



**UNIVERSITI PUTRA MALAYSIA**

***SYNTHESIS AND CHARACTERIZATION OF CARBON NANOTUBES  
AND  
NANO ALUMINA-REINFORCED ALUMINUM MATRIX  
NANOCOMPOSITES***

**MEYSAM TOOZANDEHJANI**

**ITMA 2018 10**



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By

**MEYSAM TOOZANDEHJANI**

**Thesis submitted to the School of Graduate Studies, Universiti Putra Malaysia, in  
Fulfillment of the Requirements for the Degree of Doctor of Philosophy**

**December 2017**

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**DEDICATION**

to

***My Parents***



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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

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**December 2017**

**Chair : Assoc. Prof. Khamirul Amin Matori, PhD**  
**Faculty : Institute of Advanced Technology**

Owing to the unique properties of carbon nanotubes (CNTs) and alumina ( $\text{Al}_2\text{O}_3$ ) nanoparticles, they have been incorporated into aluminum matrix as the reinforcement to fabricate light-weight Al- $\text{Al}_2\text{O}_3$  and Al-CNTs nanocomposites. Al- $\text{Al}_2\text{O}_3$  and Al-CNTs have been widely used in different applications in aerospace, automotive and military industries due to excellent combination of properties including high strength, high stiffness and good wear resistance as well as improved thermal and electrical properties. Addition of even small amount of these nanoparticles provides effective improvement of the overall physical and mechanical behaviour of nanocomposites. However, incorporating of CNTs and  $\text{Al}_2\text{O}_3$  nanoparticles into aluminum matrix is difficult task particularly when larger contents of these nanoparticles are used.

The aim of this research was to investigate the feasibility of synthesizing of Al- $\text{Al}_2\text{O}_3$  and Al-CNTs as well as hybrid Al-CNTs- $\text{Al}_2\text{O}_3$  nanocomposites by using a combination of mechanical ball milling and powder metallurgy route. The effect of hybridization of Al matrix by the addition of both CNTs and  $\text{Al}_2\text{O}_3$  nanoparticles on morphology and microstructural features and subsequent effect on physical and mechanical behavior of hybridized nanocomposites was investigated and compared with single-reinforced Al-CNTs and Al- $\text{Al}_2\text{O}_3$  nanocomposites. Finally, the applicability of ultrasonic non-destructive evaluation technique for characterization of the microstructure and mechanical properties of all nanocomposites was studied.

To achieve the proposed research objectives, all nanocomposites were synthesized using a combination of ball milling and powder metallurgy route. Ball milling at different times was employed to disperse different contents of CNTs and  $\text{Al}_2\text{O}_3$  nanoparticles within aluminum powder. Nanocomposite powders were then consolidated by compaction at 150 MPa followed by sintering at 530 °C. Structural and microstructural features were characterized as a function of milling time and

nanoparticles contents using SEM, FESEM, TEM, HRTEM, particle size analyzer, XRD and Raman spectroscopy. Physical and mechanical behavior of nanocomposites was characterized using density, hardness, compression and nanoindentation tests. Ultrasonic measurements were carried out to measure ultrasonic parameters; ultrasonic velocity and attenuation in order to establish a correlation between ultrasonic parameters and structural, microstructural and physical and mechanical properties of these nanocomposites.

The results revealed that designated ball milling contributes to a homogenous dispersion of different amount of CNTs and  $\text{Al}_2\text{O}_3$  nanoparticles, reduction of particle clustering, reduction of inter-particle spacing, reduction in particle size and grain refining. However, designated ball milling was found to be not efficient in dispersion of CNTs content more than 2 wt.% while it was efficient in dispersion of different amount of  $\text{Al}_2\text{O}_3$  up to 10 wt.%. These morphological and microstructural variations within milling process significantly affect the physical and mechanical behaviour of Al-CNTs and Al- $\text{Al}_2\text{O}_3$  nanocomposites. Most interestingly, hybridization of Al-CNTs- $\text{Al}_2\text{O}_3$  nanocomposites exerts better physical and mechanical properties as compared to Al-CNTs and Al- $\text{Al}_2\text{O}_3$  nanocomposites. It was found that micro-hardness, nano-hardness and Young's modulus of Al-2CNTs- $\text{Al}_2\text{O}_3$  nanocomposites significantly increases by increasing  $\text{Al}_2\text{O}_3$  content by 35%, 30 % and 38%, respectively. Further, ultrasonic measurement revealed a good correlation between ultrasonic parameters with microstructural features and mechanical properties induced by ball milling and further powder metallurgy route.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

## **SINTESIS DAN PENCIRIAN MATRIK ALUMINUM KOMPOSIT DIPERKUATKAN DENGAN TIUB NANO KARBON DAN NANO ALUMINA**

Oleh

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Disebabkan oleh keunikan sifat-sifat yang terdapat pada CNTs dan  $Al_2O_3$  nanopartikel, kedua-duanya telah digabungkan ke dalam matrik aluminium sebagai bahan pengukuh untuk menghasilkan Al- $Al_2O_3$  dan Al-CNTs nanokomposit yang ringan. Al- $Al_2O_3$  dan Al-CNTs telah digunakan secara meluas di pelbagai aplikasi dalam industri aeroangkasa, automotif dan ketenteraan kerana kombinasi ciri-ciri yang sangat baik termasuk kekuatan tinggi, kekukuhan tinggi dan rintangan haus yang baik serta dapat meningkatkan sifat terma dan elektrik. Penambahan walaupun sedikit nanopartikel ini memberikan peningkatan yang berkesan terhadap tingkah laku fizikal dan mekanikal nanokomposit. Walau bagaimanapun, penggabungan CNTs dan  $Al_2O_3$  nanopartikel ke dalam matriks aluminium adalah sesuatu yang sukar terutamanya apabila kandungan nanopartikel yang digunakan adalah lebih besar.

Tujuan penyelidikan ini adalah untuk mengkaji kebolehlaksanaan sintesis Al- $Al_2O_3$  dan Al-CNTs serta hibrid Al-CNTs- $Al_2O_3$  nanokomposit dengan menggunakan kombinasi kaedah pengisaran bebola mekanikal dan metalurgi serbuk. Kesan hibridisasi Al matriks dengan penambahan kedua-dua CNT dan nanopartikel  $Al_2O_3$  pada morfologi dan ciri-ciri mikrostruktur serta kesan selanjutnya terhadap tingkah laku fizikal dan mekanik nanokomposit hibrid telah dikaji dan dibandingkan dengan Al-CNTs dan Al- $Al_2O_3$  nanokomposit bahan pengukuh tunggal. Akhir sekali, kebolegunaan ultrasonik sebagai teknik penilaian yang tidak merosakkan untuk pencirian mikrostruktur dan sifat-sifat mekanik semua nanocomposites telah dikaji.

Untuk mencapai matlamat penyelidikan yang dicadangkan, nanokomposit disintesis menggunakan teknik gabungan pengisaran bebola mekanikal dan metalurgi serbuk. Teknik pengisaran bebola mekanikal dengan perbezaan masa yang berlainan digunakan untuk menyebarkan CNTs dan  $Al_2O_3$  nanopartikel yang berbeza kandungan ke dalam serbuk aluminium. Serbuk nanokomposit ini kemudiannya dibentuk dengan proses

pemadatan pada tekanan 150 MPa dan diikuti dengan proses pensinteran pada 530 °C. Ciri-ciri struktur dan mikrostruktur diukur merujuk kepada perbezaan masa pengisaran dan kandungan nanopartikel dengan menggunakan SEM, FESEM, TEM, HRTEM, penganalisis saiz zarah, XRD dan Raman spektroskopi. Ciri-ciri fizikal dan mekanik nanokomposit dianalisis merujuk dengan menggunakan ujikaji ketumpatan, kekerasan, ujian mampatan dan ujian nanoindentasi. Analisis ultrasonik digunakan untuk mengukur parameter ultrasonik; halaju dan pelemahan ultrasonik untuk membentuk hubungan antara parameter ultrasonik dan tingkah laku struktur mikrostruktural dan fizikal nanokomposit.

Keputusan kajian menunjukkan bahawa pengilangan bola yang ditetapkan secara amnya menyumbang kepada penyebaran homogen nanopartikel CNTs dan Al<sub>2</sub>O<sub>3</sub> dalam jumlah yang berbeza, pengurangan pengelompokan zarah, pengurangan jarak antar partikel antara zarah, pengurangan saiz zarah dan penghalusan butiran. Walau bagaimanapun, pengisaran bola yang ditetapkan adalah tidak berkesan untuk penyebaran kandungan CNTs yang lebih daripada 2% tetapi ianya lebih efisien dengan penyebaran sehingga 10% berat Al<sub>2</sub>O<sub>3</sub>. Variasi morfologi dan mikrostruktur dengan menggunakan proses ini telah meningkatkan keupayaan fizikal dan mekanikal kedua-dua Al-CNTs dan Al-Al<sub>2</sub>O<sub>3</sub> nanokomposit. Paling menarik, Al-CNTs-Al<sub>2</sub>O<sub>3</sub> hibrid nanokomposit telah menunjukkan ciri-ciri mekanikal yang lebih baik berbanding dengan Al-CNTs dan Al-Al<sub>2</sub>O<sub>3</sub> nanokomposit. Telah didapati bahawa kekerasan mikro, kekerasan nano dan modulus Young daripada Al-2CNTs-Al<sub>2</sub>O<sub>3</sub> nanokomposit meningkat dengan ketara dengan meningkatkan kandungan Al<sub>2</sub>O<sub>3</sub> sebanyak 35%, 30% dan 38%. Selanjutnya, pengukuran ultrasonik menunjukkan korelasi yang baik antara parameter ultrasonik dengan ciri-ciri mikrostruktur dan sifat-sifat mekanikal yang disebabkan oleh teknik pengisaran bebola mekanikal dan metalurgi serbuk.



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I certify that a Thesis Examination Committee has met on 29 DEC 2017 to conduct the final examination of MEYSAM TOOZANDEHJANI on his thesis entitled “Synthesis and characterization of carbon nanotubes and nano alumina-reinforced aluminum matrix nanocomposites” in accordance with the Universities and Universiti College Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The committee recommends that the student be awarded the doctor of philosophy. Members of the Thesis Examination Committee were as follows:

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## LIST OF ABBRAVIATIONS

Al	Aluminum
MMCs	Metal Matrix Composites
AMCs	Aluminum Matrix Composites
CNTs	Carbon Nanotubes
PM	Powder Metallurgy
NDEs	Nondestructive Evaluation Techniques
MWCNTs	Multi-walled Carbon Nanotubes
SWCNTs	Single-walled Nanotubes
EDX	Energy-dispersive X-Ray Spectrometer
Al <sub>2</sub> O <sub>3</sub>	Alumina
SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
HRTEM	High-resolution Transmission Electron Microscopy
XRD	X-Ray Diffraction
FESEM	Field Emission Scanning Electron Microscopy
PDC	Physical Dimensional Changes
CVD	Chemical Vapor Deposition

# CHAPTER 1

## INTRODUCTION

### 1.1 Introduction

In various industrial applications, aluminum (Al) and its alloys as a lightweight alloy are being replaced by aluminum matrix composites (MMCs) to achieve lightweight, high performance, and environmental friendly materials as well as energy saving (Miracle, 2005; Rohatgi and Schultz, 2007; Everett and Arsenault, 1991). Aluminum matrix composites (AMCs) are widely employed in different applications such as aerospace, automotive and military industries. The main reason behind the wide application of AMCs is their excellent combination of properties as compared to the unreinforced aluminum alloys. Those properties are high strength and stiffness, good wear resistance and improved thermal and electrical properties. In addition, the main contribution of AMCs in engineering applications is the weight reduction which exerts energy saving along with a reduction in the cost (Surappa, 2003; Kaczmar et al., 2000; Davis, 1999).

Like other MMCs, the properties of AMCs is closely associated to the reinforcement (its amount and type) and synthesizing processing of the composite (Rohatgi and Schultz, 2007; Casati and Vedani, 2014; Kaczmar, et al., 2000). A wide variety of reinforcements have been used to reinforce aluminum matrix such as SiC, B<sub>4</sub>C, Al<sub>2</sub>O<sub>3</sub>, AlN, Si<sub>3</sub>N<sub>4</sub>, TiC, TiO<sub>2</sub>, TiB<sub>2</sub> in different type, shape, and sizes. The most widely used reinforcements are SiC, 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 (Wessel, 2004; Casati and Vedani, 2014). Carbon nanotubes (CNTs) have also gained interest among researchers as a reinforcement material for fabrication of novel AMCs (Salvetat et al., 1999; Bakshi and Agarwal, 2011; Bradbury, 2014; Esawi et al., 2010; Popov, 2004).

Different techniques have been employed to manufacture AMCs including liquid state and solid state techniques. It is well known that different manufacturing techniques, the processing and the finishing, and also different reinforcements provide various characteristic profiles while the same composition and amounts of components are used (Uozumi et al., 2008). Among available techniques, powder metallurgy (PM) has shown its potential in the field as the most feasible and economical method for manufacturing AMCs. PM composites are increasingly used in aerospace and automotive industries due to the improved properties owing to the ability of PM process to provide uniform dispersion of reinforcement (Srivatsan et al., 1991; Torralba et al., 2003; O'Donnell and Looney, 2001). In fact, reinforcing aluminum matrix particularly with those in nano-sized range, necessitate a proper technique to yield a uniform dispersion of reinforcements. Mechanical milling process has found to be effective in homogeneous dispersion of reinforcements in the Al matrix along with refinement of the microstructure (Toozandehjani et al., 2017; Ostovan, Matori and Toozandehjani et al., 2016; Suryanarayana, 2001). Mechanical milling through ball milling provides better homogeneous dispersion of the reinforcement particles within



the aluminum matrix which avoids the segregation and agglomeration of the reinforcement particles (Razavi Hesabi et al., 2007; Esawi and Morsi, 2007; Ostovan, Matori and Toozandehjani et al., 2015; Fogagnolo, Velasco, et al., 2003; Toozandehjani et al., 2017).

Non-destructive evaluation techniques (NDEs) have been introduced to the MMCs (Mott and Liaw, 1988; Liaw et al., 1992; Liaw et al., 1994; Lu and Liaw, 1995; Gür and Ogel, 2001; El-Daly et al., 2010). NDEs are generally used for defect detection and characterization of microstructure. However, NDEs have also shown their potential in characterization of microstructure and mechanical properties of materials. Ultrasonic technique is considered to be the most practically feasible technique for evaluation of microstructure and mechanical properties of alloys (Toozandehjani, Matori, et al., 2015). The ultrasonic parameters; velocity and attenuation provide information about the structural and geometrical parameters of the material which can be used for the analysis and design of AMCs. It has been proven that indirect assessment of the microstructure and mechanical properties of AMCs by measurement of ultrasonic parameters is possible (Toozandehjani, Matori, et al., 2015).

## 1.2 Problem Statement

Owing to the unique properties of CNTs and  $\text{Al}_2\text{O}_3$  nanoparticles, they have incorporated into the aluminum matrix as a reinforcement to fabricate alumina reinforced aluminum matrix composites (Al- $\text{Al}_2\text{O}_3$ ) and carbon nanotubes reinforced aluminum matrix composites (Al-CNTs). Addition of even small amount of these nanoparticle reinforcements provides an effective improvement of the physical and mechanical behavior of these nanocomposites. Incorporating CNTs and  $\text{Al}_2\text{O}_3$  nanoparticles into the aluminum matrix is difficult task particularly when a larger content of these nanoparticles is used (Ostovan, 2015). As a matter of fact, dispersion technique of CNTs and  $\text{Al}_2\text{O}_3$  into aluminum matrix affects the degree of dispersion CNTs and  $\text{Al}_2\text{O}_3$  nanoparticles, interfacial features of constituent and consequently physical and mechanical behavior of nanocomposites. It might even decrease the mechanical properties of aluminum (Estruga et al., 2013; Suryanarayana and Al-Aqeeli, 2013; Uozumi et al., 2008). Clustering of reinforcements is a challenging issue in aluminum composites. In particular case of hybrid nanocomposites, simultaneous incorporation of two different nanoparticle reinforcements and their dispersion seems to be more difficult since  $\text{Al}_2\text{O}_3$  and CNTs nanoparticles are different in nature, one sphere shape and one fibrous (nanotubes shape). A review of the existed literature revealed that only limited studies have been carried out on the intrinsic effect of the hybridization of aluminum using  $\text{Al}_2\text{O}_3$  and CNTs nanoparticles by adopting a combination of ball milling and powder metallurgy processing route. Due to the technological interest of ball milling, the effect of ball milling time on the dispersion of  $\text{Al}_2\text{O}_3$  and CNTs nanoparticles in hybridized Al-CNTs- $\text{Al}_2\text{O}_3$  nanocomposites is not fully investigated.

In addition, different characterization techniques have been employed to investigate the effect of the addition of CNT and  $\text{Al}_2\text{O}_3$  nanoparticles into Al matrix and synthesizing process parameters on the final properties of nanocomposites. Recently, ultrasonic

characterization is of interest of researchers for evaluation of properties of MMCs since ultrasonic wave parameters; velocity and attenuation are functionally related to structural and microstructural features of MMCs. Although, some works have been done on the ultrasonic characterization of AMCs (Gür and Ogel, 2001; El-Daly et al., 2010; El-Daly et al., 2013), but the ultrasonic characterization of Al-CNTs, Al-Al<sub>2</sub>O<sub>3</sub> and hybrid Al-CNTs-Al<sub>2</sub>O<sub>3</sub> nanocomposites has not been reported as far as deduced from the literature. There is still a high necessity to study in depth the effect of nanoparticle reinforcement and processing parameters on the ultrasonic properties of these nanocomposites. In addition, the effect of CNTs and Al<sub>2</sub>O<sub>3</sub> content and their dispersion state on ultrasonic parameters; velocity and attenuation are not fully studied yet. It is a well-known fact that the amount and dispersion of the nanoparticle reinforcements considerably influence the microstructure and mechanical properties of AMCs. Therefore, monitoring such variation with the help of nondestructive methods would be useful.

For the above-mentioned reasons, an experimental work was carried out in order to investigate the feasibility of synthesizing Al-CNTs and Al-Al<sub>2</sub>O<sub>3</sub> as well as hybridized Al-CNTs-Al<sub>2</sub>O<sub>3</sub> nanocomposite by a combination of ball milling and powder metallurgy route. The effect of the addition of CNTs and Al<sub>2</sub>O<sub>3</sub> nanoparticles and processing parameters on the morphological and microstructural features and subsequent effect on the physical and mechanical behavior of Al-CNTs, Al-Al<sub>2</sub>O<sub>3</sub>, and hybridized Al-CNTs-Al<sub>2</sub>O<sub>3</sub> nanocomposite is investigated. Additionally, the effect of hybridization of the aluminum matrix by the addition of CNT and Al<sub>2</sub>O<sub>3</sub> nanoparticles is compared with those of single reinforced Al-CNTs and Al-Al<sub>2</sub>O<sub>3</sub> nanocomposites. In addition, ultrasonic characterization has been used to characterize the synthesized nanocomposites in order to establish a correlation between microstructural features of nanocomposites and ultrasonic parameters; ultrasonic wave velocity and ultrasonic wave attenuation.

### 1.3 Hypothesis

The hypothesis may include the following:

1. Mechanical alloying through ball milling as a part of powder metallurgy route is a suitable for synthesizing of Al-CNTs and Al-Al<sub>2</sub>O<sub>3</sub> nanocomposites since it provides a good dispersion of CNTs and Al<sub>2</sub>O<sub>3</sub> nanoparticles and probably a combination of both reinforcements.
2. CNTs and Al<sub>2</sub>O<sub>3</sub> nanoparticle reinforcements significantly improve the mechanical properties of the pure aluminum matrix.
3. Hybridization of aluminum matrix contributes to the further enhancements of mechanical properties compared to the single reinforced nanocomposites.
4. The microstructural and morphological variation within ball milling of Al-CNTs and Al-Al<sub>2</sub>O<sub>3</sub> nanocomposites are correlated to the ultrasonic parameters.

## 1.4 Objectives

This study embarks on the following specific objectives:

1. To study the feasibility of synthesizing Al-CNTs and Al-Al<sub>2</sub>O<sub>3</sub> as well as hybrid Al-CNTs-Al<sub>2</sub>O<sub>3</sub> nanocomposite using a combination of mechanical ball milling and powder metallurgy.
2. To investigate the effect of addition of CNTs and Al<sub>2</sub>O<sub>3</sub> nanoparticles and milling time on morphology, microstructure features and subsequent effect on the physical and mechanical behavior of Al-CNTs and Al-Al<sub>2</sub>O<sub>3</sub> nanocomposites.
3. To explore the effect of hybridization of Al matrix by simultaneous addition of both CNTs and Al<sub>2</sub>O<sub>3</sub> nanoparticles on morphology, microstructure features and subsequent effect on the physical and mechanical behavior of hybrid Al-CNTs-Al<sub>2</sub>O<sub>3</sub> nanocomposites compared with those of single reinforced Al-CNTs and Al-Al<sub>2</sub>O<sub>3</sub> nanocomposites.
4. To illustrate the applicability of ultrasonic techniques in characterization of the morphological, microstructural, physical and mechanical behavior of Al-CNTs and Al-Al<sub>2</sub>O<sub>3</sub> nanocomposites as well as hybridize Al-CNTs-Al<sub>2</sub>O<sub>3</sub> nanocomposites.

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