Powder Metallurgy Processing of Aluminum Matrix Reinforced with Carbon Nanotubes

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CERTIFICATION OF APPROVAL

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Approved by,

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MUHAMMAD AZLAN BIN MAS'OD

ABSTRACT

Carbon nanotubes have been used as reinforcing agents in materials because of their exceptional mechanical properties. For the project, the objective is to evaluate the mechanical and physical properties of aluminum matrix reinforced with carbon nanotube produced by powder metallurgical processing. In addition of carbon nanotubes in aluminum, it is predicted to affect wear performances to monolithic aluminum alloys. The hardness of the composite also will be studied. Since carbon particles are lighter than metallic alloys in term of its density, aluminum-carbon nanotube composites are expected to reduce the density of the components.

In order to investigate the influence of properties, two kinds of materials will be involved which are aluminum matrix reinforced with carbon nanotubes and the aluminum composite itself. The aluminum and carbon nanotubes powders will be mixed using ball mill machine in various compositions which are 2 wt%, 4 wt%, 6 wt%, 8 wt% and will be compacted at suitable pressure. The sintered products then will be prepared for analyzing.

The sample will be prepared with regard to the standard of ASTM B925. Microstructure, physical and mechanical properties and density of the said materials will be analyzed by using Field Emission Scanning Electron Microscope (FESEM), Energy Dispersive X-ray Analysis (EDX), X-Ray Diffraction (XRD) and density measurement instrument. The Vickers Hardness Testing will be use to evaluate the hardness of the composite as per ASTM E92-82 wear characteristics follow the standard of ASTM Standard G99 (Eyre, 1991) using DUCOM Multi Specimen Tester.

The nanotubes content affects significantly mechanical properties of composites. The 2.0 wt. % CNTs is found to exhibit the highest hardness strength and the best weight loss. The maximal increment of hardness strength and weight loss of the composite, compared with the pure aluminum matrix is 85% and 8%, respectively.

ACKNOWLEDGEMENT

First and foremost, praise to Allah the Almighty for with His blessings and guidance for me to successfully and smoothly complete this final year project. I am very grateful to my parents, who taught me the meaning of struggle in this life. Their generous supports, advices and loves are my strength in my life.

I would like to share my most gratitude to all people who involved directly or indirectly to my project. The special appreciation goes to my Supervisor, Dr. Ahmad Majdi Bin Abdul Rani and Co-Supervisor, AP Dr. Patthi Bin Hussain for their knowledge and experiences shared along this project and also invaluable contribution in this paper. They always provide me full support, time and discussion for matter regarding this research. Whenever I faced problems, they helped me to resolve thus achieve the target of the project. Besides, I appreciate their assistant in correcting all the mistakes throughout the research despite tight working schedule.

I am also thankful to all materials laboratories technicians and GAs for help and guidance in using the equipments. Heartfelt appreciation is given to University Technology PETRONAS (UTP) and Mechanical Engineering Department for equipping students with essential skills and excellent theoretical through my undergraduates study.

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CHAPTER 1
INTRODUCTION

1.1 Project Background

The importance in finding super reinforcements for metallic matrices has been growing very much over the past few years especially in focusing on investigating their contribution to the mechanical properties of metal matrix composites. The significance of using carbon nanotubes (CNTs) as reinforcement are the most promising of all nanomaterials for their potential application in storage systems, composite materials, field emission displays and other electronic applications [1].

Parallel to the enhancement of materials, Metal Matrix Composite (MMC) is the most reliable methods. MMC is widely used in aircraft and space system industry. This is a great need for new materials which improved strength at a reduced mass and size make lighter aircraft with lower fuel consumption. Reinforcement, matrix and interface are the three entities that measure the characteristics of a composite. Over the years, the matrix microstructure and its mechanical property have an influence on the overall performance of the composites [2].

With the increase in demand, carbon nanotube expected to give a solution to the phenomena and the composite is capable to make a high strength, light weight and high performance components. Current R&D is looking at improved macroscopic materials using nanomaterial additives which show the same promising properties on the macroscale as on the nanoscale. These are the reasons why the aeronautics industry is actively researching the exploitation possibilities of micro and nanotech.

In order to investigate the involvement of CNTs in aluminum matrix, Powder Metallurgy (PM) techniques will be the ideal method to produce sample from the materials. PM has several processes and is able to fabricate high quality parts with complex shapes and also high tolerance standards in an inexpensive fashion [3].

1.2 Problem Statement

The global passenger traffic is expected to increase steadily and it is important for the aviation company in making their aero plane like airbus to the best. To meet the expectation, the main things are about increased of safety, reduced noise, enhance payload, increase range and increased capacity.

Science and technology has become more urban especially in aircraft and automotive industry. So, the components and parts related also need to be enhanced. It is recommended to use lightweight material such as aluminum. In addition of carbon, it is expected to reduce the weight of the component and improves physical and mechanical properties in term of wear resistance and hardness. To investigate the influence of CNTs, aluminum matrix and CNTs will be processed via PM.

1.3 Objectives and Scope of Study

The objectives of this work are to investigate the density and hardness of the composite and also to determine the wear performance of the aluminum composite with various composition of carbon nanotubes which are 2 wt%, 4 wt%, 6 wt% and 8 wt%.

The scope of study would start with the knowledge gathering and theoretical studies. The two powder materials are mixed using ball mill and compact the mixture of powders using hydraulic press. After the green product already sintered, the samples are analyzed in which green compact and sintered densities are compared. Characterization of materials is using Field Emission Scanning Electron Microscope (FESEM) and Energy Dispersive X-ray Analysis (EDX) as well as X-Ray Diffraction (XRD). DUCOM Multi Specimen Tester will be used to investigate wear resistance in term of weight loss accordance with ASTM Standard G99 (Eyre, 1991) and hardness testing are conducted by Micro Hardness Testing Machine as per ASTM E92-82.

1.4 The Relevancy of the Project

Since the research regarding nanomaterials has been widely analyzed nowadays, many institutions decided to have a role in this particular subject of study. Thus, education level being so challenging and this is the good sign in term of making connection with what is being learned in the real world of industry. In doing the powder metallurgical processing of aluminum matrix reinforced with carbon nanotubes perhaps can be as a contribution effort to the world of nanomaterials. Universiti Teknologi PETRONAS (UTP) has latest technology in term of equipments and tools and its enable researchers to play their part in conjunction of studying nanosized materials.

1.5 Feasibility of the Project

In order to make the project practicable, articles, journals, books, internet and even lecturers have to be referred. The project begins with gathering materials to make sample from powder because the method that will be used is Powder Metallurgy. The powder of materials in which can be obtained at laboratory or factory outside of the university. The project should be in line with the time frame due to all the needed equipments already installed in the UTP research center. The equipment consists of powder metallurgy processing and the testing of samples such as density measurement machine, DUCOM Multi Specimen Tester and Micro-Hardness Testing Machine. Time duration is about eight months to make this project successful.

CHAPTER 2

LITERATURE REVIEW

Throughout chapter 2, the materials reviewed and the information related to the materials involved will be discussed deeper. It is important to know the details about materials being researched and the process involved from the beginning until the end.

2.1 Metal Powder

To use powder and powder compaction is proven to be a more economical method than other manufacturing methods. One type of material used in powder compaction is materials with a very high melting point where casting would not be economic because of the high melting point. Powdered metals include iron, aluminum, zinc, copper, bronze, brass, steel, stainless steel, nickel, platinum, palladium, titanium and silver.

Metal powders are used in powder metallurgy method. The technology of powdered metals related especially the production and utilization of metallic powders for fabricating massive materials and shaped objects. Powder metallurgy has become competitive with processes, particularly for relative complex parts made of high-strength and hard alloys. The most commonly powder used in powder metallurgy are iron, copper, aluminum, tin, nickel, titanium and the refractory metals [3].

2.2 Nanostructured Materials

Nanotechnology basically deals with the study of the controlling of matter on an atomic and molecular scale. The structured of nanotechnology sized between 1 to 100 nanometer in at least one dimension. According to the report of Nanoforum [4], nanostructured metals offer greater strength and hardness as well as good resistance in corrosion. These kinds of properties are much needed for applications including aerospace components such as landing gear and bulldozer blades.

The advantages of nanomaterials are:

1. High strength to weight ratio
2. Improved hardness, wear resistance and resilience
3. Thermal shock, fatigue and creep resistance
4. Multi-functional materials can reduce weight by reducing the number of components.
5. Enhanced anti-microbial activity (substance that kills the growth of microorganisms).

2.3 Composite

A composite material is known as a combination of two or more chemically dissimilar and insoluble phases in such a manner that its properties are superior compare to those constituents acting independently [3]. There are two categories of constituent materials which are matrix and reinforcement. Basically, the matrix material surrounds and supports the reinforcement materials by maintaining their relative positions. The reinforcement offers their special mechanical and physical properties to enhance the matrix properties. The very familiar classifications of composite materials based on matrix phase are Metal Matrix Composites (MMCs) and Ceramic Matrix Composites (CMCs).

Matrix materials in MMCs are usually aluminum, aluminum-lithium alloy, magnesium, copper, titanium and super alloys. Usually the matrix phase is the continuous phase and the other phase is said to be the dispersed phase. The terms such as "metal-matrix" indicate a metallic material used to form the continuous phase. The advantages of a metal matrix are higher elastic modulus, toughness, ductility and higher resistance to elevated temperatures. Besides, it serves very high specific stiffness, lightweight and high thermal conductivity. CMCs are important because it offers resistance to high temperatures and corrosive environments. Ceramics are strong and stiff but they generally lack toughness. Fiber materials are usually carbon and aluminum oxide and the matrix materials are silicon carbide, silicon nitride, aluminum oxide, carbon and mullite.

For the project, the author used Metal Matrix Composites from the material of aluminum as the matrix material and carbon nanotube as the reinforcement material. The class of composite is Aluminum Matrix Composites (AMCs)

2.3.1 Aluminum Matrix Composites (AMCs)

Table 2.1 and Table 2.2 below show the physical and mechanical properties of aluminum itself [5].

Table 2.1: Physical Properties of Aluminum [5]

Properties	**Aluminum**
Colour	Silvery white
Density	2.7 g/cm^3
Melting point	660.32 °C
Boiling point	2519 °C

Table 2.2: Mechanical Properties of Aluminum [5]

Properties	**Aluminum**
Crystal Structure	Face-centered cubic
Young Modulus	70 GPa
Vickers Hardness	167 MPa
Thermal Conductivity	237 $W.m^{-1}.K^{-1}$
Thermal Expansion	23.1 $\mu m \cdot m^{-1} \cdot K^{-1}$

The aluminum matrix composite give specific advantages and disadvantages compared to unreinforced aluminum alloys and to ceramic-matrix composite. Table 2.3 and Table 2.4 show the comparison between them [17]:

Table 2.3: Compared to un-reinforced aluminum alloys

Advantages	**Disadvantages**
Higher specific strength	Lower toughness and ductility
Higher specific stiffness	Complicated and expensive production method
Improved creep resistance	-

Table 2.4: Compared to ceramic matrix composites

Advantages	**Disadvantages**
Higher toughness and ductility	Inferior high temperature capability
Ease of fabrication	-
Lower cost	-

In particular, aluminum based MMC offer a unique combination of lightweight, high specific stiffness, good fatigue properties and good thermal and electrical conductivity [5]. This is because aluminum has low density, lightweight, capability to be strengthened by precipitation, good corrosion resistance, high thermal and electrical conductivity and last but not least their high damping capacity. Aluminum and its alloys are the principal matrix materials for MMCs. These composites are suitable for structural where high strength-to-weight and stiffness-to-weight ratios are required. Aircraft and spacecraft are weight-sensitive structures in which composite materials are cost-effective.

2.3.2 Carbon Nanotubes (CNTs)

Over the past 15 years, carbon nanotubes have been evolved into one of the most intensively studied materials of the decade. Carbon Nanotubes is a tube-shaped material made of carbon having a diameter measuring on the nanometer scale. CNTs are formed from essentially the same graphite sheet and have many structures, different in length, thickness and have number of layers. Their characteristics differ depending on these variations so it will be either as metals or as semiconductors [6].

Carbon nanotubes has emerged as a material that has excellent properties more than any conventional materials. Even though CNTs is so tiny in size but they have a superb strength. The reason why is the way the carbon atoms are bonded together in the structure. Based on the carbon nanotube structure, every carbon atoms connected to three carbon atoms thus form a very good bond. Physical property is the properties that can be observed or measured without changing the composition of matter. The overview of the physical properties of carbon nanotube can be referred to Table 2.5 below.

Table 2.5: Physical properties of CNTs [6]

Physical Properties	**Carbon Nanotubes**
Color	Clumpy, black or dark gray powder
Density	1.3-1.4 g/cm^3
Melting point	3,652~3,697 °C
Boiling point	Negligible
Solubility in water	Insoluble
Odor	Odorless

CNT have diameters ranging from <1 nm up to 50 nm and can be categorized by their structures as per below [6]:

1. Single-wall Nanotubes (SWNT)
2. Double-wall Nanotubes (DWNT)
3. Multi-wall Nanotubes (MWNT)

1. Single-Walled Nanotubes (SWNT)

   Most SWNT have diameter of 1 nanometer and the tube length can be many millions of time longer. It is an important variety of carbon nanotube because they exhibit electric properties that are not shared by the MWNT variants. The band gap can be wary from zero to 2 eV and electrical conductivity can show metallic or semiconducting behavior. SWNT is very useful in the development of the first intramolecular field effect transistors (FET) in electronic industry.

2. Multi-Walled Nanotubes (MWNT)

MWNT consists of multiple rolled layer of graphite and is a zero gap metals. This material is unique because their morphology and properties are similar to SWNT but their resistance to chemicals is improved. This behavior is useful in the aviation industry as the materials will interact with air and other environment most of the time.

3. Double-wall Nanotubes (DWNT)

DWNT are the simplest archetypical manifestation of MWNT and combine the properties of SWNT with the possibility to study coaxial intertube interactions with high precision. This material is the synthetic blend of SWNT and MWNT thus exhibit the electrical and thermal stability of the latter and the flexibility of the former [3].

Compared to other engineering fibers like High Strength Steel, Carbon Fiber and E/S glass, CNTs has good mechanical properties in term of specific density, modulus of elasticity (E) and strength. The said properties of CNTs is the lowest among all which is 1.3 g/cm^3, E is 1 Tpa and has 10-60 Gpa strength [6].

The author managed to use MWNT in this experiment as the reinforcement for aluminum. MWNT is easy to require and less expensive compared to other type of carbon nanotubes.

2.4 Testing Properties

It is important to understand the theory regarding the testing that was applied to the project. Following sub-section will describe the significance of particular testing in order to evaluate the effects of CNTs reinforcement on aluminum properties.

2.4.1 Density

The materials to build the structures of aircraft are vital and play an important role for the duration of the airplanes. One of the factors related to the subject is to consider the density. The best materials for aircraft are those with high specific properties such as mechanical properties / density. The early 1920s, most of the aircraft built mainly of wooden structural members but as aircraft became bigger, serious problems due to fungal rot forced designers to consider metallic aircraft [18]. Density determines the factor of mass that can be occupied per one unit of volume. Lower the density, better for aviation.

2.4.2 Hardness

Hardness values determine the resistant of solid matter to defy various kind of permanent shape change due to force applied. In other word, it is the property to stand firm from plastic deformation, penetration, indentation and scratching. Several methods have been developed for hardness testing with the likes of Brinell, Rockwell, Vickers, Tukon, Sclerscope, and the files test take have their own uniqueness to measure strength. In designing a new aircraft, it has to be light structures and strong enough airframe [19]. Hence, the combination of density and hardness strength aspect has to be just right in order to build a very successful aircraft.

2.4.3 Wear Properties

Components like brake steel rotors installed at aircraft structure really depend on wear properties. The most critical part is when a fully loaded aircraft aborting a takeoff just before the actual takeoff. To meet the condition of ever increasing higher speeds and loads, the primary requirement of modern brake systems is to be able to apply a braking torque while resisting failure due to high friction temperatures developed. The need to overcome the situation related to speed and weight in brake design thus leads to study the wear properties to the aluminum matrix reinforced with CNTs [20].

2.5 Aluminum Matrix Reinforced Carbon Nanotubes

I.-Y. Kim et al. / Wear 267 (2009) 593-598 [7] worked on aluminum matrix reinforced carbon nanotubes, the specimens were fabricated using spark plasma sintering with varies weight percentage (1, 3 and 5) of CNT in aluminum.

Reinforcement of CNTs in aluminum shows that increased in hardness value compared to the pure aluminum. It is believed that CNTs plays an important role in hardness strengthening. In relation of hardness to wear amount, higher hardness strength leads to lower wear amount with respect to weight loss. Figure 2.1 and Figure 2.2 shows the graph of Hardness (Hv) vs Carbon Nanotubes (wt. %) and wear amount of composites in varying CNT content respectively.

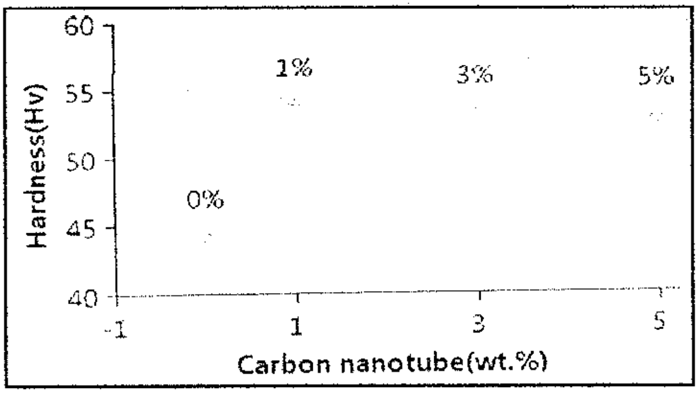


Figure 2.1: Hardness value according to CNTs content [7]

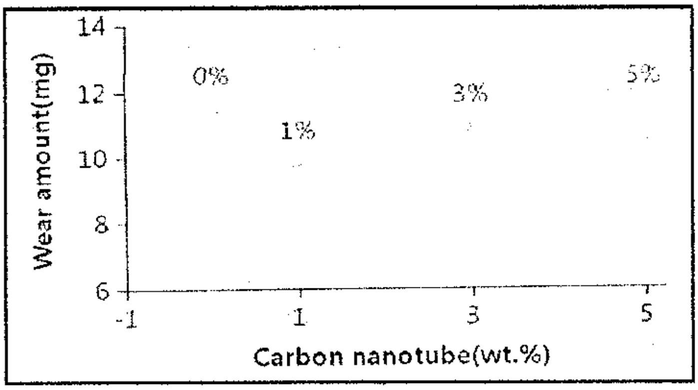


Figure 2.2: Wear amount with varies CNTs content [7]

The study by Tahir Ahmed and Othman Mamat from UTP has to be considered [8]. The study is about aluminum silica sand nanoparticle composites. The green and sintered density for the pure aluminum is in the range of 2.264 - 2.266 g/cm^3. Based on the research performed, the density of reinforced aluminum is lower than unreinforced aluminum and as the weight percentage of CNTs increased, the lower the green density recorded. Figure 2.3 indicates the result of green density getting lower with respect to increment of silica sand nanoparticles percentage in aluminum.

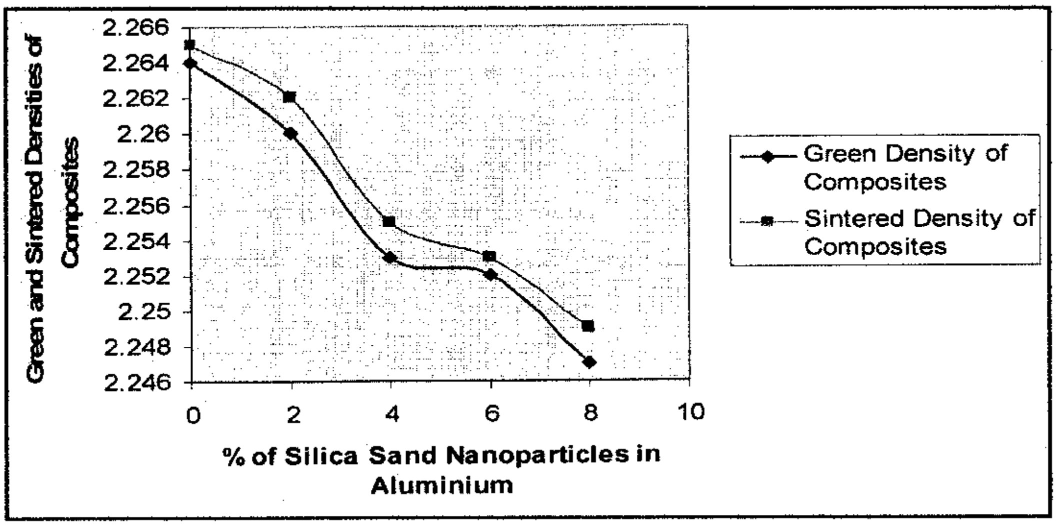


Figure 2.3: Green and Sintered Densities of Composites [8]

From the experiment that has been carried out by the researchers, these information and analysis can be the references to the ongoing project in term of determining the affect of various compositions of carbon nanotubes in aluminum matrix. The data that are obtained from the author's project are compared with the previous work of the researchers hence the objectives of the project can be evaluated.

CHAPTER 3
PROJECT WORK

With regard to the project milestone for final year project, the task activities have been discussed and summarized as in Table 3.2 and Table 3.3 at page 22 and page 23, respectively. This is to ensure that the project follows the time line that has been given and the author managed to finish the study successfully in time.

Powder Metallurgy (PM) is the method that the author employed throughout the project for making the product from aluminum reinforced with multi-walled nanotubes. This technique deals with making parts and products with metal powders as the source material as well as it is very cost effective in producing parts. Figure 3.1 below shows the outline of processes and operations involve in making powder-metallurgy parts in the project [3].

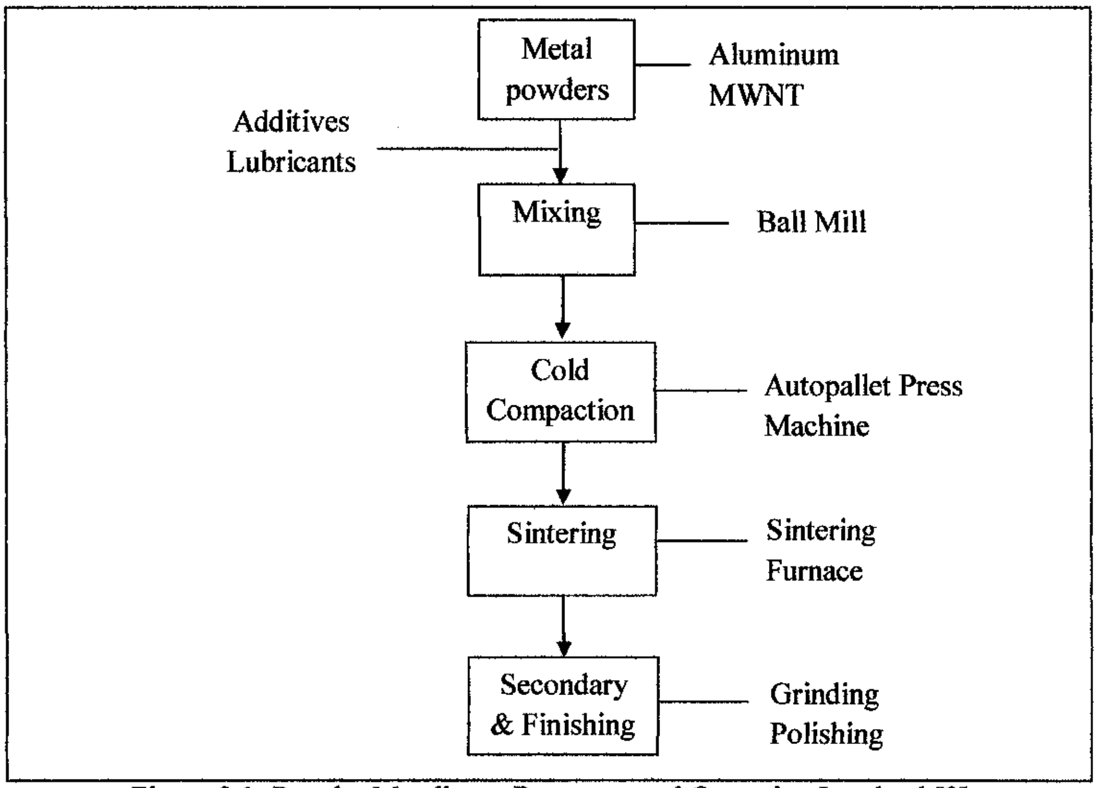


Figure 3.1: Powder Metallurgy Processes and Operation Involved [3]

3.1 Powder Metallurgy Process

3.1.1 Metal Powder

The powders involved in the project are aluminum and multi-walled nanotubes. The aluminum is the matrix material and the MWNT is the reinforcement material thus forming an aluminum matrix composite reinforced with carbon nanotube.

3.1.2 Mixing Process

The mixing process between the powders of aluminum and carbon nanotubes was comprised of 2wt%, 4wt%, 6wt% and 8wt% of MWNT. The mixing process was done by Ball Mill as indicated at Figure 3.2.

Figure 3.2: Ball Mill Machine

Ball Mill Machine [9] – Tag No.: 9-14/MPL/17-00-004/17

- Brand Type: US Stoneware
- Model: 764 AVM
- Speed range of between 20 and 300 RPM
- Motor is 1/4 HP, 60/50 cycles, 115 or 220V, single phase
- 764AVM is a two-jar capacity unit

The mixing process was carries out for eight hours of time duration in order to ensure that the mixing elements were perfectly homogeneous and get the uniform distribution of the mixture. This duration referred to the experimental study did by A. Esawi and K. Morsi in the experiment of Dispersion of Carbon Nanotubes (CNTs) in Aluminum Powder [10].

Current approaches for dispersing CNTs in metal matrices were reported using conventional mechanical mixing techniques. Ball milling is the one most commonly used and can be divided into low and high energy ball milling. However, the high energy mechanical alloying could damage the CNTs and destroys their tubular structure and affect to the overall mechanical properties [14]. Low energy ball milling is simple and less energetic than high energy ball milling. The advantage of low energy ball milling was that gravity separation was absent because the powder mixtures were rotated up and down with the mixing container thus effectively avoided the Al and CNTs separation due to density differences [2].

3.1.3 Compaction Process

After mixing process, the powders are pressed into shapes in dies to get the required shape, density and particle-to-particle contact and to build the part sufficiently strong for further processing. The shape mentioned is cylinder shaped that has diameter of 13mm and 5mm in height. Uniaxial press was used to compact the mixture of aluminum powder and CNT. PM involved cold pressing or hot pressing followed by sintering. Cold pressing to produce green body which is about 80% dense and can be handle easily. Cold pressing and sintering is a less desirable process since the high pressure needed to press the powder to the required density can break the fibers or the sintering process can degrade the fibers [2].

Pallet would be form based on the required shape as the result of the compaction process. For each composition, there would be 3 pallets created. This is to make sure the author obtained the average data and result later on for analyzing and testing of materials. Autopallet Press Machine was used to compact the powders mixtures at suitable load force. The picture of Autopallet Press Machine can be seen as in Figure 3.3.

Figure 3.3: Auto pallet Press Machine

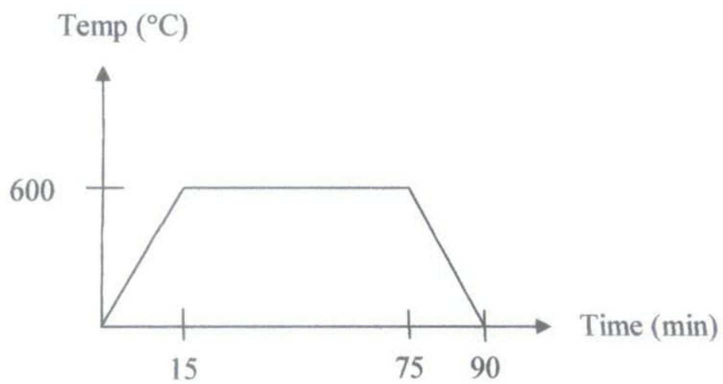
Auto pallet Press Machine - 25 ton and 40 ton clamp force
- Model: 9887.4SDOA00
- Motor HP: ¼
- Manufactured by: Carver Inc
- Includes 13mm KBr pellet die

The process will use the suitable load for compaction of powders in the dies in appropriate dwell time. This is to make sure that the force applied at the powder not damaged the material and affect the structure of the materials. The dies consist of the mold itself which have upper and lower mold and plunger. The overall operation takes time about five minutes to produce one unit of pallet. In this project, suitable load of 8000 lb was applied during compaction with the dwell time of two minutes.

3.1.4 Sintering Process

In this process, the compressed powder was heated to a temperature close to melting point in a controlled atmosphere furnace. It is significant to let the temperature sufficiently high enough to allow bonding between the particles. Throughout this project, the green compact were heated in the sintering furnace at 2/3 of the melting temperature of aluminum. The temperature was set to 600 °C in 90 minutes [8] as indicated at the heat treatment parameter. The sintering process was done in argon atmosphere because argon can include the sweeping of volatile reaction products from the furnace. Besides, it is capable to prevent of undesirable reactions and enabling of desirable reactions [11].

Heat Treatment Parameter



Temperature vs Time

The machine was used for sintering process modeled as Linn High Therm VMK Series which is a gas furnace that has maximum temperature of 1200 °C. The picture of said machine is illustrated as in Figure 3.4.

Figure 3.4: Sintering Furnace

Before operation, the machine was ensured that it is in good condition and in good safety.

<u>Standard Operating Procedure</u>

Starting the program:

1. Go to OPERATION display. Press E
2. Search for Programmer. Press E
3. Press F to start the heating process
4. ON the heater switch
5. OPEN the gas flow

Stopping the program:

1. After the furnace had cooled down
2. Press F
3. Press arrow up key to stop the program in reset mode
4. OFF the heater switch
5. OFF the gas

After operation, switch off POWER and do the CLEANING PROCESS. It recommended to machine running 24 hours for better analysis and stabilizes.

3.1.5 Secondary & Finishing

With the intention of further improve the properties of sintered powder metallurgy products, several additional operations may be carried out after sintering. Finishing operations are to provide additional complexity and precision also to enhance the performance properties of the parts in order to be prepared for testing like hardness and wear characteristics. The examples of finishing operation are machining, grinding, plating and heat treating [3].

3.2 Testing Equipments

3.2.1 Density Measurement

The densities of green compact and sintered pallets were determined by using density measurement machine, Mettler Toledo AX205. Density determinations were performed by Archimedes' Principle. The machine is illustrated in Figure 3.5.

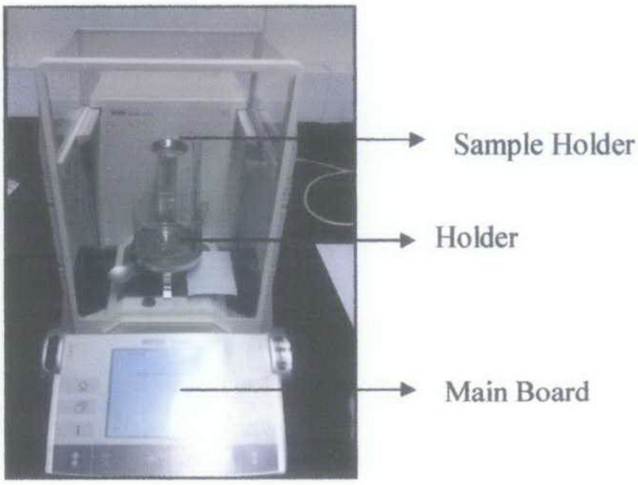


Figure 3.5: Density Measurement Machine

3.2.2 Hardness Testing

The basic principle of hardness testing as with all common measures of hardness is to observe the material's ability to resist plastic deformation from a standard source. Micro-Vickers Hardness Testing Machine was used to measure the hardness value of the composites and the machine is shown in Figure 3.6. Vickers test can be used for all metals and has one of the widest scales among hardness tests. The test load used for the study is 300 gf and dwell time is 15 sec.

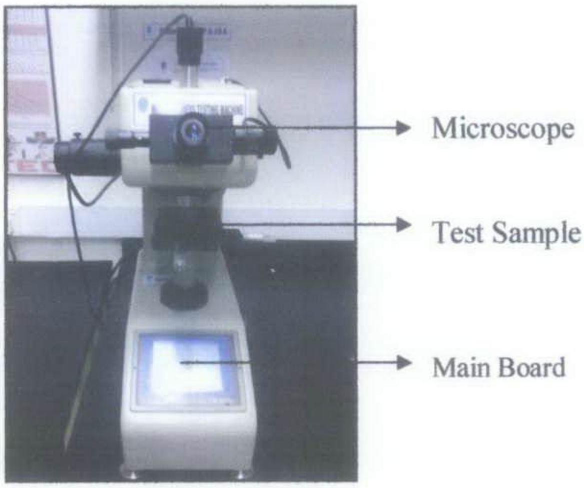


Figure 3.6: Micro Hardness Testing Machine

Operational Instructions:

1. Check upper lever is turned it comes to a standstill, towards the user.
2. Turn the hand wheel to raise the test sample until it comes in contact with the indenter. The display will show value 9 and the yellow lights up. Continue turning the hand wheel slowly until the value 0 appears.
3. Push the lever in a clockwise direction. The word STOP will appear and will remain displayed for the pre-set time. The red LED will flash intermittently and you will hear an acoustic signal.
4. At this point, pull the lever in a counterclockwise direction until it stops. Read the hardness value on the comparator.
5. Unscrewed the hand wheel to free the sample and remove it.

3.2.3 Microstructure and Elemental Analysis

FESEM is a microscope used to visualize very small topographic details on the surface objects. Electrons particles with a negative charge are liberated by a field emission source instead of light and then scanned by electrons according to a zig-zag pattern. Researchers apply this technique to observe structures that small as 1 nanometer within the high vacuum column. Usually, the electrons that are emitted from each spot on the object catch by detector and produce an electronic signal. This signal is amplified and transformed to a video scan-image that can be viewed on a monitor [15]. The illustration of FESEM machine can be obtained below as in Figure 3.7.

Figure 3.7: Field Emission Scanning Electron Microscope

One of the most useful features of FESEM analysis is Energy Dispersive X-ray Analysis (EDX). The machine is an analytical tool allows simultaneous non-destructive elemental analysis of the sample. X-ray technique used to identify the elemental composition of a specimen or chemical characterization of a sample. The basic principle is that the number and energy of the X-rays emitted from a specimen can be measured by an energy-dispersive spectrometer. As the energy of the atomic structure of the element from which they were emitted allows the elemental composition of the specimen to be determined

For the investigation of crystalline phase change during the milling process, X-Ray Diffraction (XRD) analysis was performed. XRD analysis in which it uses X-Rays of a known wavelength is passed through a sample to be identified just to know the crystal structure of the materials involved. Each of the materials has a unique pattern of reflection peaks at different angles and different intensity. The procedure starts with the X-Ray detector moves around the sample and measures the intensity of these peaks and the position of these peaks. The highest peak is defined as the 100% * peak and the intensity of all the other peaks are measured as a percentage of the 100% peak. Figure 3.8 shows the picture of XRD instrument used for crystalline phase change analysis.

Figure 3.8: XRD-Bruker AXS D8 Advance

3.2.4 Wear Resistance

The pin-on-disk method was used to analyze the wear characteristic of the aluminum matrix reinforced with carbon nanotubes. Wear resistance was determined by using DUCOM Multi Specimen Tester (Figure 3.8). It was performed according to ASTM Standard G99 (Eyre, 1991). This type of apparatus offers far better control of experimental conditions and become increasingly used in preference to other tribometers. Besides, it allows experiments to be conducted under relatively steady conditions without systematic variations [12].

Figure 3.9: DUCOM Multi Specimen Tester

The details regarding the pin-on-disk type wear testing apparatus can be referred to Figure 3.9. When using this machine, user can set the parameters by key in the values. The parameters for the DUCOM Multi Specimen Tester as per Table 3.1:

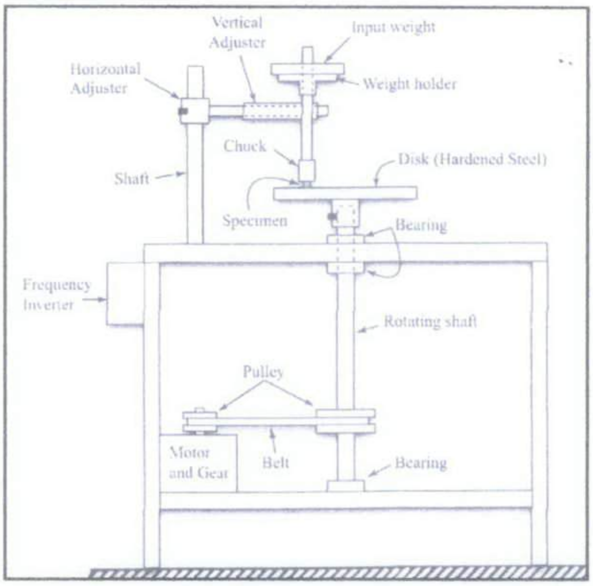


Figure 3.10: Schematic diagram of the pin-on-disk apparatus [12]

Table 3.1: DUCOM Multi Specimen Tester Parameters

Variables	**Unit**	**Values**
Load	Kg	1.5, 2.5, 3.5
Rotating speed	RPM	350, 450, 550
Time	Minute	3, 6, 9
Temperature	°C	Room temperature (24)
Geometry	-	Circular
Atmosphere	-	Relative Humidity: 70% Atmospheric pressure: 1atm
Material used	-	1) Test specimens (Counterface-1) 2) Disk Material Hardened Steel (Counterface-2)
Surface finish	-	Machined surface finish
Type of lubricant	-	Dry

The mechanism of pin-on-disc can be described as follow:

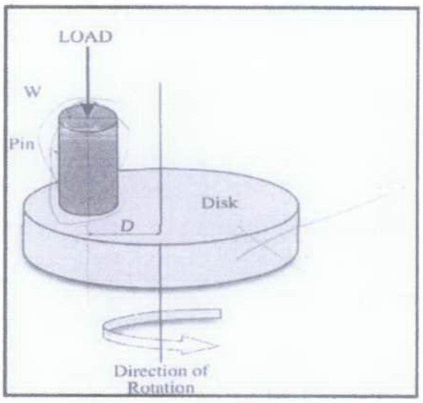


Figure 3.11: Diagram for pin-on-disc test

Samples located at the pin as the load would be applied. Disk would be turned in clockwise direction and the sample would experience friction that lead to abrasive type of wear. The author also managed to manufacture a holder that enables the pin to hold the 13mm diameter pallet. The holder is illustrates as below:

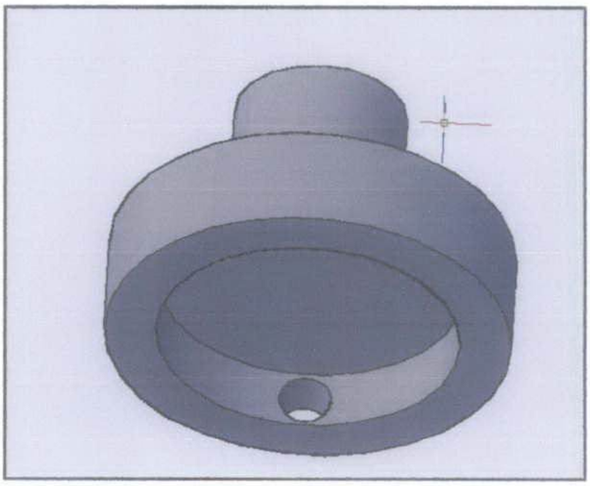


Figure 3.12: Sample holder of pin-on-disc

Table 3.2: Project Milestone for Final Year Project I

	ACTIVITIES	DETAIL	WEEK 1	2	3	4	5	6		7	8	9	10	11	12	13	14	15
1	FYP Documentation	Submission of title proposal	▲						MID SEMESTER BREAK									
2	Literature review	Concept of Powder Metallurgy	■	■	■	■												
3	FYP Documentation	Submission of Preliminary Report				▲												
4	Project Work	Gathering Materials			■	■	■	■										
4	Literature review	Aluminum				■	■	■										
5	Literature review	Carbon Nanotube				■	■	■										
6	Literature review	Method of Powder Metallurgy				■	■	■										
7	Literature review	Testing required on samples				■	■	■										
8	FYP Documentation	Submission of Progress Report									▲							
9	FYP Documentation	Seminar									▲							
10	Develop methodology	Develop procedure of powder metallurgy									■	■	■	■	■			
11	Literature review	Continue on literature review										■	■	■	■			
12	Project Work	Gathering of raw materials										■	■	■	■			
13	Project Work	Experimental set up										■	■	■	■			
14	Project Work	Preliminary study on raw materials										■	■	■	■			
13	Data and result documentation	Compilation of data and result													■	■	■	
14	FYP Documentation	Submission of Interim Report															▲	
15	FYP Documentation	Oral Presentation																■

■ : Process

▲ : Suggested Milestone

Table 3.3: Project Milestone for Final Year Project II

NO	ACTIVITIES	DETAIL	WEEK 1	2	3	4	5	6	7	MID SEMESTER BREAK	8	9	10	11	12	13	14	15
1	Literature review	Study on testing required on samples	■	■	■													
3	Literature review	Study on microstructure and elemental analysis	■	■	■	▲												
4	Project work	Sample set up		■	■	■												
4	Project work	Density & Hardness testing				■	■	■	■									
6	Project work	FESEM with EDX analysis						■										
	Project work	Fabricate pallet holder for wear testing				■	■	■	■									
7	Project work	Wear resistance testing							■		■							
8	Literature review	Continue on literature review									■							
9	FYP Documentation	Submission of Progress Report									▲							
10	Project work	Project work continue									■	■	■	■				
11	FYP Documentation	Pre-EDX												▲				
12	FYP Documentation	Submission of Draft Report													▲			
13	FYP Documentation	Submission of Dissertation (soft bound)														▲		
14	FYP Documentation	Submission of Technical Paper														▲		
13	FYP Documentation	Oral Presentation															▲	
14	FYP Documentation	Submission of Project Dissertation (Hard Bound)																▲

■ : Process

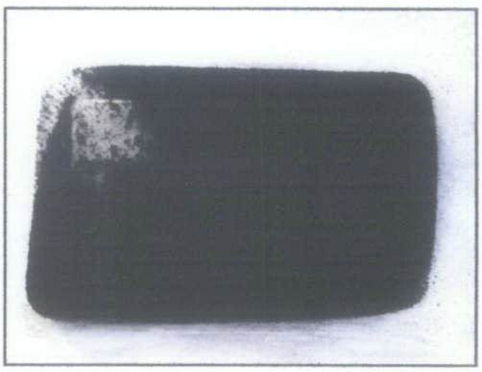
▲ : Suggested Milestone

CHAPTER 4
RESULT AND DISCUSSION

4.1 Materials and Microstructures of Composites

4.1.1 Analysis of Aluminum and CNTs

The powders of aluminum and carbon nanotube that have been the subject of study as per information below:

Aluminum Powder	
Aluminum, 99%, powder, 74 μm Molecular Weight=26.98 Al Acros Organics	 Figure 4.1: Aluminum Powder
Carbon Nanotube	
MWCNT 80 Carbon Purity: >80% Diameter: 5 to 15 nm Length: 1-5 μm	 Figure 4.2: MWCNT Powder

Size and distribution of Multi-Walled Carbon Nanotubes are shown in Figure 4.3. It is clearly observed of the agglomerations of the nanoparticles in the sample of aluminum reinforced with CNTs. It is clear evident that they are tangled together as their diameters are between 10 and 40 nm and lengths range from hundreds of nanometers to micrometers. Referred to the figure, most of CNTs are not straight and acquire some defects and localized kinks and bends [21]. Figure 4.4 shows the EDX analysis of the specimen illustrates the presence of CNTs in the samples. The weight and atomic percentage from the point of study that indicates CNT are 98.26 wt. % of C, 1.74 wt. % of Al and 99.22 wt. % of C, 0.78 wt. % Al, respectively.

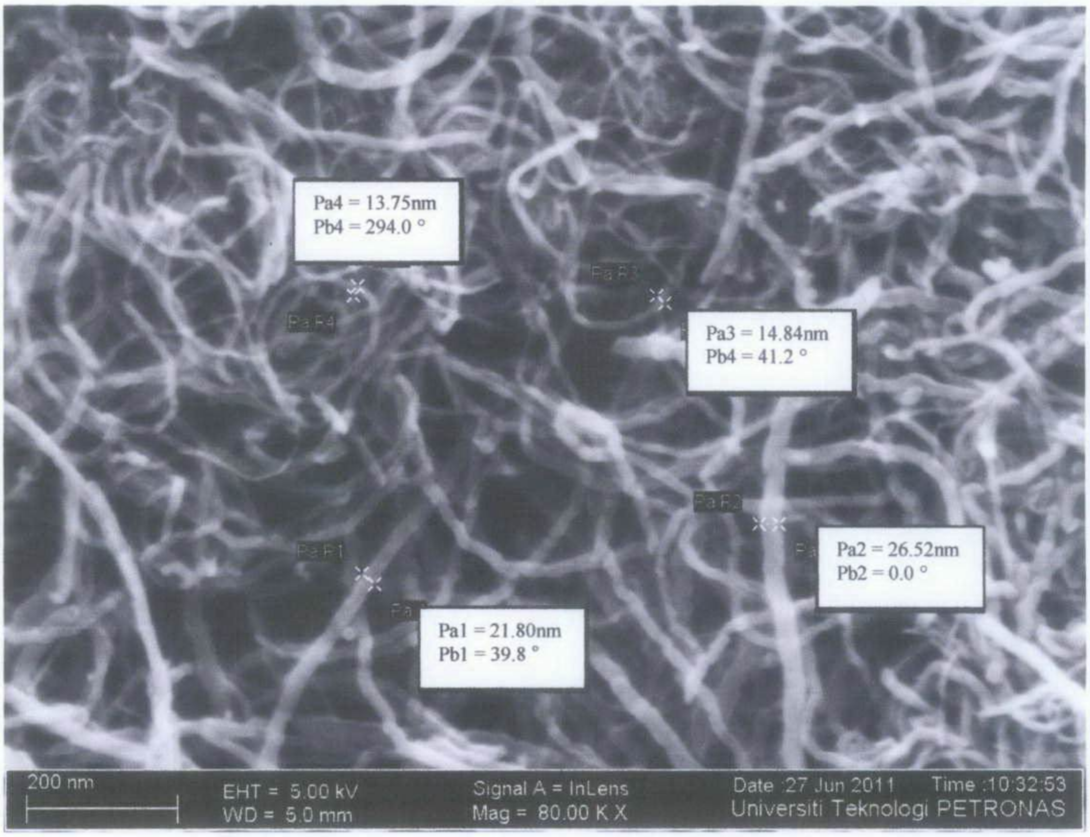


Figure 4.3: FESEM image of the CNTs

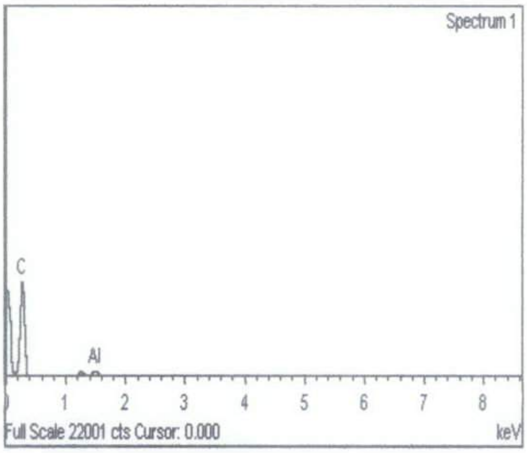


Figure 4.4: Presence of CNTs in EDX analysis

Regards to the study, here are the surface microstructure and the composition details of the aluminum can be seen in Figure 4.5. Weight and atomic percentage from the point of study of aluminum are 100%.

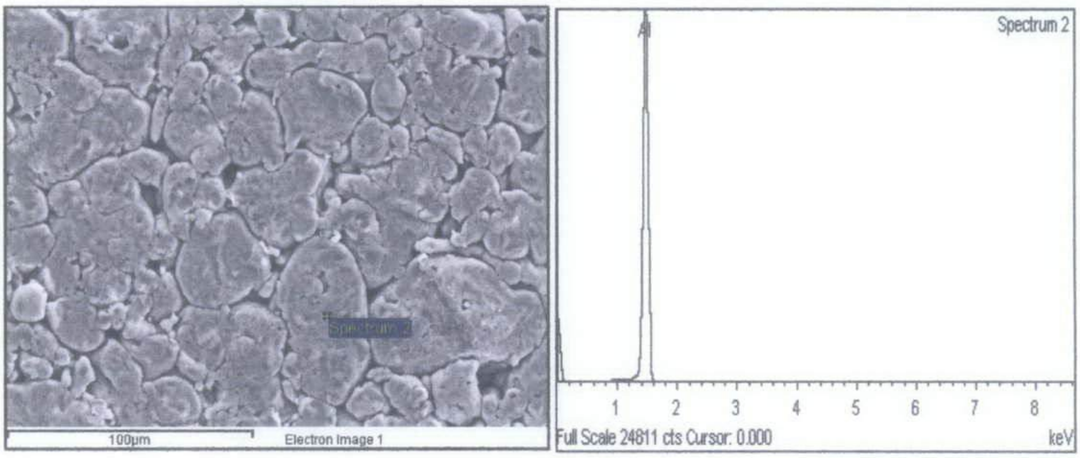


Figure 4.5: EDX analysis of pure aluminum

4.1.2 Analysis of the Aluminum Matrix Reinforced with CNTs Composites

The FESEM images averagely indicate homogeneously blended powders of carbon nanotubes and aluminum powders. It is apparent from the figures that the CNTs are distributed uniformly on the surfaces of the matrix and there is also agglomeration of carbon nanotubes were identified in the study. High carbon nanotubes content recognized would increase the agglomeration of CNTs in the mixed powders thus the material are prone to agglomerate when they are increment of carbon nanotubes quantity [21]. The FESEM images captured in Figure 4.6(a) notice that there are little amount of CNTs was detected in the aluminum that represent as black region. More presence of CNTs were identified in the matrix as compare to Figure 4.6(a) because this proved that the addition of 4 wt. % CNTs (Figure 4.6(b)). At the same time more amount of CNTs were found as highlighted in Figure 4.6(c) as compared with Figure 4.6(a) and Figure 4.6(b). In the picture of 8 wt. % CNTs (Figure 4.6(d)), the CNTs amount in the composite was inevitably seen as this is the highest reinforcement composition.

What is also noticeable in the figure is the formation of void scattered on the surface of specimens. Regions of CNTs resulted from powder agglomeration and residues of porosity can be seen clearly. However, compare the pictures of pure aluminum and 2 wt. % CNTs, the overall porosity of the composites is increased due to the addition. It is clearly identify that in addition of 4 wt. % CNTs, more porosity were found and as further addition of CNTs (6 and 8 wt. %), it is obvious and most pronounced increased in porosity. What stress here is that due to the fact more quantity of CNTs impedes the densification process, it will resulting in the increase of the microvoids. EDX analysis confirmed the presence of the reinforcement materials as per Figure 4.7(a), (b), (c) and (d). The curve of C more pronounced as increasing trend of CNTs embedded in aluminum.

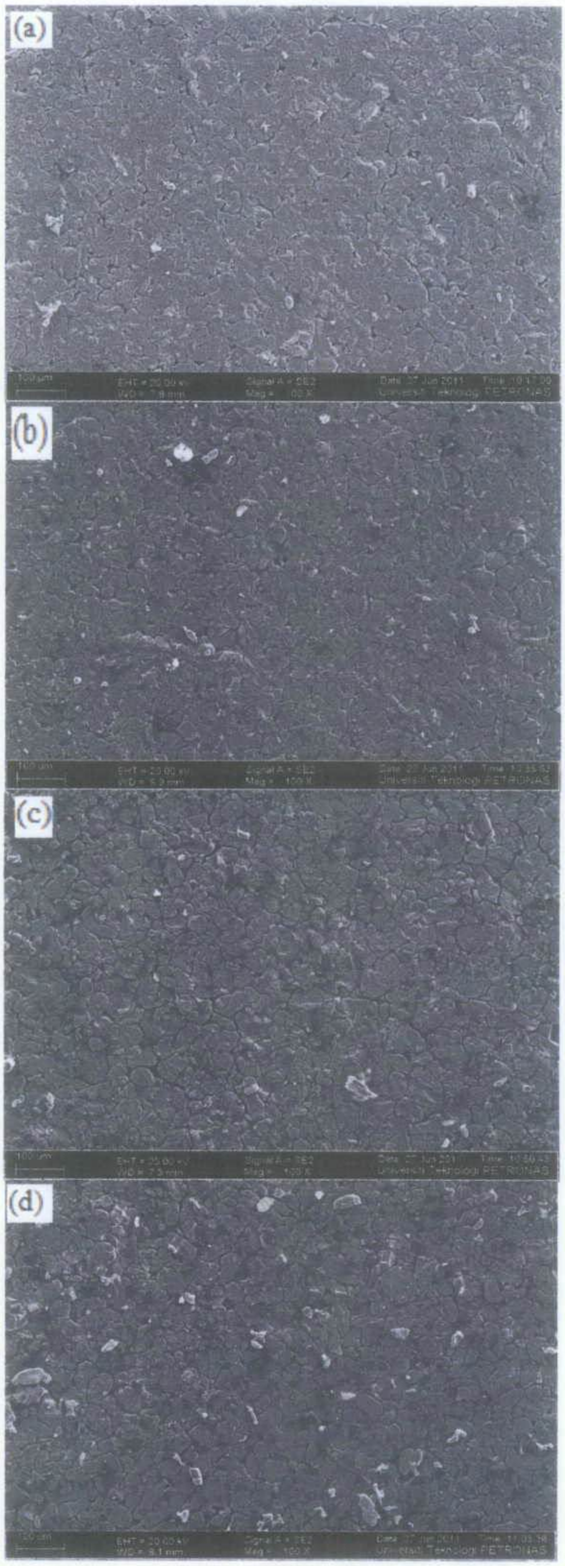


Figure 4.6: FESEM analysis of Al-CNTs with (a) 2 wt. % CNTs (b) 4 wt. % CNTs (c) 6 wt. % CNTs and (d) 8 wt. % CNTs

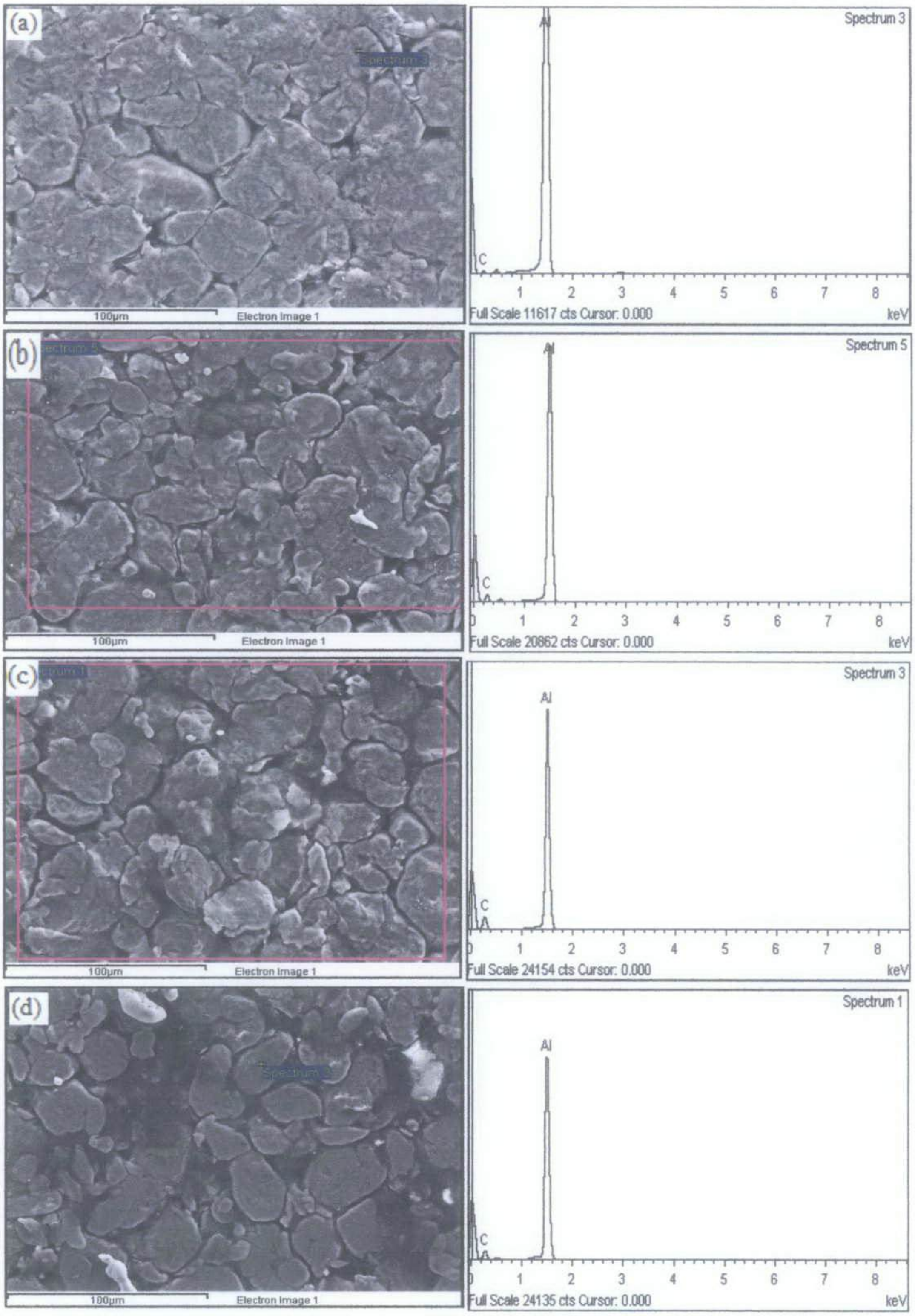


Figure 4.7: EDX analysis of Al-CNTs with (a) 2 wt. % CNTs (b) 4 wt. % CNTs (c) 6 wt. % CNTs and (d) 8 wt. % CNTs

Figure 4.8 shows the XRD pattern of pure aluminum, CNTs, 2 and 4 wt. % CNTs. From the result, it is observed that during the fabrication process of composition, there is no structural phase change occurs due to all peaks for composites are similar. It is notice that, CNTs is in amorphous phase and aluminum is crystalline phase. When one material is crystalline and another one is amorphous, it is very hard to see a bonding between them because no transfer or mutual sharing of electron between them. However, large amount of CNTs when mixed with aluminum lead to agglomeration and cause defects and affect the properties. Agglomeration is an irreversible reaction between fine particles to bond together. Increasing trend of CNTs in aluminum decreased the properties of composites because there is no pure bonding occurs between these materials. Up to 2 wt. % CNTs, it diffuses in aluminum. There is some weak bonding because some of the time CNTs show crystalline behavior and this is the reason why 2 wt. % CNTs showing better result from the pure aluminum. More addition of CNTs acts as amorphous behaviour and when there are a lot of them, difficult to mix with the crystalline of aluminum matrix.

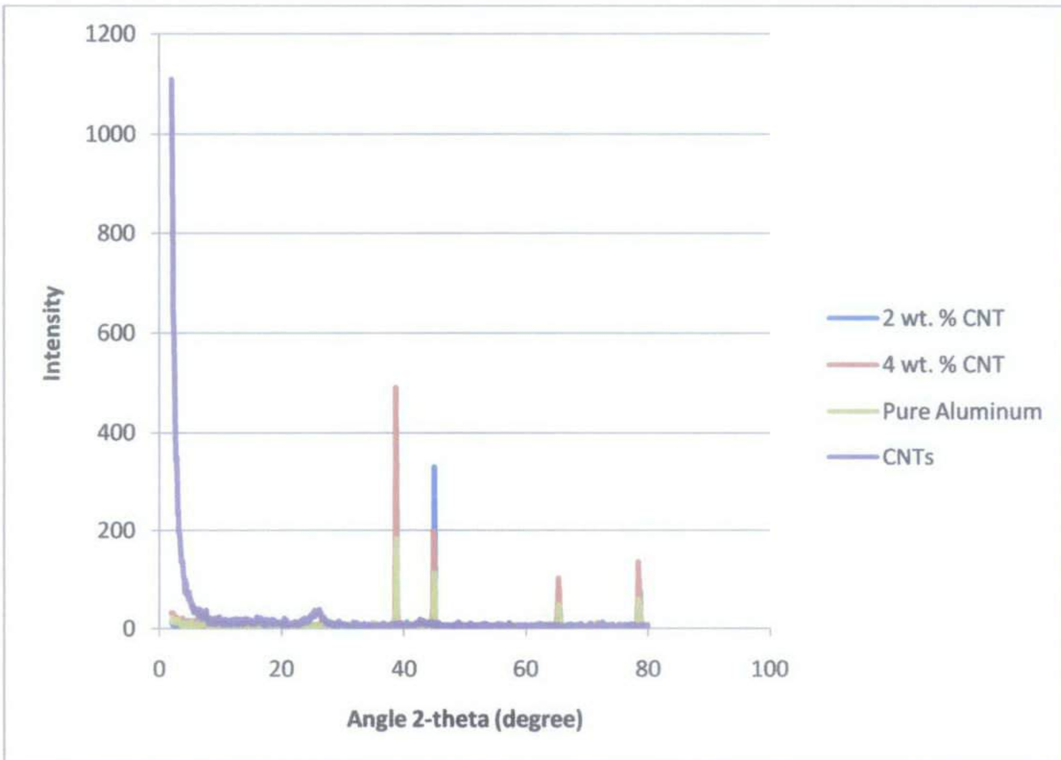


Figure 4.8: XRD analysis of Al, CNTs, 2 and 4 wt. % CNTs

4.2 Density

The density of aluminum is **2.70 g/cm³** and carbon nanotube is **1.4 g/cm³**. The calculation of the composition of powders in term of weight percentage can be referred below:

The formula for pallet volume:

$$V = \Pi r^2 H$$

$$V = \Pi(13/2)^2 x5$$

$$V = 663.66mm^3$$

<u>2 wt% CNT and 98 wt% Al</u>

Volume of Al $\rightarrow$ $0.98 \times 663.66 = 650.39mm^3$

Volume of CNT $\rightarrow$ $663.66 - 650.39 = 13.27mm^3$

$$Mass_{Al} = \rho \times volume$$

$$= 2.70g/cm^3 \times 650.39mm^3$$

$$= 1.76g$$

$$Mass_{CNT} = 1.4g/cm^3 \times 13.27mm^3$$

$$= 0.0186g$$

Then, $\rho_{mix} = mass/volume$

$$= (1.76 + 0.0186)/663.66$$

$$= 2.68g/cm^3$$

The theoretical density for 2wt% CNT and 98wt% Al is 2.68g/cm³.

The powders needed in gram:

$$Mass_{mix} = 2.68 \times 663.66$$

$$= 1.78g$$

$Mass_{Al} = 98/100 \times 1.78 = \underline{1.743g}$ $Mass_{CNT} = 2/100 \times 1.78 = \underline{0.0349g}$

Table 4.1 summarized the computation result of various compositions.

Table 4.1: Powder required in making sample

No.	CNT (wt %)	Mass of Al (g)	Mass of CNT (g)	Mass of Al X 3 pallets (g)	Mass of CNT X 3 pallets (g)
1	0	1.792	0	5.376	0
2	2	1.743	0.0349	5.229	0.1047
3	4	1.687	0.0703	5.061	0.2109
4	6	1.636	0.1044	4.908	0.3132
5	8	1.585	0.1378	4.755	0.4134
TOTAL				**25.329**	**1.0422**

Based on Table 4.1, total of aluminum powder needed is **25.329g** where else the carbon nanotubes is **1.0422g.**

Figure 4.9 shows the specimens in crucible after sintering process. The most right is pure aluminum followed by 2, 4, 6 and 8 wt% CNTs. Based on the observation, there are not much different in term of physical appearance of the aluminum composites. No reduction of volume was identified for all samples and the colours stay the same as before sintering.

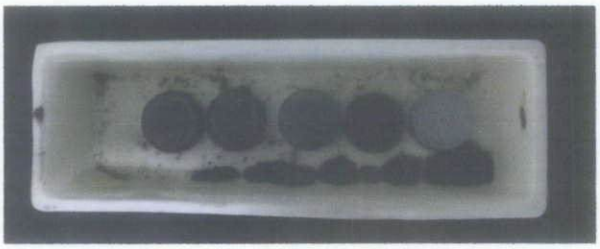


Figure 4.9: Specimens after sintering

The data obtained of green and sintered densities for composites are shown in Table 4.2.

Table 4.2: Measured data of green & sintered aluminum composites

CNT (Wt %)	Sample	Diameter (mm)	Thickness (mm)	Green Density (g/cm^3)	Sintered Density (g/cm^3)
0	1	13.04	4.61	2.347	2.490
	2	13.06	4.52	2.329	2.493
	3	13.04	4.82	2.477	2.521
Average				**2.384**	**2.501**
2	1	13.04	3.38	2.180	2.281
	2	13.04	4.30	2.292	2.399
	3	13.10	3.70	2.274	2.280
Average				**2.249**	**2.320**
4	1	13.04	4.40	2.151	2.215
	2	13.04	4.62	2.222	2.318
	3	13.10	3.92	2.159	2.216
Average				**2.177**	**2.249**
6	1	13.12	4.02	2.059	2.194
	2	13.20	4.12	2.009	2.162
	3	13.16	4.30	1.992	2.045
Average				**2.020**	**2.134**
8	1	13.54	4.20	1.841	1.897
	2	13.50	4.22	1.957	2.012
	3	13.42	4.40	1.914	1.944
Average				**1.904**	**1.951**

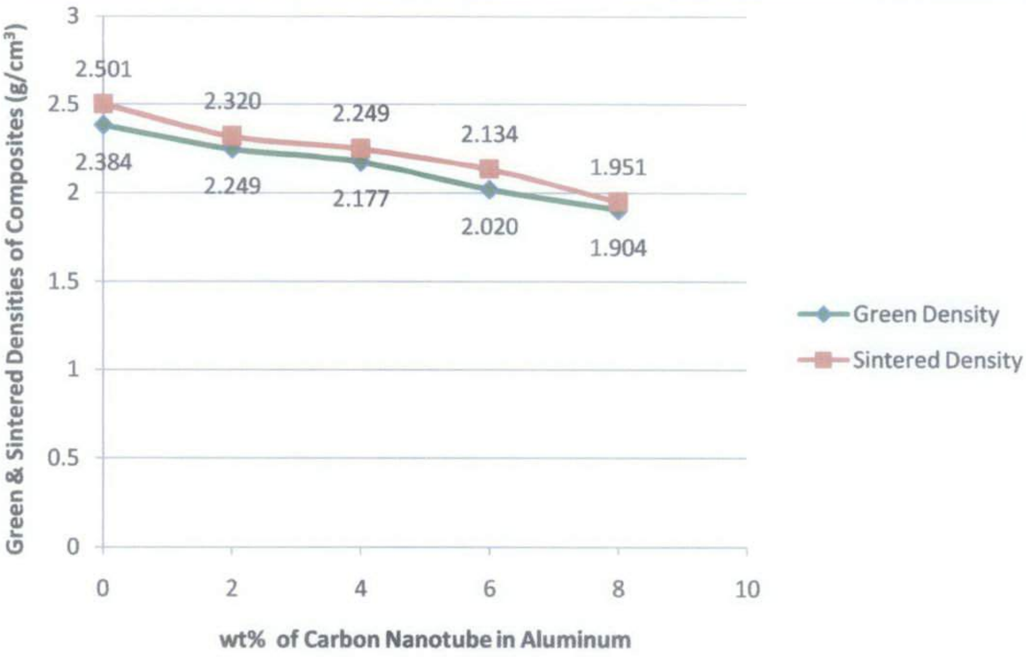


Figure 4.10: Green & Sintered Densities vs. Weight Percentage of CNTs in Al

Comparison to the data measured between green and sintered density, averagely the sintered density of aluminum composites slightly higher than the green compact density. Taking into account the data of 2wt% CNT, it is about three percent increment in density from **2.249g/cm^3** to **2.320g/cm^3**. Same result was obtained for the rest of the aluminum composites. As amount of carbon nanotube increasing in aluminum, it is resulting in decrement in green density from the pure aluminum value. After sintering, the result of reinforcement lead to the improvements of densities value compared to the green body densities.

Contradict to the matter happened, it is believed that porosity play a vital role in the result. After the samples have been sintered, the porosity of the specimens decrease and the effect of reinforcement in aluminum possibly reduce the mobility of aluminum grain boundaries. So, samples become packed and denser than before sintering and lead to the improvement of densities in the composite [8, 16]. CNTs reinforcement in aluminum matrix totally brings better density and it meets the objective of the project.

4.3 Hardness

Table 4.3 shows the result of hardness for specimens under the test load of 300 gf. The average of measured hardness from five different locations of the each specimen surface was considered the hardness of the specimen.

Table 4.3: Data obtained of hardness testing

		Weight Percentage of CNTs (wt%)				
	Reading	0	2	4	6	8
Hardness Strength (HV)	1	30.0	56.9	46.4	19.1	20.3
	2	31.3	69.2	41.2	20.0	18.1
	3	34.5	46.9	31.8	18.4	18.4
	4	26.5	48.0	24.7	24.1	19.0
	5	30.4	61.9	21.9	29.2	21.1
Average		**30.54**	**56.58**	**33.2**	**22.16**	**19.38**

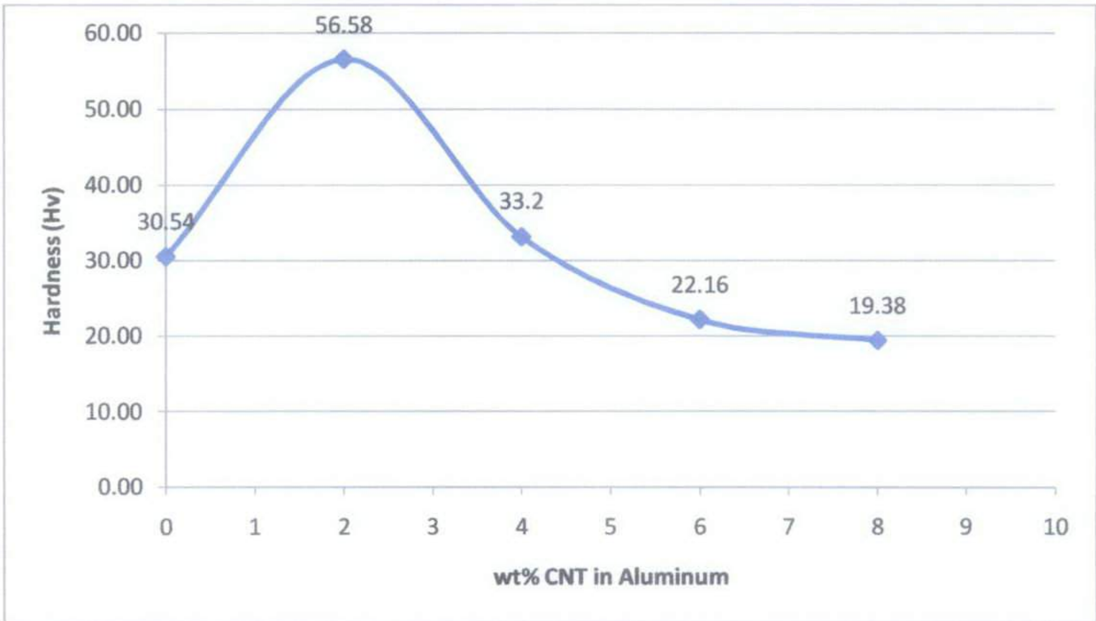


Figure 4.11: Hardness vs. Weight Percentage of CNTs in Al

It is shown that the Vickers hardness of the composites valued between 19 Hv to 57 Hv. The hardness strength result in Figure 4.5 shows that the addition of carbon nanotube enhances the properties of aluminum in certain amount of weight percentage. Taking into account, 2 wt% CNTs in aluminum shows that the hardness value is higher than pure aluminum and it is about 85% increment. Such behaviour is due to replacement of soft aluminum matrix by harder carbon nanotube particle thus forming a layer of reinforcement which occupies the porosities region between aluminum matrix.

The large aspect ratio of CNTs as reinforcement in aluminum used in the study was difficult to disperse at CNTs weight fraction greater than 2 [13]. Large amount of CNTs are prone to tangle together in blended powders of aluminum powders and carbon nanotubes [21]. The expected enhancement in mechanical properties with increase in CNTs weight content cannot fully realized. This is the reason why after 2 wt% CNTs in aluminum matrix, the hardness strength starts to decrease gradually as the amount of CNTs weight content increasing. If many of CNTs is added to the aluminum, the overload CNTs that remains after filling the microvoids form agglomerates with the aluminum particles. This agglomeration is the factor that causes defects [7, 16]. It can be concluded that the reinforcement of CNTs in aluminum really lead to the better result of hardness strength but in significant amount of weight percentage.

4.4 Wear Resistance Testing

Follow the standard as mentioned in Chapter 3, the result obtained based on the parameters applied for the test as below:

a. Speed: 350rpm
b. Time: 30sec
c. Test Load: 2kg
d. Temperature: 24°C
e. Counterface-1: Aluminum Matrix Reinforced CNTs
f. Counterface-2: Hardened Steel Disk

Table 4.4: Data obtained of wear resistance testing

CNT (wt%)	Weight before test (g)	Weight after test (g)	Weight Loss (g)
0	1.57959	1.44798	0.13161
2	1.37410	1.25226	0.12184
4	1.24171	1.09308	0.14863
6	1.13971	0.94086	0.19885
8	1.05137	0.81692	0.23445

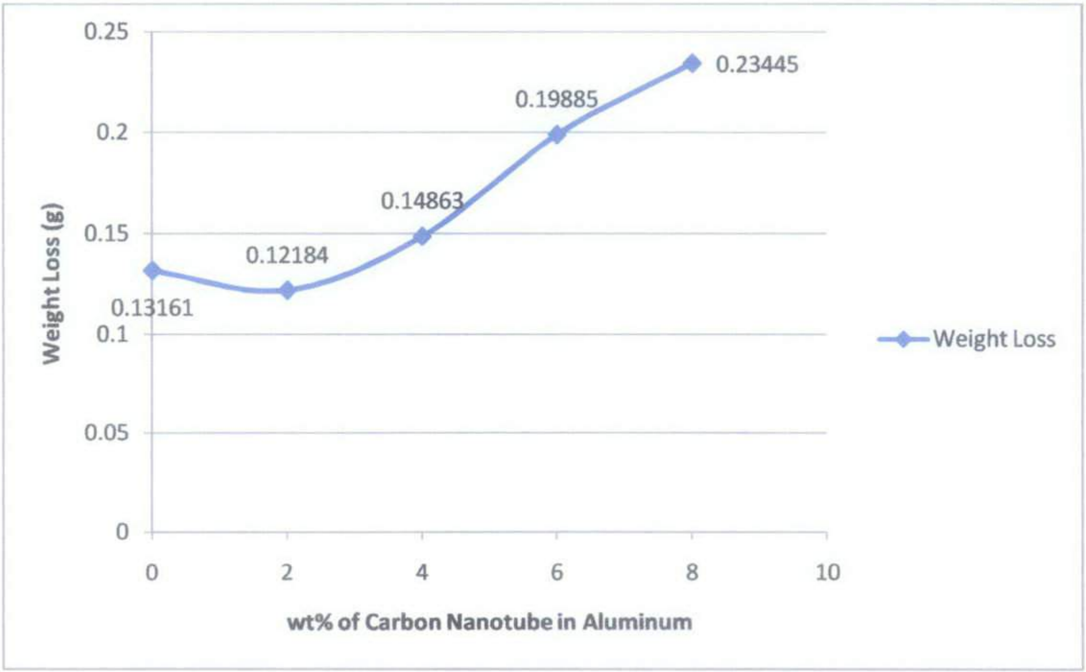


Figure 4.12: Relationship between weight loss of composites and CNTs content

5.2 Recommendations

Regardless the results of present study, some improvements in this area can be done to achieve better result from the current experiment. Powder Metallurgy method can be improved by selecting other options in pressing process. Instead of using cold compaction, it is recommended to apply hot compaction as both pressing and sintering were combined together to bring better result of samples. The continuation of this project in the future can be realized by modifying the parameters of powder metallurgy itself. For examples, the mixing process can be varied based on the investigation throughout time of blending in the ball mill. Besides, pressing pressure and sintering temperature also can be modified and study about the properties of materials with regards to the parameters.

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