



## Carbon Nanotubes Reinforced Aluminum Matrix Composites - A Review of Processing Techniques

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**Abstract** – Carbon nanotube reinforced aluminium matrix composites (Al-CNTs) have been widely used in aerospace and automotive industries where high quality and strength is required. The enhanced mechanical properties of Al-CNTs are closely related to processing technique due to challenges within production of these composite materials. In the current review, solid state processing techniques used for synthesizing Al-CNTs have been reviewed to provide an insight into the features and capabilities of each technique regarding the incorporation of CNT reinforcements. To conclude, the mechanical performance of Al-CNT composites is mainly decided by the capability of each technique in the dispersion of CNTs within the aluminum matrix.

**Keywords:** Metal Matrix Composites (MMC), Aluminum matrix composites (AMCs); Carbon nanotubes (CNTs); CNT reinforced aluminum composites (Al-CNTs)

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### Introduction

Light metal aluminum and its alloys are one of the most widely used materials in aerospace and automotive industries. The main properties contributing to its widespread use are lightness, low toxicity, good formability, electrical and heat conductivity and corrosion resistance. As a potential candidate for structural applications, these materials require high specific strength and stiffness. Then, further strengthening of aluminum is required (Toozandehjani, 2014; Toozandehjani et al., 2015).

One way to strengthen the aluminum is to manufacture a composite by the addition of one or more secondary phases or so called reinforcements. In fact, the second phase composition, shape and distribution can be tailored to meet specific requirements. Recently, carbon nanotubes (CNTs) have been used by many researchers to reinforce aluminium metal matrix composites (AMCs) in order to improve mechanical, thermal and even electrical properties of AMCs (Salvetat et al., 1999).

Only a small amount of these nano-particles can improve mechanical properties of various metal matrices including aluminum (Salvetat et al., 1999). Reinforcing of light AMCs using CNTs contributes to the fabrication of composites with high strength, toughness, hardness and creep resistance which can be used for different purposes in different industries (Salvetat et al., 1999; Munirozzaman & Winey, 2006; Thostenson et al., 2001).

A variety of processing methods are available to synthesize AMCs, however, full potential of these processes in the field of Al-CNTs is not fully discovered. Therefore, starting with a brief understanding of the aluminum and CNTs, a review over researches conducted in the field of processing of Al-CNTs is provided. The main purpose of the current review is to address the features and capability of solid state processing method regarding to the reported challenges of the CNT reinforcements within the aluminum matrix.

### **Composite materials**

A composite refers to a poly phase material showing a noteworthy extent of the properties of both components which implies better combination of properties than the individual components used (Samal & Bal, 2008; Balasubramaniam, 2007). It typically consists of a dispersed reinforcement phase embedded in a hard matrix which transfers the stress to the soft reinforcement phase in order to facilitate manufacturing of the composite material. Every single constituent material in a composite plays a role determining the final properties of the composite. Therefore, utilizing of particular properties of the constituent of composite is a good way to meet desired properties of a composite (Samal & Bal, 2008; Balasubramaniam, 2007; Chawla, 2009; Miracle, 2005).

The composite materials are generally classified according to their reinforcement, matrix and processing route. In the case of reinforcements, they can be categorized based on their nature (polymer, ceramic, metal), Shape and the orientation. According to the matrix of the composite, they are categorized as polymer matrix composites (PMCs), metal matrix composites (MMCs) and ceramic matrix composites (CMCs). Few valuable reviews and books have provided detailed discussion on the various composite materials and their properties (Koo, 2006; Hull & Clyne, 1996; Chawla & Chawla, 2005; W. Kaczmar et al., 2000).

#### *Metal Matrix Composites (MMCs)*

MMCs are distinct from other type of composite materials in many ways. These distinctions mainly arise from the inherent differences among metals, polymers and ceramics and to some extent from nature of the reinforcements. MMC systems are generally designated according to the matrix or metal alloy designation, type and volume fraction of reinforcement. However, these designations do not cover consolidation process, subsequent heat treatment or specific fiber orientations. Different pure metals and alloy systems have been utilized in MMCs as a matrix such as aluminum, copper, iron (steels), magnesium, nickel, and titanium. These metal matrices are available in the various forms including castings, wrought and powder materials which employed during the manufacturing process of MMCs. The choice of matrix in MMCs is mainly influenced by consideration of the demanded properties of composite in the particular application. Another important consideration in the selection

of the matrix is the potential reinforcement/matrix reactions which influence the performance of composites (Clyne, T. W., & Withers, 1995; He et al., 2008; Tjong, 2007; Kainer, 2006; Everett & Arsenault, 1991). The main objectives of reinforcing metal matrices are weight reduction, improvement of corrosion resistance, fatigue resistance and creep resistance at higher temperatures, strength properties coupled with thermal characteristics (Clyne, T. W., & Withers, 1995; He et al., 2008).

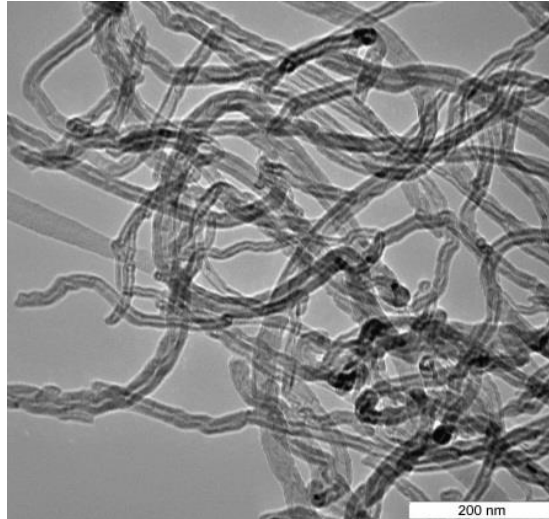
### **Aluminum matrix composites (AMCs)**

AMCs have been widely used in structural, automotive and aerospace industries as a light composite material. Due the fact that the desired strength and mechanical properties cannot be achieved by using of aluminum itself or its alloys therefore, they have been replaced by AMCs in these industries. In fact, reinforcing of aluminum alloys gives the possibility of production of parts with optimized mechanical, thermal and physical properties and an accurate chemical composition. Besides, new manufacturing methods have provided more opportunities of production of parts with more enhanced mechanical and physical properties. These enhancements through reinforcing help the aluminum industry to prevail over new markets for their products.

Pure aluminum and different commercially available aluminum alloys are used as matrix for AMCs. A wide variety of reinforcement particulates such as SiC, B<sub>4</sub>C, Al<sub>2</sub>O<sub>3</sub>, AlN, TiC, TiO<sub>2</sub>, TiB<sub>2</sub> and graphite have been used to reinforce aluminum matrix in different type, shape, and sizes. The most widely used reinforcement particulates are silicon carbide (SiC), aluminium oxide (Al<sub>2</sub>O<sub>3</sub>) and graphite in the form of particles or whiskers due to high availability, low cost and overall good properties (Surappa, 2003; Kevorkijan, 1999; Rohatgi, 1993; Lloyd, 1994; Sinclair & Gregson, 1997; Miracle & Donaldson, 2001). As a matter of fact, addition of reinforcements to soft aluminum alloys can significantly improve the properties of the composite particularly when particles are in nano-scale. Recently, carbon nanotubes (CNTs) have also attracting a great interest in the scientific community as a new kind of reinforcement material for the production of novel MMCs.

### *Carbon nanotubes (CNTs)*

Carbon nanotube (CNT) is one of the various allotropes of carbon built up out of graphene sheets. Graphene is a one atom dense lab of carbon bound together in a network of repeating hexagons through sp<sup>2</sup> near planar hybridization. While stacked graphene sheets result in graphite, a graphene sheet wrapped into a sphere is called a Buckyball and the same sheet rolled up to form a cylinder is a carbon nanotube (Geim & Philip, 2008; Popov, 2004).



*Figure 1:* TEM micrograph of a MWCNTs (Ostovan et al., 2015).

CNTs are hollow cylinders with diameters in the order of nanometers but with a much larger length. Some properties of the carbon nanotubes are low density, vast particular surface zone and surface penetrability originating from its tubular shape (Figure. 1). The strength and stiffness of CNTs are very high due to the strong covalent  $sp^2$  bonds of the carbon atoms. In fact, thermal, electrical and mechanical properties are strongly dependent on carbon atomic architecture and  $sp^2$  bonding (Geim & Philip, 2008).

While CNTs have been observed since the 1950s (Monthieux, & Vladimir, 2006), it is only with the landmark paper by Iijima (1991) that CNTs has held the interest of worldwide researchers. CNTs have been an ideal candidate as a new reinforcement for different matrices (Geim & Philip, 2008; Popov, 2004; Robertson, 2004). It has been found that CNTs improve both strength and stiffness of MMC and PMCs, while CNTs increases the fracture toughness and thermal conductivity of CMCs. This enhancement is attributed to the exceptional mechanical properties of CNTs including strength ( $\sim 63$  GPa), stiffness ( $\sim 1$  TPa) and thermal conductivity of up to 3000 W/m K (Popov, 2000; Treacy & Ebbesen, 1996; Yu et al., 2000; Yu et al., 2000; Demczyk et al., 2002; Lau et al., 2004). CNTs can be found in two different types; single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs) which are simply compose of concentric SWCNTs. MWCNTs can be considered as a SWCNT with the same diameter as the MWCNT outer diameters (Lau et al., 2004 ). It must be noted that the presence of primary flaws in CNTs can significantly lower mechanical strength. Carbon nanotube defects are classified as functionalization (or chemical) defects, stone-wales defects, rehybridization defects due to deformation and incomplete bonding defects, mainly as vacancies (Yang et al., 2007; Tunvir et al., 2008).

In the particular interest of authors, CNTs have been used to reinforce metal metric like copper, magnesium, nickel, titanium and aluminum to stretch the performance boundary of monolithic materials during past decades (Maqbool et al., 2013; Sun et al., 2013; Karim et al., 2013; Esawi & Morsi, 2007; Silvestre et al., 2014; Li et al., 2013). The improved properties of CNT reinforced composites compared to monolithic materials have been reported even at lower weight percentage of

CNTs in the matrix. However, few challenges have been found during fabrication of AMCs which will be discussed later.

### **Processing of CNT reinforced aluminum matrix composites (Al-CNTs)**

Like other types of composite materials, properties of Al-CNTs are associated to the fabrication method and its corresponding parameters. Fabrication methods of MMCs are being typically divided into liquid or solid state manufacturing processes. Liquid state processes involve the melting of the matrix wherein the interfacial bonding between the metal matrix and reinforcement is favored leading to a stronger bond between them. However, the presence of a liquid also raises the issues of possible formation of interfacial reaction layers, reinforcement wettability and reinforcement agglomeration. For instance, liquid processing of Al-CNT composites is problematic due to CNT segregation and lack of infiltration resulting from their poor wettability (George et al., 2005). Due to the difference between surface tensions of matrix and CNT, the wettability is difficult to achieve which considered being an important issue in metal-CNT systems (So et al., 2011). These potential problems are added to the disadvantages of traditional metal liquid processing such as segregation and porosities.

The solid state manufacturing processes mainly involve powder metallurgy (PM) route. PM process has showed greater efficiency than other solid state process with respect to the economic aspects. In PM processing method, products are formed from composite constituent in the form of powder without passing through the liquid phase. Therefore, production of parts with complex shapes, high quality and precision is practical. While being typically more complex and expensive, PM process can prevent the problems associated with liquid state techniques (Schey, 2007; Tang et al., 2002; Dowson, 1990; Fayed & Otten, 1984). According to Suryanarayana and Al-Aqeeli (2013), synthesizing the AMCs is more feasible with solid-state processing methods.

However, solid state processing methods are more feasible, but there are few challenges during fabrication of Al-CNTs which influence the final properties of composites. The major challenges mainly arise from the blending part. The main factor in the synthesizing Al-CNTs is to obtain homogeneous and uniform dispersion of CNTs within the matrix powder and effective retention of CNTs after the consolidation process (Zhou et al., 2007). As a matter of fact, CNTs due to their high aspect ratio and high van der Waals forces (which hold the CNTs particle together) between carbon layers, inherently tend to be agglomerated. Larger specific surface of these nano-sized materials compared to micron-sized materials, increases the effects of van der Waals forces and makes CNTs entangled with other tubes (Suryanarayana, 2001). Agglomeration and clustering of CNTs must be avoided because CNT agglomerates reduce the poor mechanical properties of final composite (Suryanarayana & Al-Aqeeli, 2013; Kim et al., 2008; Suryanarayana, 2001). CNT agglomerates result in a greater plastic strain in their vicinity and promote the formation of voids, thus reducing the ductility, strength, toughness and resistance to fracture as well as fatigue properties. According to the literature, the percolation threshold of the CNTs has been reported to be less than 1% (Zhou et al., 2007; Li et al., 2007; Borah, 2010). Further CNT volume fraction causes CNTs to interconnect together which retard the uniform dispersion of CNTs within the matrix, thereby affecting the

mechanical properties of the composite (Borah, 2010). Further, an effective interface and wettability between CNTs and aluminum matrix should be also controlled to guaranty the successful load transfer in AMCs. A favored interfacial bonding between the matrix and CNTs is required to obtain optimum mechanical properties. Improved wettability results in better interfacial bonding between the matrix and CNTs (Suryanarayana & Al-Aqeeli, 2013; Esawi & Morsi, 2007). Additionally, the damage of CNTs at elevated temperature and in highly reactive environment which CNT experience during the fabrication of Al-CNTs should be taken into account.

Due to the above mentioned challenges, achieving a defect-free and non-clustered microstructure of Al-CNTs is a very difficult task. Therefore, different available solid state processing methods for synthesizing Al-CNTs have reviewed in the following paragraphs.

#### *Mechanical alloying*

Mechanical alloying (MA) is a solid-state powder processing wherein powder mixtures are cold welded, fractured, and rewelded repeatedly in a high-energy ball mill until alloying happens and a homogeneous alloy mixture is obtained. The MA technique has unique advantages including ease of obtaining the microstructure, introducing high volume fraction of the reinforcement and ease of consolidation of the milled powder to full density. The process variables are milling speed and time, ball-to-powder weight ratio (BPR), milling atmosphere and the grinding medium as well as process control agent (PCA), which are required to be optimized during milling to achieve demanded microstructure (Suryanarayana, 2001; Suryanarayana, 2004, Suryanarayana & Al-Aqeeli, 2013).

As a well-known process method, mechanical alloying through ball milling is an effective technique to disperse CNTs in a metal matrix. In a series of works by Esawi and Morsi (Esawi & Morsi, 2007; Morsi & Esawi, 2007; Morsi et al., 2010; Esawi et al., 2010; Esawi et al., 2009) and George (2005), MWCNTs have been incorporated into aluminum matrix using MA technique. In order to understand the effect of MA on the dispersion of MWCNTs, different weight percentage of MWCNTs (0.5-5 wt. %) and aluminum matrix have been subjected to ball milling. MWCNTs are more favorable for reinforcing of aluminum alloys because of exceptional mechanical properties however, lower than SWCNTs, and ease of synthesizing (Suryanarayana & Al-Aqeeli, 2013). A good dispersion of MWCNTs within the aluminum matrix has been reported. It has been also reported that ball milled powder not much suffer from agglomeration however, milled powders get strain hardened within milling which result in difficulties during consolidation process (Esawi et al., 2009). Al-Aqeeli et al. (2012) have reported that prior ultra-sonication of CNTs retards agglomeration of CNTs during milling particularly at higher CNT contents. It is also found that prior sonication provides better dispersion of CNTs even at shorter milling time. Esawi et al. (2007) have ball milled 2 wt.% MWCNT with pure aluminum as a matrix inside a planetary machine for various times. The whole procedure has been done in argon atmosphere. Figure 2 shows the morphology of mechanically alloyed Al-2wt.% CNT at different milling time (Esawi et al., 2007).

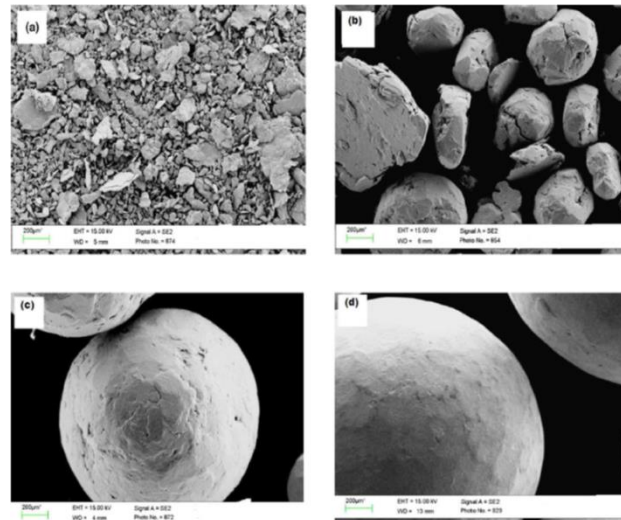


Figure 2: SEM image of mechanically alloyed 2 wt% CNT in Al powder after (a) 30 minutes, (b) 6 hours, (c) 18 hours and (d) 48 hours (Esawi et al., 2007).

The variation of particle size with milling time can be observed as a result of the dynamic balance between cold welding and fracturing that take place during milling (Esawi et al., 2007; Morsi & Esawi, 2007). Indeed, at the preliminary stages of milling, ductile particles are flattened due to the influence of ball milling through the initial impact of the grinding balls. Afterwards, the particles begin to weld together as milling time increases. Finally, the particles size is stabilized representative of steady state. The mechanism of composite formation via milling is comprehensively explained in (Suryanarayana, 2001; Suryanarayana & Al-Aqeeli, 2013; Fogagnolo et al., 2003). Figure 3 reveals incorporation of individual CNTs into the matrix contributing to the good dispersion of CNTs within the matrix (Esawi et al., 2007).

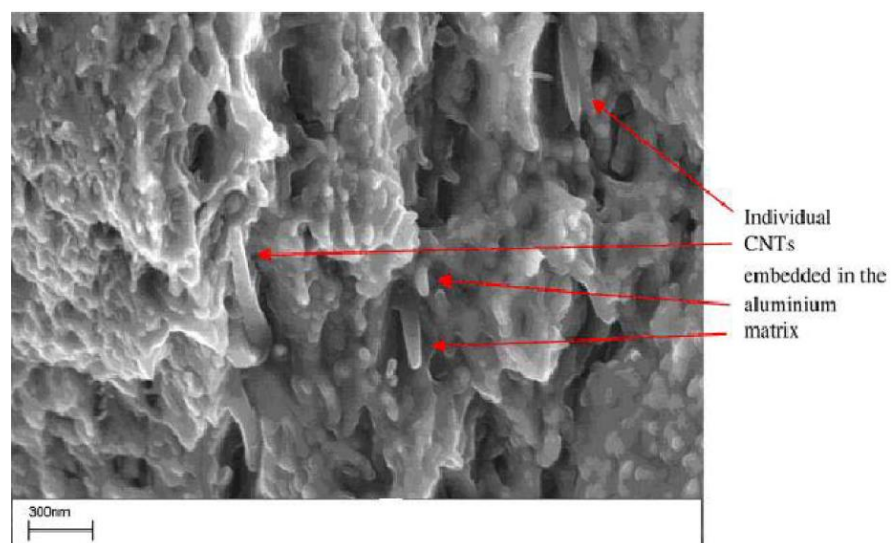


Figure 3: FESEM image of 2 wt.% CNT/Al mechanically alloyed for 48 hours (Esawi et al., 2007).

Perez-Bustamante et al. (2010) and Perez-Bustamante et al. (2008) have found the higher mechanical properties: strength and hardness as result of increasing milling time itself and CNT contents in pure Al–MWCNT composite as also reported by Jafari et al. (2012). For example, young's modulus is found to be increased about 25% by addition of 2 vol.% CNT in a Al2024–MWCNT nanocomposite. Hence, it has been found that increasing the MWCNT content to 3 vol.% leads the nanocomposite to be highly brittle (Jafari et al., 2012). Further, the effect of MWCNTs on the strength properties is more dominant at longer milling time as reported by Perez-Bustamante et al. (2008).

Morsi and Esawi (2007) have showed that process control agent (PCA) facilitate production of finer particles particularly at lower amount of CNT used. PCA reduces the effect of cold welding of soft powder particles as well as minimize powders agglomeration. During milling, PCAs decompose and adsorb on the surfaces of particles interacting with powders which result in the formation of compounds that inserted into the powder mixture as inclusions and/or dispersoids (Suryanarayana & Al-Aqeeli, 2013). During milling of aluminum powders, oxides ( $\gamma$ -Al<sub>2</sub>O<sub>3</sub>) and carbides (Al<sub>4</sub>C<sub>3</sub>) introduces to the powder particles (Suryanarayana & Al-Aqeeli, 2013; Singer et al., 1980).

Poirier et al. (2009) and Jafari et al. (2012) have reported the interaction between the aluminum matrix and CNTs and formation of Al<sub>4</sub>C<sub>3</sub> phase during milling. The formation of Al<sub>4</sub>C<sub>3</sub> phase is due to the chemical interaction between Al and C atoms resulting from the damages of CNTs during the milling. Poirier et al. (2009) have found that Al<sub>4</sub>C<sub>3</sub> phase forms during processing route of Al–MWCNT composite including milling, cold compaction and sintering at 630 °C. While, Jafari et al. (2012) reported the presence of Al<sub>4</sub>C<sub>3</sub> phase after consolidation by hot pressing of Al2024-MWCNT at 500 °C. However, Perez-Bustamante et al. (2010) and Perez-Bustamante et al. (2008) have not reported any interaction between Al and CNTs and presence of any reaction product.

The consolidation of milled composite powders is usually carried out by conventional solid state consolidation including cold isostatic pressing (CIP) which follows by sintering. One of the major problems related to cold pressing is the lower density of the bulk material. Besides, it is more serious when dealing with milling of hard powders and composite powders with higher reinforcement contents. It must be noted that, holding the powders at elevated sintering temperatures for extended duration resulting in grain growth and consequently loss of mechanical properties. Conventional hot isostatic pressing (HIP) also have been used in order to improve density of final composite through simultaneous applying high temperature and pressure (Long et al., 2014; Poletti et al., 2008). HIP is diffusion bonding of powder under hydrostatic pressure which enables concurrent compaction and sintering of the powders at elevated temperatures. It is reported that hot pressed composite powders also show high level of porosity and cracks in regions with high concentration of reinforcements like clusters (Singh et al., 2011).

Therefore, a final consolidation or secondary process is required to obtain a completely dense structure (Derakhshandeh Haghighi et al., 2012). Such processes are hot extrusion, rolling, forging, spark plasma extrusion (SPE) or alternative methods like severe plastic deformations processes (SPD). These secondary processes introduce large strain to the composite powders and enable more uniform



dispersion of reinforcements (Tan & Zhang, 1998; Casati & Vedani, 2014). Extrusion is the commonly used process for consolidation of AMCs wherein powders are consolidated under pressure of an extrusion press. Extrusion processes are hot extrusion (at elevated temperature) and cold extrusion (at temperatures below re-crystallization temperature of the metal). One important feature in powder processing route of AMCs is the presence of fine oxide particles which act as a dispersion strengthening agent influencing the properties of the matrix particularly at high temperatures (Singh et al., 2011; Derakhshandeh Haghighi et al., 2012). These oxide layers break under shear stresses produced by hot extrusion. It is also noted that extrusion reduces the cross section of composite and more forces is required to extrude the composite (Derakhshandeh Haghighi et al., 2012). In the context of Al-CNTs, they have been successfully synthesized using hot extrusion process and improved strength and hardness of these composites is reported.

#### *Cold isostatic pressing followed by hot extrusion*

Hot extrusion is usually followed by cold isostatic pressing process or direct hot extrusion of powders. Deng, Wang, Zhang, et al., (2007) have processed different weight percentages of CNTs with Al2024 aluminum alloy. However, Deng, Zhang, Ma, et al., (2007) and Deng, Zhang, Wang, Lin, et al., (2007) have previously reported improved strength properties of Al2024-CNTs (1 wt.%) composites synthesized by mechanical milling and cold isotactic pressing. They cold pressed powders under 300 MPa pressure and then subjected to the hot extrusion at 470 °C. It was seen that by increasing the CNT contents up to 1 wt.%, the relative density and hardness as well as strength of the composites increases as shown in Figure 4 and Figure 5 (Deng, Wang, Zhang, et al., 2007). It is due to the fact that the addition of small amounts of CNTs provides a uniform dispersion CNTs within the aluminum matrix which leads to the enhancement of density due to the filling up the voids. However, further increase of CNT content to 2 wt.%, reduces the hardness and the relative density. The increase in the tensile strength and Young's modulus of hot extruded mechanically alloyed pure Al-MWCNT (4 vol.%) is also reported by Choi et al., (2008). They observed tightly bonded MWCNTs which are separately dispersed and uniaxially aligned grains in consolidated composite rods due to high temperature and pressure of extrusion. Higher CNT contents affect the densification process resulting in lower densities. The higher CNT contents contributes to agglomeration of CNTs in which the bonding between the CNTs becomes weak, leading to a loss in mechanical properties as shown in Figure 5 (Deng, Wang, Zhang, et al., 2007). Strength properties of Al-CNTs can be affected by the energy absorption of CNTs (Zhan et al. 2003) and the bonding between the CNTs and Al matrix (Tham et al., 2001). Improvement in strength of the composite is because of transferring of load from the Al matrix to the CNTs. It was reported that after the tensile tests some amount of carbon nanotubes were exuded of the aluminum. But the size of the CNTs pulled out for 1 wt.% of Al2024-CNT composite was much shorter in length compared to that of 2 wt.% of CNT. This indicates a stronger interfacial bonding in the composites containing 1 wt.% CNT compared to those containing 2 wt.%.

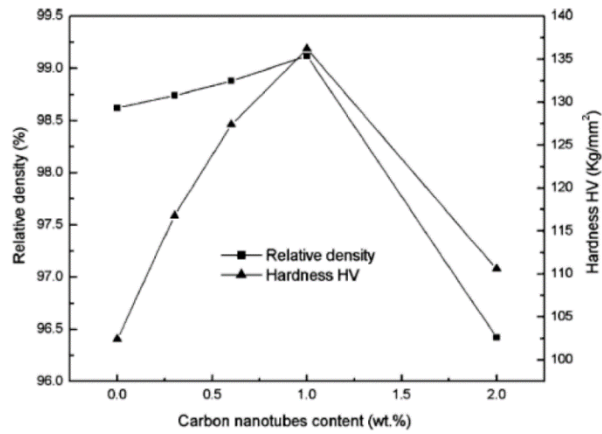


Figure 4: Hardness and density of different weigh percentages of CNT/2024 Al composites (Deng, Wang, Zhang, et al., 2007).

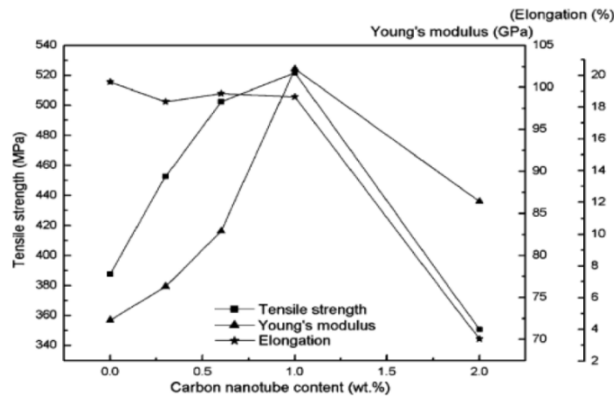


Figure 5: Effect of CNTs addition on tensile strength, Young's modulus and elongation of an Al matrix (Deng, Wang, Zhang, et al., 2007).

#### Ball milling followed by hot extrusion

George et al., (2005) fabricated Al-SWCNT and Al-MWCNT composites by combination of powder metallurgy and ball milling. They consolidated milled powders by sintering followed by hot extrusion at 560 °C. They tried to provide a comparison between the theoretically and experimental mechanical properties as shown in Table 1 and 2 (George et al., 2005). They reported improved mechanical properties of both composites compared to those of the pure aluminum as a result of CNT addition and hot extrusion. The similar result in the improvement of strength properties (hardness and yield strength) in hot extruded Al2024 containing 0–2.5 wt.% carbon-coated Ag nanoparticles has been also reported by Carreno-Gallardo et al. (2009). According to George et al., (2005), the strengthening is correlated to orowan looping mechanism. In Orowan looping mechanism, it is believed that the movement of the dislocations is restricted by the carbon nanotubes thereby producing back stress which further prevents migration of the dislocations resulting in an increase in the yield stress (George et al., 2005). Generally, the mechanical properties of a composite are decided by three different strengthening mechanisms namely Orowan looping, shear lag and thermal mismatch between the matrix and the reinforcement (Casati & Vedani, 2014; Zhang & Chen, 2006; Zhang & Chen, 2008; Sanaty-Zadeh, 2012). However, no single model is satisfactorily capable of explaining the mechanical

properties then strengthening of the composites determined by the synergistic effect of three above mentioned mechanisms. For example, the shear lag model is able to explain the increase in Young's modulus while Orowan looping and thermal mismatch justifies the increase in yield strength (George et al., 2005).

*Table 1:* Effect of MWCNT volume on mechanical properties of Al-MWCNT composites (George et al., 2005).

MWCNT vol%	Shear Lag Young's Modulus (MPa)	Experimental Young's modulus (MPa)	Experimental Yield Strength (MPa)	Ultimate Tensile strength (MPa)
<b>0.5</b>	74.305	78.1	86	134
<b>0.5 + K<sub>2</sub>ZrF<sub>6</sub></b>	74.305	75.20	93	150
<b>2</b>	84.376	85.85	99	138

*Table 2:* Effect of SWCNT volume on mechanical properties of Al-MWCNT composites (George et al., 2005).

SWCNT vol%	Shear Lag Young's Modulus (MPa)	Experimental Young's modulus (MPa)	Experimental Yield Strength (MPa)	Ultimate Tensile strength (MPa)
<b>0.5</b>	79.167	70	79.8	141
<b>0.5 + K<sub>2</sub>ZrF<sub>6</sub></b>	79.167	93.7	98.7	181
<b>2</b>	88.355	79.3	90.8	134

Kwon & Leparoux (2012) successfully extruded ball milled Al-MWCNT composites using hot extrusion processes. They found alignment of well dispersed CNTs with the extrusion direction. They have also reported the significant increase in strength properties of pure Al matrix by addition of just 1 vol.% of CNTs. They also detected Al<sub>4</sub>C<sub>3</sub> phases at the grain boundaries which chemically linked interface between the matrix and the CNTs. The synergistic effects of well dispersed CNTs and Al<sub>4</sub>C<sub>3</sub> phases result in enhanced strength properties.

Choi et al., (2010) have hot rolled the attritor-milled Al-Si-MWCNTs to produce sheets at 480 °C. They observed an increase of 25% in the yield strength of composite sheets along with 5% plastic elongation to failure. The good dispersion of MWCNTs was found during milling which contributes to the easy consolidation of powders. The higher strength of Al-Si-MWCNTs composite has been attributed to the presence of MWCNTs and reduced grain size as well as solid solution formation. Besides, Esawi et al., (2009) and Esawi & Borady (2008) have also reported about 50% increase in strength of pure Al-CNT containing different amount of CNTs (up to 2%) than pure bulk Al during milling.

*Spark plasma sintering (SPS) or extrusion (SPE)*

Spark plasma sintering (SPS) is developed process of sintering for consolidation purposes wherein powders are heated using a pulsed direct current while compacting under uniaxial pressure inside a

graphite die. One step sintering and compaction during SPS process helps to eliminate the contaminants and oxidation of particles contributing the effective bonding between particles (Dudina & Mukherjee, 2013; Tokita, 1993; Saheb et al., 2012; Orrù et al., 2009). One major advantage of SPS is that powders are densified at shorter sintering time, lower sintering temperature, and higher rates to produce near to fully dense structure with limited grain growth. Further information about SPS process, its mechanism and sintering capability in the case of MMCs can be found elsewhere (Anselmi-Tamburini et al., 2005; Anselmi-Tamburini et al., 2005; Munir et al., 2006; Munir et al., 2011).

Al-MWCNT nanocomposites containing (1 and 5 vol-% MWCNTs) have been synthesized by a combination of spark plasma sintering and hot extrusion by Kwon, Estili, Takagi, et al., (2009), Kwon & Kawasaki, (2009) and Kwon, Park, et al., (2010). The CNTs were found to be homogeneously dispersed into Al matrix to fabricate a highly densified nanocomposite. The structural defects of CNTs were analyzed by Raman spectroscopy analysis showed little damages to the CNTs. The highly dispersed and undamaged CNTs contribute to the improvement of tensile strength of nanocomposites than pure aluminum (Kwon & Kawasaki, 2009). They also have found small amount of carbide as a result of reaction between Al and damaged CNTs which affect the bonding between particles and consequently mechanical properties.

Although SPS has many advantages, but it has limitations for processing of complicated shapes due to its inherent geometric configuration. Spark plasma extrusion (SPE) has been introduced as an extension of SPS wherein cold compacted powders are simultaneously extruded at elevated temperature under the influence of current providing unique properties (Morsi et al., 2010; Morsi et al., 2009; Morsi et al., 2010; Mamedov, 2002). SPE process enables the production of composite with extended geometries using powder materials (Morsi et al., 2009). Besides, SPE process allows bulk stress-induced deformation as a result of electric current. Further, grain refinement can be take place due to the material recrystallization during SPE which does not normally during SPS (Morsi et al., 2009).

Morsi, Esawi, Lanka, et al., (2010) and Morsi, Esawi, Borah, et al., (2010) have shown the feasibility of the SPE process for consolidation of Al-CNT composites. Both references reported successful consolidation of ball milled Al-CNTs. Morsi, Esawi, Lanka, et al., (2010) have consolidated ball milled pure Al-CNTs by means of spark plasma extrusion for the first time. They reported improved hardness (33%) and compressive strength (33%) of spark plasma extruded composites than pure aluminum. This improvement has been attributed to the dispersion strengthening effect of CNTs and refinement of aluminum particles than the milled powder. Morsi, Esawi, Borah, et al., (2010) have observed that extrusion temperature have no significant effect on the hardness while higher extrusion onset temperature reduces the compressive strength.

#### *Severe plastic deformation techniques (SPD)*

Recently, various severe plastic deformation (SPD) methods have been used to synthesize Al-CNT composites by imposing high levels of strains. The commonly used processes are equal channel

angular pressing (ECAP) (Balog et al., 2009; Derakhshandeh & Jenabali Jahromi, 2011) and high pressure torsion (HPT) (Valiev & Langdon, 2006; Valiev et al., 1996; Edalati & Horita, 2010). The initial purpose of SPD is the grain refinement in various materials at room temperature (Jenei et al., 2011; Musa & Schauerl, 2013). Lesser residual defect and contamination in the composite powders and compaction are the main advantages of SPD when compare with gas condensation or ball milling with subsequent consolidation (Quang et al., 2007).

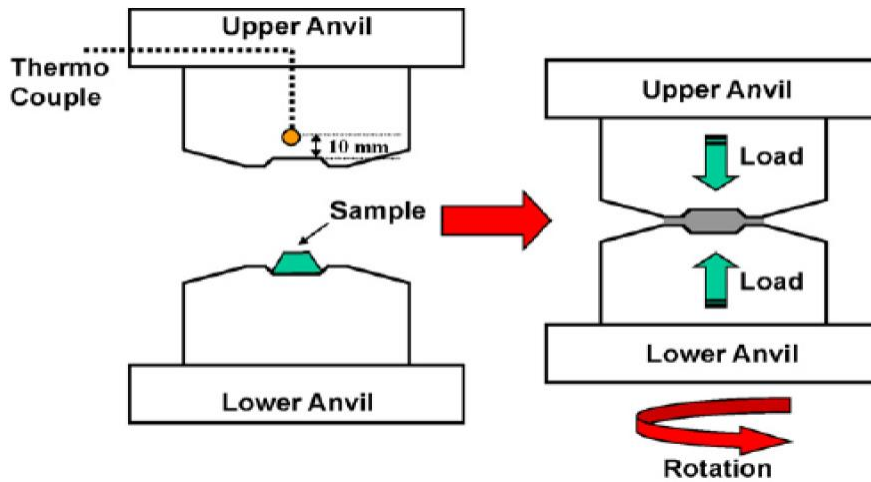


Figure 6: A diagram of the high pressure torsion apparatus used (Tokunaga et al., 2008).

#### High pressure torsion (HPT)

High pressure torsion (HPT) process is a novel technique for fabrication of AMCs wherein these composites are fabricated under torsion pressure which neither requires subsequent sintering or heating. In fact, simultaneous acting of a high compaction pressure and large shear strain enables refinement, synthesizing and consolidating of composite powders at room temperature (Zhilyaev & Langdon, 2008; Tokunaga et al., 2008). The schematic of the high pressure torsion apparatus is shown in Figure 6 (Tokunaga et al., 2008).

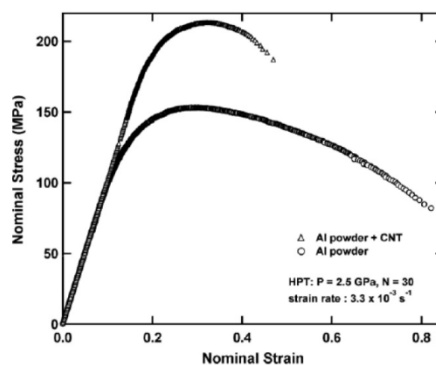


Figure 7: Stress strain curve obtained for Al/CNT samples and Al powders samples (Tokunaga et al., 2008).

During the HPT process, CNTs are substantially deformed and fractured into very fine particles under high compaction pressure and homogeneously distributed within the aluminum matrix. The huge shear strain which impose to the particles in a shorter duration contribute to further refinement of CNT particle powders and obtaining a full density composite even at high volume fractions of reinforcements. Further, HPT has shown to be a superior consolidation process even at low sintering temperatures (Edalati & Horita, 2010). In the case of consolidation, HPT had also been reported to be superior than hot extrusion equal channel angular extrusion (ECAE) and ECAP where high temperature is required (Zhilyaev & Langdon, 2008; Tokunaga et al., 2008; Edalati & Horita, 2010). This novel technique proves to be a promising method for fabrication of Al-CNTs. Tokunaga et al. (2008) were initially mixed 5 wt.% CNTs with pure aluminum via ultra-sonication using ethanol for 5 min. They were fabricated both pure aluminum and pure Al-5 wt.% CNTs using HPT process. The pressure applied during torsion was 2.5 GPa and the lower anvil was rotated for 30 turns. They reported that the experimental density close to the theoretical density since their samples did not reveal any porosity.

The strength of Al-CNT composites was found to be higher than the pure aluminum samples as can be seen in Figure 7 (Tokunaga et al., 2008). The tensile strength was reported to be more than 200 MPa with reasonable ductility. Besides, Al-CNT composites showed finer grain size (100 nm) much less as compared to the samples without CNTs. In another study, Joo et al., (2010) have consolidated ball milled Al- 5 Vol.% CNT using the HPT process at elevated temperature of 200 °C. They observed equilibrium grain boundaries with high misorientation in HPTed composites. This ultrafine grained microstructural features of Al-CNTs leads to higher strength and ductility of HPTed composites (Zhilyaev & Langdon, 2008).

#### *Equal channel angular pressing (ECAP)*

Equal channel angular pressing (ECAP) involves the passage of material being extruded through specially designed channel die which impose exceptionally high shear strain in each passage. This high strain introduces a high density of dislocations into the crystalline lattice of material being extruded (Derakhshandeh Haghighi & Jenabali Jahromi, 2012; Valiev & Langdon, 2006; Musa & Schauerl, 2013; Quang et al., 2007; Derakhshandeh & Jenabali Jahromi, 2011). A schematic of ECAP process is shown in Figure 8 (Poletti et al., 2008). Valiev & Langdon (2006) and Musa & Schauerl, (2013) have reviewed fundamental, principles and variables of ECAP processing method. ECAP has revealed to be an efficient method in powder consolidation of AMCs to obtain a full dense composite.

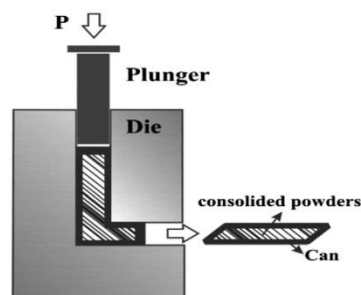


Figure 8: Schematic of a general ECAP method (Poletti et al., 2008).

As can be seen in Figure 8, composite powders are canned and pressed through channels inside a die wherein powders severely deformed and both densification of the powders and the refinement of microstructure happen (Quang et al., 2007; kollo et al., 2012). A backpressure is applied at specific speed depending on the material and intended size of the pressed specimen in order to press the powders (Quang et al., 2007). These channels are intersecting at the certain angles and having same shape and size, therefore, no transverse section changes during process is expected (Quang et al., 2007). This procedure repeats several times until a compaction with a dense microstructure is obtained under the effect of intense plastic strain. The consolidation of powders of powders is carried out at low temperature, because the induced shear deformation is able to provide efficient bonding of particles (Poletti et al., 2008; Casati & Vedani, 2014). Therefore, complete compaction can be done in shorter times in contrast to conventional sintering processes which need a long time for atomic diffusion (Poletti et al., 2008). Process variables of ECAP which influence the process are number of passes, processing temperature and the different routes of process.

Generally, it is accepted that ECAP is a feasible process for production of these composite with a fully dense microstructure with good matrix-reinforcement bonding, yielding high strength properties. However, there is only limited investigation of the consolidation of Al-CNT composites using ECAP. Kollo et al., (2012) have investigated the consolidation of Al-SiC and Al-CNT powders using ECAP method. Kollo et al., (2012) found an improvement of about 60% in hardness and strength of both composites compared to conventional direct extrusion while loss of ductility about two times is reported. It is attributed to the grain refinement of microstructure under heavy shear deformation (cumulative strain) during the ECAP process. The same result has been found in the Cu-CNT composites by Quang et al., (2007). They also reported a homogenous distribution of CNTs within the aluminum matrix and reduction of porosities as a result of multiple passes. Kollo et al., (2012) have reported that produced composites are brittle due to the lack of back pressure during pressing. In the absence of sufficient back-pressure, no enough stresses which needed for consolidation are transferred to the particles. In this case, many researchers have suggested applying backpressure for increasing the hydrostatic pressure to provide better consolidation. Higher back pressure increases the pressing load and result in the increasing of density, the reduction of particle clustering and the reduction of cracks which sometimes form during ECAP consolidation. It is also reported that as number of passes increases, CNTs are broken under high shearing stress which contributes to the homogeneous distribution of CNT in the matrix. Senthil Saravanan et al., (2011) employed ECAP for consolidation of mechanically alloyed AA 4032-MWCNT powders. First, they synthesized MWCNTs through electric arc discharge method in open air atmosphere. Then, mixture of AA 4032 and MWCNTs were ball milled followed by consolidation by ECAP process. They observed a uniform dispersion of MWCNTs within the matrix along with good metallurgical bonds between AA 4032 and MWCNT particles. Further, they reported improvement in the hardness of composite.

## **Conclusion**

This work has reviewed the available solid state techniques for synthesizing of Al-CNTs composites and their corresponding effect on mechanical performance, however, further investigation of these approaches would be contributed to achieve high performance materials. With a brief introduction to

different available processes, capabilities of each processing technique have been reviewed in terms of mentioned challenges in fabrication of Al-CNTs. However, it is proven that addition of CNTs contributes to the improvement of mechanical properties but technique of dispersion of CNTs within aluminum matrix also could affect its distribution, A-CNT interface and consequently the mechanical properties.

#### **List of abbreviations**

<b>AMCs</b>	Aluminum metal matrix composites
<b>CNT</b>	Carbon nanotubes
<b>Al-CNTs</b>	CNT reinforced aluminum composites
<b>MMC</b>	Metal matrix composites
<b>PMC</b>	Polymer matrix composites
<b>CMC</b>	Ceramic matrix composites
<b>PM</b>	Powder metallurgy
<b>MA</b>	Mechanical alloying
<b>BPR</b>	Ball-to-powder weight ratio
<b>PCA</b>	Process control agent
<b>CIP</b>	Cold isostatic pressing
<b>HIP</b>	Hot isostatic pressing
<b>SPE</b>	Spark plasma extrusion
<b>SPD</b>	Sever plastic deformation
<b>HPT</b>	High pressure torsion
<b>ECAP</b>	Equal channel angular pressing
<b>ECAE</b>	Equal channel angular extrusion

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