atoms to partially fill the CNTs embedded in the structure changing their aspect ratios, i.e. enlarging tube diameters and decreasing lengths. (Choi et al., 2009) were able to prove this using HETEM images and Raman spectrographs where the increase in inter-atomic distances of carbon atoms in the CNT structures causes changes in vibrational excitation energies of CNT molecules resulting in substantial peak shift on the Raman spectra, as shown in Figure 2, relative to the initial CNTs structures. The hot rolling process was extremely advantageous in orienting CNTs within the material parallel to the rolling direction and hence maximized the structure load bearing capacity.

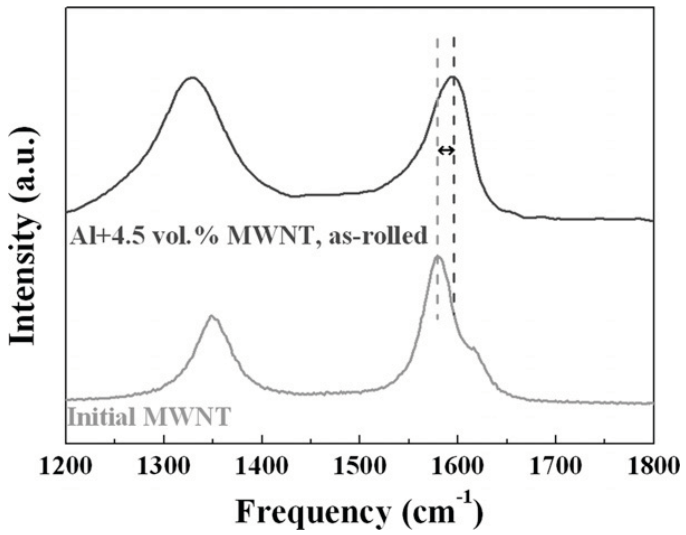

Figure 2 Raman spectra of starting CNTs and Al-4.5 vol.% CNT composite showing peak shifts resulting from CNT structural changes.(Choi et al., 2009)

The mechanical properties reported by (Choi et al., 2009) confirmed the effectiveness of the involved processing technique in strengthening and toughening of Al-CNT composites. They reported a yield strength and fracture toughness of 610 MPa and 60.79 $MPa\sqrt{m}$ for the Al-4.5 vol.% CNT condition, compared to 262 MPa and 33.22 $MPa\sqrt{m}$ for pure aluminum prepared with the same procedure, respectively.

(Morsi et al., 2010c) reported that conventional mechanical milling processes strain hardens milled composite powders which would affect further processing steps and would reflect on the final properties

2.1.1. Synthesis of Al-CNT composites by the roll bonding technique

(Lahiri et al., 2009) have reported the fabrication of Al-CNTs composites of 2, 7.5, 9.5 vol.% CNTs by the roll bonding method. The authors have reported the effectiveness of the technique in homogenous dispersion of CNTs within the Al matrix for CNT concentrations up to 2 vol.%, also showed that CNT agglomeration took place at higher volume fractions of 7.5 9.5 vol.%. The authors have reported a dual strengthening role of CNTs that was connected to their uniform dispersion in the composite. They have indicated that uniformly dispersed CNTs in the composite at low CNT fractions of 2 vol.% improve the homogenous behavior of the composite structure which reflects higher resistance to plastic deformation at low stresses, e.g. higher modulus of elasticity. They have also figured out that CNT precipitates at higher

volume fractions have lessen this uniform response of the structure, which led to a lower modulus and higher tensile strength. The authors reported an ultimate increase in composite modulus up to 59% at 2 vol. CNTs and an increase in tensile strength up to 250% in 9.5 vol.% CNTs.(Lahiri et al., 2009)

2.1.2. Spark plasma Sintering/Extrusion synthesis of Al-CNT composites

The SPS technique has gathered a lot of researcher's attention due to its advantages of high densification, fast and uniform processing. A number of studies were concerned with the spark plasma sintering techniques featuring Al-CNT composites and others used it combined with a complementary densification process.(Morsi et al., 2010a, Morsi et al., 2010c, Morsi et al., 2010b)

(Kwon et al., 2010) have adopted the use of a combination of spark plasma sintering and hot extrusion processes in producing Al-CNT composites from their raw constituents. They reported high strength enhancement compared to starting aluminum while maintaining the composite ductility. They attributed such outstanding scheme of properties enhancement to the presence of CNTs at the boundaries and to the alignment of CNTs during the extrusion process as well as to the effective load transfer between the CNTs and the matrix caused by the formation of Aluminum carbides at the interface.

(Kwon et al., 2010) used CNTs having average diameter and length of 20 nm and 30 μm, respectively, in addition to 99.5% pure aluminum powders having average particle size of 15 μm. They have adopted a precursor dispersion method (Kwon et al., 2009) to mix Al and CNT in the form of dry powders. This included mixing of 1 vol.% CNT and 99 vol.% aluminum powders with natural rubber in a solution of benzene and consequently heating the mixture for 2 hours at 500 C in an argon atmosphere. This was believed to achieve uniform dispersion of CNTs on aluminum powders surfaces after treatment which was then confirmed by SEM investigations. They also reported that the aluminum particles retained their average particle size and spherical morphologies while CNTs were observed a little bit shorter than the starting length.

(Kwon et al., 2010) exposed the Al-CNT mixture after treatment to spark plasma sintering process forming a bulk compact using 50 MPa pressure and variable temperatures of 480, 500,

560, and 600 C with a constant heating rate and holding time of 40 C/min and 20 minutes, respectively. A successive hot extrusion process at 400C and extrusion ratio of 20:1 was employed to further enhance densification and CNT alignment. They reported some important findings for sintered and extruded Al-CNT composite parts at this stage and this included that aligned CNT reinforcements uniformly reside at the grain boundaries of the structure and minute interfacial defects resulting from chemical reactions between aluminum and defective CNTs were observed, also that grain growth of composite particles was neither observed in sintered parts nor in the extruded parts. They have attributed this to the pinning effect associated with high heating rate (40 C/min) during both processes.

(Kwon et al., 2010) reported a good enhancement in mechanical properties compared to bulk aluminum processed using the same procedure, such that a maximum tensile strength of 207.5 MPa and maximum elongation of 21% were achieved by the Al- 1vol.% CNT composite sintered at 480 C and that for processed aluminum were 52 MPa and 19.5%, respectively. The authors attributed the enhancement in strength to the formation of Al_4C_3 at the interface that ameliorate stress transfer between the matrix and the reinforcements. They presented SEM fractographs shown in Figure 3 where a highly dimpled structure (the mark of high ductility) is observable, bridging and few broken CNTs were also distinguished.

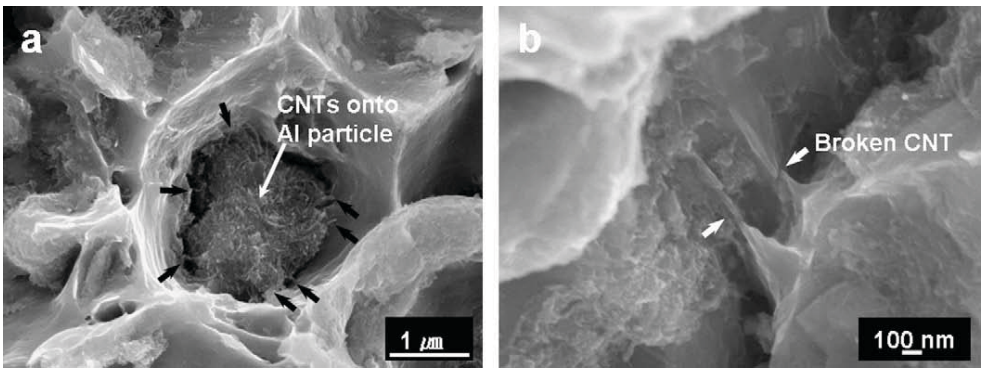

Figure 3 FESEM fractographs of the extruded Al-1 vol.% CNT composites after tension test showing (a)low magnification where black arrows are indicative of CNT bridging, and (b) higher magnification where the white arrows refer to broken CNT.(Kwon et al., 2010)

In a similar investigation, (Kwon et al., 2009) used exactly the same precursor treatment, the spark plasma sintering, and the hot extrusion procedure to produce Al-5 vol.% CNT composites. The author reported an enhancement in Ultimate strength and Vickers macro

hardness of Al-CNT composite compared to those of pure aluminum processed the same way. The reported properties were 198 MPa and 52 HV for the ultimate strength and macro hardness properties, respectively, for the Al-5 vol.% CNT composite, and 110 MPa and 22 HV for pure aluminum.

(Morsi et al., 2010c) used the mechanical milling process as a good dispersion method of CNTs into aluminum matrix in conjunction with spark plasma extrusion process to produce Al-2.5 wt.% CNT, they demonstrated the advantage of using SPE compared to earlier SPS as allowing the production of powder based materials with extended-geometries and bulk deformation under the influence of direct electric current. The authors used aluminum powders with 45 μm average particle size and CNTs having 30-50 nm in average diameter and 10-20 μm in length. They utilized milling conditions of BPR 5:1, methanol PCA 1.5 wt.%, milling time of 60min and 90min for the pure aluminum and the Al-2.5wt.% CNT compositions, respectively. A cold compaction step was involved followed by the SPE process at a temperature of 433 C and ram speeds of 5.6 and 6.3 mm/s for pure aluminum and Al-2.5 wt.% CNT compositions, respectively. The authors reported successful extrusion of the pure and composite conditions with no evidence of carbides formation. They also demonstrated the effect of CNT in providing internal lubrication during SPE. The reported mechanical properties showed an enhancement in Al-2.5 wt.% CNT composite compressive strength and Vickers hardness of 415.3 MPa and 99.1 HV compared to those of pure aluminum prepared the same way of 377.2 MPa and 74.7 HV, respectively. The authors have attributed these enhancements to the dual effects of reinforcement and reduced grain size of aluminum matrix due to processing.

In the same year of 2010, (Morsi et al., 2010a) published the results of their investigation on two compositions of Al-CNT composites (Al-2.5 wt.% CNT & Al-5 wt.% CNT processed by a combination of mechanical milling and the spark plasma sintering technique. Generally they used the same starting materials and milling parameters as mentioned in the previous paragraph, in addition to those of the SPS process which was conducted using a pressure of 250 MPa at $380\,^{o}C$ and $324\,^{o}C$ for the 2.5 wt.% CNT and the 5 wt.% CNT conditions, respectively. The authors reported the effectiveness of the mechanical milling process in dispersion of CNTs within the aluminum matrix and they highlighted that the PCA has a critical effect on the size and morphology of milled composite powders in conjunction with the

percentage CNT loading and milling time, such that high CNT loadings favors the fracturing phase of the milling process and reduces particle size, incidentally increased milling time would consume the added PCA and increase the particle size back. The reported micro hardness for Al- 2.5 wt.% CNT and Al-5 wt.% CNT were 91.3 HV and 107.3 HV, respectively, which is close to that reported for same Al- 2.5 wt.% CNT produced by the spark plasma extrusion process.

2.1.3. High pressure torsion synthesis of Al-CNT composites

(Tokunaga et al., 2008) have utilized the high pressure torsion (HPT) process to synthesize Al-CNT composites as shown in Figure 4. The authors used a mixture of high purity aluminum powder and 5 wt.% SWCNTs of 1-2 nm in diameter in the synthesize process. The HPT process showed high potential in producing high density disks of Al-CNT composites with no introduced heating. Refinement in grain size was observed down to 500 nm for HPT processed aluminum compared to 1200 nm for bulk aluminum. The existence of CNTs has shown to magnify the refinement effect down to 100 nm. Significant increases in composite hardness up to 76 HV and in the composite strength to more than 200 MPa were recorded. The hardness of the composite is found higher at the compact disk edges and decreases toward the disk center. The authors attributed this to an odd distribution of strains induced by the HPT process such that a steady state condition has not yet reached. (Tokunaga et al., 2008)

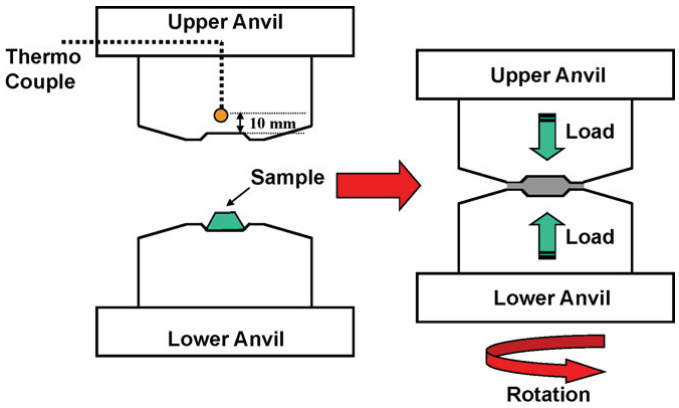

Figure 4 Schematic representation of the HPT process.(Tokunaga et al., 2008)

2.1.4. Emphasis on dual matrix Al-CNT composites

(Morsi et al., 2010b, Esawi et al., 2012) have investigated both single and dual matrix Al-CNT composites. (Morsi et al., 2010b) were interested to identify differences in structural performance between single matrix and dual matrix composites. The authors demonstrated

the structural differences between single and dual matrix composites in terms of the geometry and dispersion of the reinforcements. They used a controlled atmosphere ball milling process to produce Al-2.5 wt.% CNT composite powders. Cold compaction and spark plasma extrusion processes were employed in consolidation of milled Al, single-, and dual-matrix composite powders. The ram speed of the SPE process was set to 6.3 mm/s and extrusion temperature was varied to identify the optimum extrusion temperature that minimizes carbide formation. The authors proved the effectiveness of the SPE process in producing Al-CNT composites both single and dual matrix with no significant formation of carbides. The authors also demonstrated that dual matrix Al-CNT composites can be spark plasma extruded with lower pressure requirements than that of single matrix composites and they attributed this to the added soft material. The dual matrix Al-CNT composite was reported to have lower compressive strength than their single matrix counterparts and also lower than milled Al conditions. On the other hand, dual matrix composites showed an enhanced ductile behavior.(Morsi et al., 2010b)

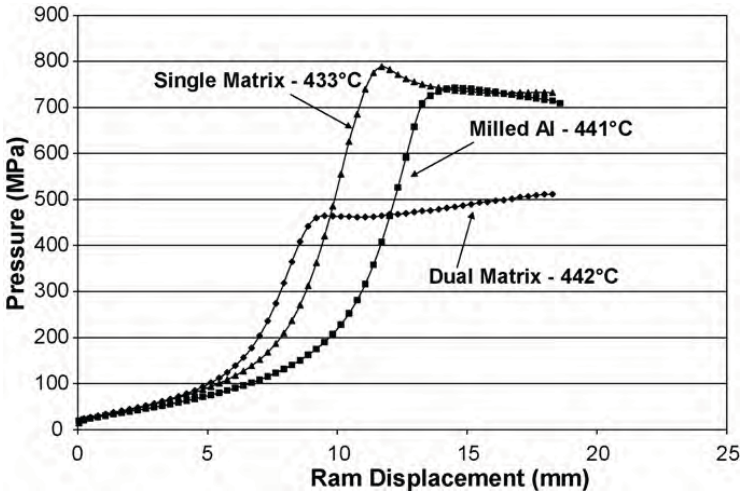

Figure 5 Pressure versus ram displacement of the SPE process showing the dual matrix effect in lowering pressure requirements.(Morsi et al., 2010b)

2.2. Strengthening in Al-CNT composites

The interactions between CNTs and the aluminum matrix have been intensively investigated in several investigations (George et al., 2005, Choi et al., 2008, Kwon et al., 2010, Choi et al., 2009, Bakshi and Agarwal, 2011, Ci et al., 2006, Pérez-Bustamante et al., 2009, Balani et al., 2007) aiming to maximize the potential of CNTs as composite reinforcements.

Possible strengthening mechanisms of Al-CNT composites have been examined and correlated to the measured experimental results of the composite properties.(George et al., 2005)

(Bakshi and Agarwal, 2011) have analyzed the factors affecting strengthening of Al-CNT composites and the possible efforts to maximize the advantage form the superior properties of CNTs. The authors demonstrated three main aspects to affect strengthening of the Al-CNTs composites. These are the dispersion of CNTs within the matrix material, the structural deformation during processing, and the interfacial interactions between the matrix and the reinforcements. The authors have showed that different composite processing techniques greatly affect the structure of the reinforcements, for example, they reported that processes such as ball milling, and hot extrusion break up CNTs into smaller length sections minimizing their aspect ratios, and hence reducing their strengthening capacity. The authors have claimed that the intense deformation accompanying processing, despite of its effect in damaging the CNT structure, it is useful in disintegration of CNT clusters and in the preferential alignment of individual CNTs within the structure. They also concluded that the higher the deformation the stronger the contact between the Aluminum and CNTs and the better the load transfer to CNTs. The authors demonstrated that Al-CNT interactions are necessary for a successful load sharing between the matrix and the reinforcements and that the characteristics of the interfacial region between the Al matrix and CNT reinforcements are significantly important. They also pointed out that the strengthening effect observed in Al-CNT composites still far below what have expected based on the superior properties of CNTs and they attributed this to the damage to CNTs tubular structures during processing as well weak interfacial bonding between the matrix and the reinforcements. The authors demonstrated that the rule of mixture predictions of the composite strength and modulus is efficient up to 2 vol.% CNTs.(Bakshi and Agarwal, 2011)

(George et al., 2005) have addressed three relevant strengthening mechanisms namely: Thermal mismatch, Orowan looping, and Shear lag model, that could be operable for the case of Al-CNT composites and asserted their theoretical predictions of the composite properties, in terms of strength and modulus of elasticity, with those experimentally measured. The authors utilized the mechanical milling process with soft conditions in producing Al-0.5 & 2 vol.% CNTs and Al-1 & 2 vol.% SWCNTs composites in the form of fine powders which were then consolidated using conventional powder metallurgy processes into suitable testing

components. The authors highlighted their speculations concerning each of the strengthening mechanisms as follows:

Thermal mismatch

(George et al., 2005) claimed strengthening in Al-CNT composites to be resulting from punching of internal dislocations at the Al/CNT interface leading to work hardening of the matrix. This was attributed to a significant mismatch between matrix and reinforcements coefficients of thermal expansion, that for aluminum is $23.6x10^{-6}K^{-1}$ and for CNTs: $10^{-6}K^{-1}$. The authors figured out that the density of generated dislocations at the interface is dependent on the surface area of the reinforcements. Since CNTs have very high surface areas thus higher density of dislocation would be achieved which leads to increased strengthening. The reported prediction of incremental strength based on this model is given in Equation 1 and Equation 2.(George et al., 2005)

$$\Delta\sigma = \alpha \times \mu \times \rho^{(1/2)} \times b$$

Equation 1

and

$$\rho = 10 \times A \times \varepsilon / \left(b \times t \left(1 - A \right) \right)$$

Equation 2

Where α is a constant equals 1.25, μ is the modulus of rigidity of matrix material (for Al is $2.64 \times 10^{10}\ N/m^2$), ρ is the dislocation density, and b is the Burgers vector.

Orowan Looping

(George et al., 2005) attributed strengthening to the hindering of internal dislocations motion by the CNTs existing within the composite structure. CNTs are believed to enforce a back stress which prevents further dislocation migration in the material and causes the material to strengthen. This strengthening mechanism showed high significance in metal matrix composites where reinforcements are very fine and inter-particle spacing is small. The incremental shear strength that could be predicted by this strengthening mechanism could be calculated from Equation 3.

$$\Delta\tau = K \times \mu b A^{(1/2)} / r \times \ln(2r / r_o)$$

Equation 3

Where K is a constant depending on type of dislocations (0.093 for edge dislocations and 0.14 for screw dislocations), r_o is the core radius of dislocation (3.5×10^{-9}), b is the Burgers vector, μ is the modulus of rigidity of matrix material (for Al is $2.64\times10^{10}\ N/m^2$, and A is the volume equivalent radius for the reinforcement (1.593×10^{-7} for CNTs and 7.087×10^{-9} for SWCNT)

The Shear lag model

This model assigned the load transfer job between the matrix and the reinforcement in Al-CNT composites to the interfacial shear stress. This in fact better incorporates the high properties of CNTs as a source for strengthening. The model assumes good wetting properties between the matrix and the reinforcement for effective stress transfer while this is not the case in Al-CNT composites in which wetting is limited because of the significant difference between the surface tension properties of both constituents (for Al is 865 mN/m and that reported for CNTs range from 100-200 mN/m). The Young's modulus of the composite based on the shear lag prediction model can be calculated through Equation 4 and Equation 5.

$$E_c = A \times E_f \left(1 - \tanh(ns)/(ns)\right) + (1 - A) \times E_m$$

Equation 4

and

$$n = \left(2E_m / \left(E_f \times (1 + \gamma_m) \times \ln(1/A)\right)\right)^{(1/2)}$$

Equation 5

Where E_f is the Young's modulus of the reinforcement material, E_m is the Young's modulus of the matrix material, s is the aspect ratio of the reinforcement ($: 100$ for MWCNTs and up to $: 1000$ for SWCNTs), A is the reinforcement volume fraction, and γ_m is the Poisson's ratio of the matrix material (0.3 for Al)

According to (George et al., 2005), an enhancement in material strength and Young's modulus took place relative to those of processed aluminum. The correlation between the measured properties and those predicted using different mechanisms mentioned above showed that, for the Al-CNT composites, the shear lag and the Orowan looping models best match the experimentally measured Young's modulus and yield strength of the composite, respectively. As for the Al-SWCNT composites the authors reported a relatively good correlation between the measured Young's modulus and the one predicted using the shear lag model. The authors also implied that the observed strengthening might also be due to synergistic effect of a combination of the mechanisms mentioned above.(George et al., 2005)

2.3. Mechanical properties of Al-CNT composites

(Esawi et al., 2010) have investigated the effect of CNT loading on the mechanical behavior of Al-CNT composites. the authors have used 99.7% pure- 200 mesh Al powder in addition to 95% pure CNTs having an average diameter in the range of 30-50 nm, an average length of 10-20 µm, and an aspect ratio of approximately 375. Five composite compositions were produced to examine their impacts on the mechanical properties of the composites. 0.5, 1, 2, and 5 wt.% CNT in addition to pure aluminum were investigated. The processing involved dispersion of CNTs with the respective composition into aluminum matrix using the high energy ball milling process. Milled composite powders were consolidated using conventional compaction and hot extrusion processes. Mechanical properties were then examined using different testing methods. The authors reported that increasing CNT contents in the matrix significantly increases the material strength up to 2 wt.% CNT at which more than 50% enhancement is achieved. Further increase in CNT content, produced slight enhancement and was considered not effective. The authors reported a similar behavior for the Young's modulus of the composite showing a maximum improvement of more than 23% at a CNT wt.% fraction of 2wt.% further increase in CNT content over 2wt.% have led to slight deterioration in the Young's modulus. The authors have reported that the milling process parameters in conjunction with the CNT greatly influence the mechanical properties of the composite.

(Bakshi and Agarwal, 2011) have provided a piece of the art survey of the published mechanical properties of Al-CNT composites as shown Table 1. The tabulated records show characteristic enhancements in the composite properties due to the dispersion of CNT in the structure. Poor dispersion of CNTs in the matrix and the formation of excessive amounts of

aluminum carbides in addition to the deterioration in composite ductility comprise the most critical limitations of Al-CNT composites.

The mechanical properties of Al-CNT composites reported by different studies as shown in Table 1 display good enhancement in composite strength and modulus up to 620 MPa and 110 GPa, respectively, however, the advantage from the superior mechanical properties of CNTs was not fully incorporated. Nonetheless, the reported composites were suffering severe reduction in ductility down to 2.2 % due to both the reinforcements and the processing techniques involved. These have motivated the current research to magnify the advantage from the high CNT properties using better processing parameters compared to those previously reported as well as to manipulate the composites ductility issue.