Developing Carbon Particle Reinforced Aluminium Composite by Powder Metallurgy

By

Mohamad Najib Bin Mohtar

Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Mechanical Engineering)

JANUARY 2008

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CERTIFICATION OF APPROVAL

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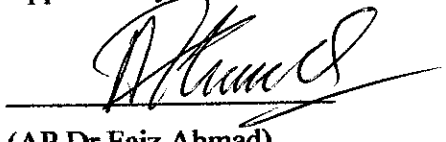
Approved by,



(AP Dr Faiz Ahmad)
Project Supervisor

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
January 2008

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.

MOHAMAD NAJIB BIN MOHTAR

ABSTRACT

Pure aluminium powder with average particle size of 25 μm was pressed at 580 MPa reaching a green density of 97.5% theoretical density to form a circular disk. Sintering was carried out under argon gas atmosphere at 620°C for 3 hours. The effect of sintering conditions (temperature, time, and atmosphere) was evaluated by cutting the disk and hardness was measured. SEM and EDX were used to study microstructural characterisation and particle content of the sintered materials. To improve the properties of pure aluminium disk, certain modification was made in the procedures. At this time, the aluminium powder was compacted with 464 MPa pressure to produce green density of 92% theoretical density. Sintering was done under nitrogen gas atmosphere for 15 minutes at temperature of 630°C. After sintering, the properties of sintered material were evaluated to compare with the first disk. The major different between these two samples is the compaction and sintering properties used to develop the end products. During cutting process, the second sample sintered in nitrogen showed higher hardness than the first sample. For composite compaction, three samples were prepared with 3%, 4% and 7% of carbon in term of volume percent to be added to aluminium powder with addition of wax as binder. By using 255 MPa compaction pressure, sample with 3% of carbon produced high strength green compact while there are some loose powder on the surface of another two samples. Sintering for the three samples was carried out under nitrogen atmosphere within two stages. Burn off process to remove wax at 350°C for one hour and sintering at maximum temperature of 620°C for one hour. The properties of composite material was evaluated using hardness test and to be compared to properties of pure aluminium compact. The hardness of composite material is less than pure aluminium compact and it is suitable to use as self-lubricating material due to the present of carbon.

ACKNOWLEDGEMENTS

I would like to take the opportunity to express my utmost gratitude to the individual that have taken the time and effort to assist me in completing the project. Without the cooperation of these individuals, no doubt I would have faced some minor complications through out the course.

First and foremost, I would like to thank my supervisor, AP Dr. Faiz Ahmad, who shared with me a lot of his expertise and research insight. Without his guidance and patience, I would not be succeeded to complete the project. I also like to express my gratitude to the Final Year Project Coordinator that have coordinated and made the necessary arrangements, especially in terms of the logistics, for this study.

I would also like to thank the technicians in Mechanical and Chemical Engineering Department especially Mr. Anuar and Mr. Yusuf for the support in terms of the preparation for experiments and guidance for this study. They continuous support and help throughout the whole period of project are very much appreciated.

I cannot finish without saying how grateful I am with my family, on whose constant encouragement and love I have relied throughout my time at the university. To all individuals that has helped me in any way but whose name is not mentioned here, I would like to thank you all.

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CHAPTER 1

1.0 INTRODUCTION

Powder metallurgy, or P/M, is a process for forming metal parts by heating compacted metal powders to just below their melting points. Although this technique has existed for more than 100 years, it has become widely used to produce high quality parts for a variety of applications. It is used to make great variety of part such as gear, cam, casing, bushing and much more. This part is used in automobiles, appliances, computer, electrical and magnetic devices, cameras, power tools and aircraft components. Despite of introduction of many others processing techniques, powder metallurgy is still being used until today because of the advantages that other techniques do not have. In this project, two type of materials are selected which are carbon particle and aluminium powder to create a reinforced composite material.

The advantage of powder metallurgy is design flexibility where the part can be produced at high density or maximum structural performance or to control porosity for application such as filters and self lubricating bearing. Another advantage is the ability to produce part that is close to final shape. This is called near net shape forming. The benefits are the reduction or elimination of secondary manufacturing operation conserving metal and reducing cost.

1.1 Background of the Project

The scientific investigation and applied research on composite materials can date back to the 1940's [1] with the advantages behind the development of metal matrix composites being the capability to combine phases providing a potential for changing material properties to meet specific and challenging requirements. Composites offer an approach for producing "designer" materials used to provide specific types of material behavior, such as their improved strength and stiffness, outstanding corrosion resistance, friction resistance and wear resistance, high electrical and thermal conductivity, and high temperature mechanical behavior [2].

Metal matrix composites (MMCs) are structured engineering materials in which reinforcement, usually a hard ceramic component, is homogenously dispersed in a ductile metal matrix in order to obtain properties that are altered compared to those of the conventional monolithic metallic matrix or alloy.

More recently, the automotive and electronic industries have been working extensively with these composites to increase cost savings, enhance performance and evolve emerging applications. In order to obtain the desired properties of a metal matrix composite, certain variables should be evaluated for optimum results including the choice of the matrix, the type and degree of the reinforcement, and the composite processing method.

1.2 Problem Statement

Powder metallurgy is widely used each with specialization in alloys, applications, and production techniques. The largest activity is associated with ferrous alloys, some iron and many steels. Ferrous alloys constitute the dominant powders and structural automotive components constitute the dominant applications. Attainment of the desired mechanical properties requires alloying to from high-strength steels during sintering. The combination of low production costs and high sintered strength dominates the powder metallurgy selection criteria [3].

The selection of carbon particle and aluminium as primary material in this project is crucial since carbon is a non-metal element. It is important to know the behaviour of a powder during the subsequent consolidation processes determined by both particle and bulk properties. A powder is characterized not only by chemical composition but also by particle shape, size and surface chemistry and in bulk by compressibility and apparent density [4]. When the carbon and aluminium are compacted together, the properties of that material such as density and melting temperature are different from the original properties. So that, sintering process will give more challenge to determine the furnace environment and furthermore aluminium is difficult in sintering.

Therefore, it is a must for the project to be done in order to obtain maximum data and information on behaviour of sintered component using these materials for further improvement.

1.3 Objectives and Scope of Study

The main objective of this project is to develop carbon particle reinforced aluminium composite by using powder metallurgy. The fabrication of metal matrix composites is focused on the combining of the reinforcements into the metallic phase, with the goal of manufacturing a new material free of defects such as pore. At the end of the project, it is expected to come out with a sintered part using these materials. Further than that, it is valuable to know and record the properties and behaviour of this composite material during green state and after sintering process. So, improvement can be made by changing the mixing ratio of composite, furnace environment and other ways.

The scope of studies involve of the method of powder metallurgy, properties of carbon particle and aluminium, material characterization such as particle size distribution and microstructure, sintering process and powder part production.

CHAPTER 2

2.0 LITERATURE REVIEW/THEORY

2.1 Literature Review

Powder metallurgy is a processing technique that consists of three major processing stages. First, the primary material is physically powdered, divided into many small individual particles. Next, the powder is poured into a mold or passed through a die to produce a weakly cohesive structure very near the dimensions of the object ultimately to be manufactured. Finally, the end part is formed by applying pressure, high temperature, long setting times or any combination thereof. The process of powder metallurgy can be depicted in the **Figure 1** below:

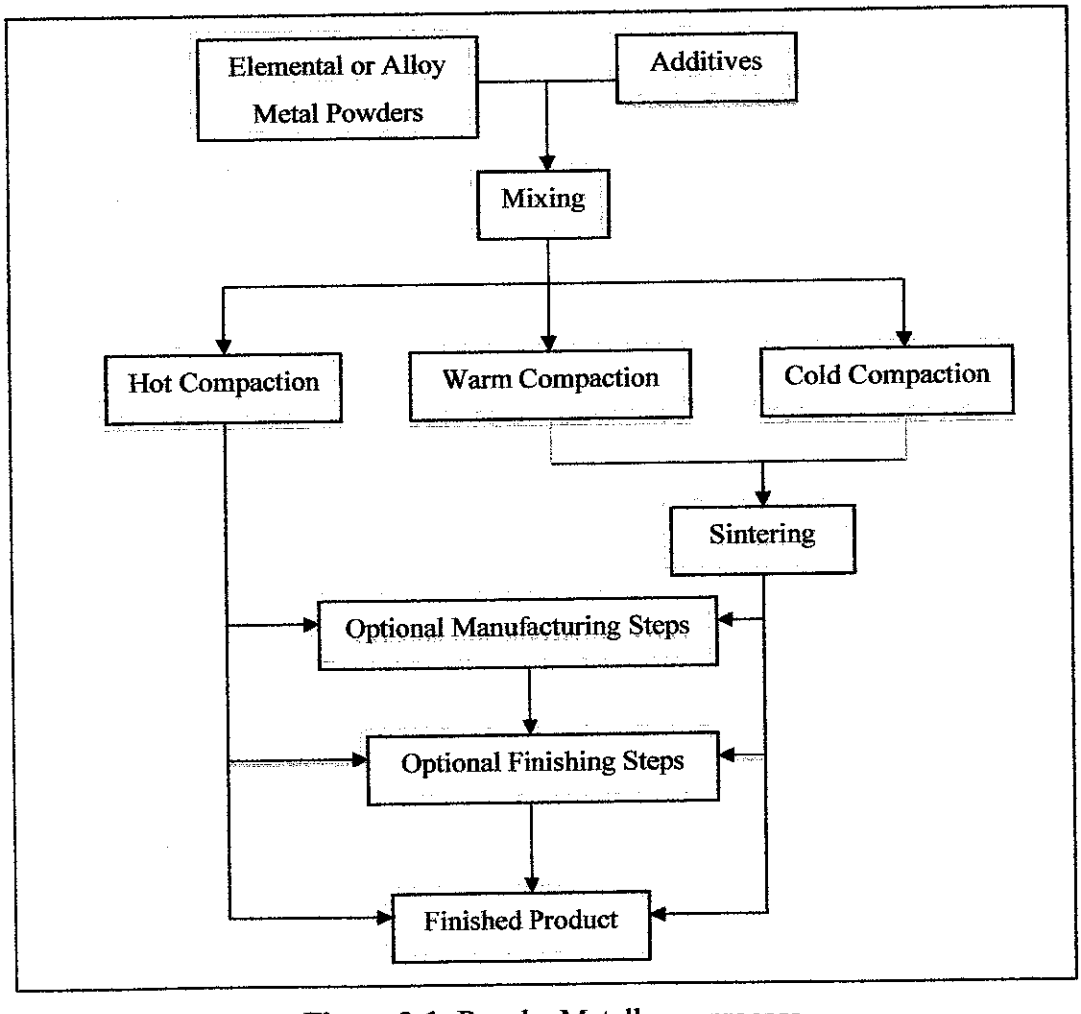


Figure 2-1: Powder Metallurgy process

2.1.1 Metal Powder Production

The first step in the overall PM process is making metal powders. There are four main processes used in powder production: solid-state reduction, atomization, electrolysis, and chemical.

1. Solid-state reduction

In solid-state reduction, selected ore is crushed, mixed with a reducing species (e.g., carbon), and passed through a continuous furnace. In the furnace, a reaction takes place that leaves a cake of sponge metal which is then crushed, separated from all non-metallic material, and sieved to produce powder. Since no refining operation is involved, the purity of the powder is dependent on the purity of the raw materials. The irregular sponge-like particles are soft, readily compressible, and give compacts of good pre-sinter ("green") strength.

2. Atomization

In this process, molten metal is separated into small droplets and frozen rapidly before the drops come into contact with each other or with a solid surface. It involved breaking up the steam of molten metal with a jet of high pressure water or inert gas such as nitrogen or argon resulting droplet solidified and settle as powder particles at the bottom of the tank. In principle, the technique is applicable to all metals that can be melted and is used commercially for the production of iron; copper; alloy steels; brass; bronze; low-melting-point metals such as aluminium, tin, lead, zinc, and cadmium; and, in selected instances, tungsten, titanium, rhenium, and other high-melting-point materials.

3. Electrolysis

By choosing suitable conditions, such as electrolyte composition and concentration, temperature, and current density, many metals can be deposited in a spongy or powdery state. Further processing–washing, drying, reducing, annealing, and crushing–is often required, ultimately yielding high-purity and high-density powders. Copper is the primary metal produced by electrolysis but iron, chromium, and magnesium powders are also produced this way. Due to its associated high energy costs, electrolysis is generally limited to high-value powders such as high-conductivity copper powders.

4. Chemical

The most common chemical powder treatments involve oxide reduction, precipitation from solutions, and thermal decomposition. The powders produced can have a great variation in properties and yet have closely controlled particle size and shape. Oxide-reduced powders are often characterized as "spongy," due to pores present within individual particles. Solution-precipitated powders can provide narrow particle size distributions and high purity. Thermal decomposition is most often used to process carbonyls. These powders, once milled and annealed, exceed 99.5 percent purity.

2.1.2 Powder Metal Part Production

Metal powder used for part production can be a combination of various elemental particle or pre-alloy powder. If elemental powder is used, each of the ingredients that will comprise the desired part is mixed in proper portion into a uniform blend. With pre-alloy powder, the proper proportion of the ingredient is already pressed in each particle. In either case, additive such as binder and lubricant are usually added.

The powder is then consolidated in mold or dies shaping into a compact of the desired part. At this point, the part or compact is in green state where the powder particles are lively joined together. In this state, the part is called green strength which is usually only sufficient for handling purposes. The common method of consolidating and shaping metal powder for part production includes:

- Hot compaction: Isostatic, extrusion, die compacting, spraying.
- Warm compaction: Die compacting, injection molding.
- Cold compaction: Die compacting, isostatic, rolling.

The part is then transferred into sintering process. During sintering, parts are heated below the melting temperature point but high enough to metallurgically bond the individual particle. Sintering is further densifying the part and increasing strength. Final part density is extremely important. Although control porosity is required and achievable for certain part, the performance of structural part increases directly with increasing density.

2.2 Metal Matrix Composites (MMCs)

The interest in MMCs is due to their generally superior mechanical properties compared to typical alloys. Composite materials generally consist of two parts. The major part or bulk of a composite is the matrix, while the second part consists of a reinforcement material. In this project, the matrix is aluminium and the reinforcement material is carbon particle. This material is chemically different from the matrix and there exists a distinct interface between the two materials. They should also have properties that are unattainable by any of the individual constituents. What makes composite materials attractive is the possibility to be able to tailor the properties according to the needs of a specific design. MMCs are fabricated to take advantage of the properties of all the materials used in making the composite.

Metal matrix composites have certain advantages over the more widely used polymer matrix composites because MMCs usually exhibit higher strength, toughness, elastic modulus, higher thermal and electrical conductivity and are more stable at higher temperatures. Advantages over the unreinforced metal include higher strength to weight ratios, increased wear resistance, and higher hardness [1]. The potential applications for these materials can be easily recognized by the significant increase in properties (table 2.1). These properties include high modulus and strength, high strength to weight ratios, higher stiffness-to-density ratios and wear resistance

Table 2.1: Comparison between properties of a conventional Al alloy and a MMC with the same Al alloy as the matrix [1]

Material	Tensile Strength (MPa)	Abrasive Resistance (Volume loss, mm^3)	Wear Resistance (Volume loss, mm^3)
A356-T6	228	0.575	0.18
A359/SiC/20p-T6	340	0.202	0.023

2.2.1 Theory

It was mentioned in the introduction that there are certain advantages of MMCs over the monolith, among them is enhanced strength. The increased strength noted in the composites is a result of microstructural differences brought about by the introduction of reinforcement. Dai et al. and Sarkar have reported that in a more general sense, there are two divisions of strengthening: direct (load transfer) and indirect (matrix strengthening) [7] [8]. Those discussed by Lloyd and Chawla are considered indirect strengthening mechanisms whereas load transfer is considered direct [5] [6]. Sarkar states that the distinction between transfer and matrix strengthening is that transfer (direct) theories consider shape and volume fraction of particle reinforcement and matrix strengthening considers particle size and volume fraction. A combination of all this factors usually contributes to the strengthening of the composite [8].

It is widely accepted that a major strengthening mechanism in metals is due to an increase in dislocation density. Strength can be correlated to the ease or difficulty of dislocation movement. As dislocations meet effective barriers, which hinders their motion, strength is increased. Many investigations have demonstrated a correlation between strength of a material and dislocation density. Likewise, strengthening through a reduction in grain and sub grain or cell size has also been demonstrated. Ma and Tjong have stated that a reduction in size of the particulate reinforcement increases the strength of the composite possibly related to the smaller spacing between particles for a given volume fraction [9].

A good correlation between microstructural changes (increasing dislocation density and reduction of subgrain size) in the matrix and the changes in yield stress of MMCs has been observed by Arsenault et al. in Al/Sic composites produced by powder metallurgy process. The data obtained indicates the dislocation density increases with an increase in volume fraction, and the density decreases with particle size [10]. MMCs offer significant improvements in mechanical properties over their monolithic counterparts. These improvements are highly dependent on (a) the ability to transfer stresses from the matrix to the reinforcing materials, (b) the volume fraction, size, and distribution of reinforcements, (c) enhanced dislocation density

and interactions, (d) precipitation in the matrix and at interfaces, and (e) the overall strengthening of each individual component of the composite [2].

CHAPTER 3

3.0 METHODOLOGY/PROJECT WORK

3.1 Powder Particles Preparation

In this project, there are two types of material used which are carbon and aluminium. Because of there is no material specification provided, several tests need to be performed to determine the particle size and shape of the powders. These tests can be done by using Particle Size Analyzer and Variable Pressure Scanning Electron Microscope (VPSEM).

3.1.1 Particle Size Analysis

Particle size analysis is performed by using Mastersizer® 2000 with Scirocco dry powder dispersion unit. This analysis is carried out by laser diffraction technique. Measurement of powder particle size distribution is by dry dispersion or suspension in an appropriate liquid. The technique uses the scattering of light, delivered from a laser that is passed through a chamber containing the particles in suspension. The scattered light is detected by a photo-detector array. The intensity of light on each detector is then converted into a particle size distribution plot that is calculated by mathematical algorithm. It is an elegant, simple, fast and flexible technique that produces high quality data.

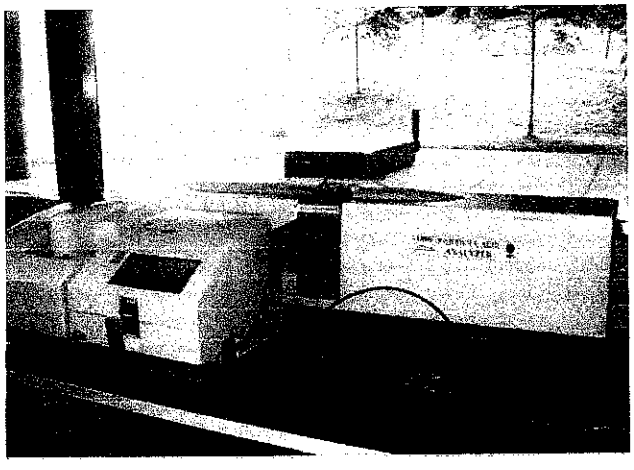


Figure 3-1: Mastersizer® 2000 with Scirocco dry powder dispersion unit

3.1.2 Microstructure Observation

The scanning electron microscope (SEM) is a type of electron microscope capable of producing high-resolution images of a sample surface. Due to the manner in which the image is created, SEM images have a characteristic three-dimensional appearance and are useful for judging the surface structure of the sample.

In a typical SEM, electrons are thermionically emitted from a tungsten or lanthanum hexaboride (LaB6) cathode and are accelerated towards an anode; alternatively, electrons can be emitted via field emission (FE). The beam passes through pairs of scanning coils or pairs of deflector plates in the electron optical column, typically in the objective lens, which deflect the beam horizontally and vertically so that it scans in a raster fashion over a rectangular area of the sample surface. When the primary electron beam interacts with the sample, the electrons lose energy by repeated scattering and absorption within a teardrop-shaped volume of the specimen known as the interaction volume, which extends from less than 100 nm to around 5 μm into the surface. The energy exchange between the electron beam and the sample results in the emission of electrons and electromagnetic radiation, which can be detected to produce an image, as described below.

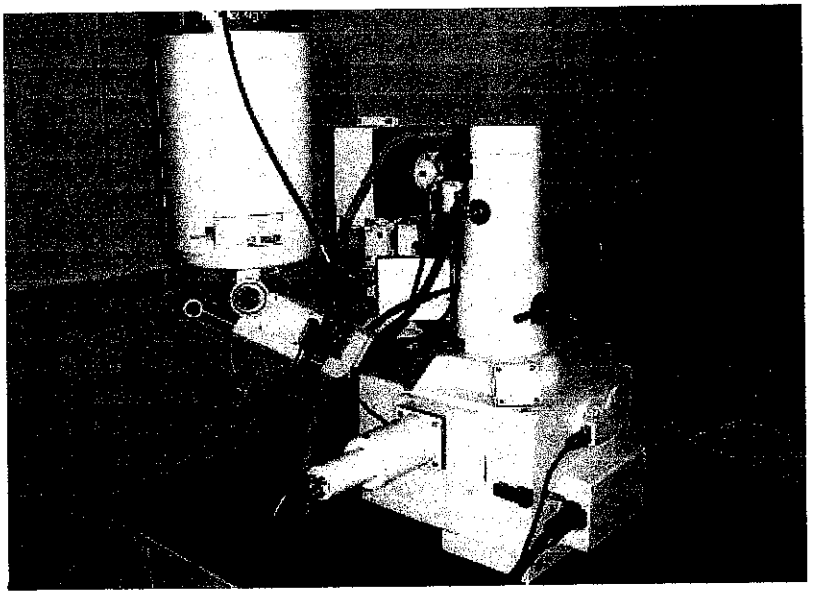


Figure 3-2: Scanning Electron Microscope (SEM)

3.2 Pure Aluminium Properties Analysis

Before composite powders mixing, it is important to test the properties and condition of pure aluminium during compaction and sintering process. This step will determine the properties and condition that will be used in during composite powder mixing.

3.2.2 Aluminium Powder Compaction

Certain amount of pure aluminium powder is pressed using precision metal dies to yield a green compact in a cylindrical disk shape without using any binder. Certain amount of compaction pressure is applied to produce a green compact with 90 – 95% density. Typical green strengths range from 450 to 1500 psi is sufficiently strong to withstand normal handling without chipping or breaking.

3.2.3 Sintering of Aluminium Compact

After produce an aluminium compact, the compact is sintered in a controlled atmosphere furnace slightly below aluminium melting temperature. This process metallurgically bonds the powder particles and develops the desired physical and mechanical properties. Aluminium powder sintering is difficult to achieve because the aluminium oxide layer is not reduced by common furnace atmospheres at sintering temperatures. Selection of furnace atmosphere, sintering time and temperature is crucially important in successful sintering process.

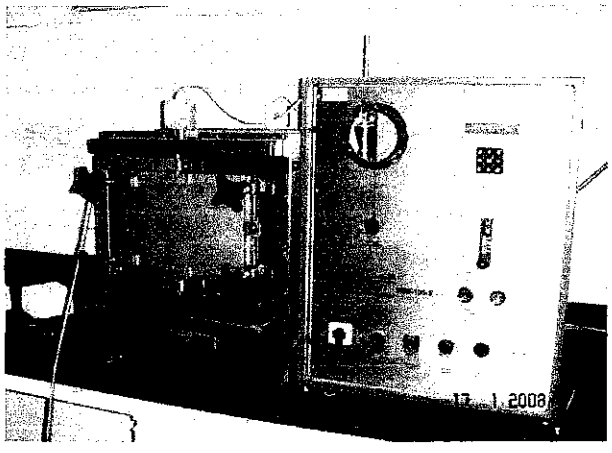


Figure 3-3: Sintering Furnace

3.3 Composite Powders Mixing

After sintering of pure aluminium is succeed, both aluminium and carbon powders can be mixed with certain proportion. This is the most important factors in determining the properties of composite relative to proportions of the matrix and reinforcing materials. In this project, 2%, 3% and 5% of carbon powder are mixed with the remaining percentage of aluminium powder. The mixing of each composite is accomplished in glass containers using spatula for at least 15 – 20 minutes to assure the particles mixing and dispersion.

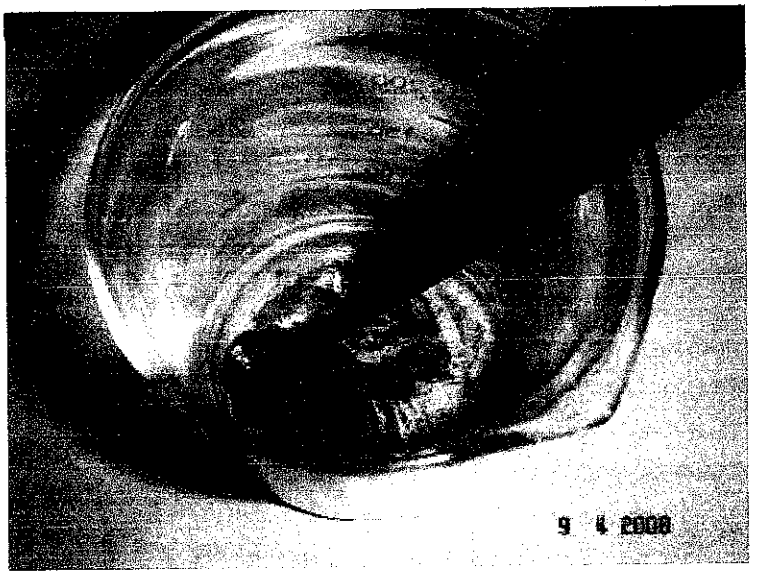


Figure 3-4: Mixing of composites

3.4 Composite Compaction

After determine the amount of each composite, the powders are then compacted by using mechanical pressing to produce a green compact. Through physical experimentation with the aluminum powder it has been determined that aluminium premixes exhibit excellent compressibility and yield high density parts at low compaction and ejection pressure.

3.5 Sintering Process

The green compact is then transferred into sintering furnace. During sintering, green compact are heated below the melting temperature point but high enough to metallurgically bond the individual particle. Sintering is carried out under nitrogen within a temperature range between 550 and 650°C in certain period of time. The effect of the sintering conditions such as temperature, time and atmosphere are evaluated by measuring density and hardness [11].

3.6 Tools and equipments

The tools and equipment which are required in this Final Year Project is mechanical pressing tool, sintering furnace, particle analyzer, scanning electron microscope (SEM), Windows based PC together with the programs such as Microsoft Office and equipment needed basically would be data from lab as well as from the internet and other references.

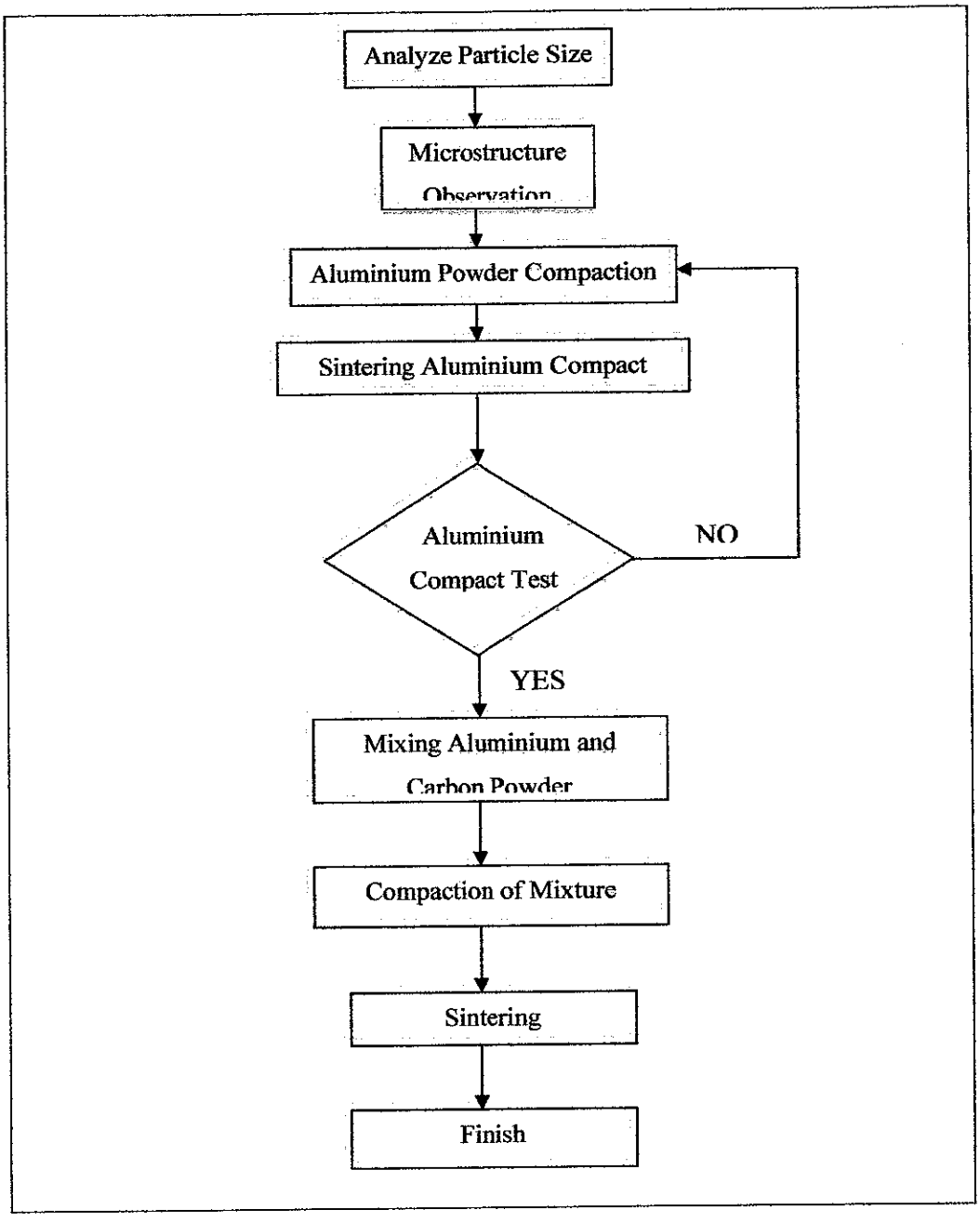


Figure 3-5: Flow of Project Work

CHAPTER 4

4.0 RESULT AND DISCUSSION

4.1 Volume and Weight Fraction Calculation

Table 4-1: Properties of Carbon and Aluminium

Property	Carbon	Aluminium
1. Molecular weight	12	26.98
2. Density, ρ (g/cm^3)	1.75	2.698
3. Melting point (°C)	3550	660.97
4. Tensile strength (MPa)	300	90

Assumption:

1. Volume percent of materials used:

   97% of aluminium and 3% of carbon

- Composite density: $\rho_c = \rho_f V_f + \rho_m V_m$

$$= 1.75(0.03) + 2.698(0.97)$$
$$= 2.67 g/cm^3$$

2. Assume the final volume of composite is $V_c = 1\ cm^3$.

$$v_f = 0.03\ (1) = 0.03\ cm^3$$

$$v_m = 0.97(1) = 0.97 cm^3$$

$$\Rightarrow w = \rho v$$

$$\therefore w_f = \rho_f v_f$$

$$= 1.75(0.03) = 0.0525 g$$

$$\therefore w_m = \rho_m v_m$$

$$= 2.698(0.97) = 0.2617 g$$

4.2 Particle Size Distribution

The average size of carbon particle for first test is 1018 μm but this size is quite large for powder metallurgy process. To reduce particle size, the carbon powder was grinded using Mortar Grinder. Then, the powder was sent back to do second test and the result is shown in **Table 4.2.**

Table 4-2: Particle size for carbon powder

Test	Particle Size (μm)
1	1018
2	5.221

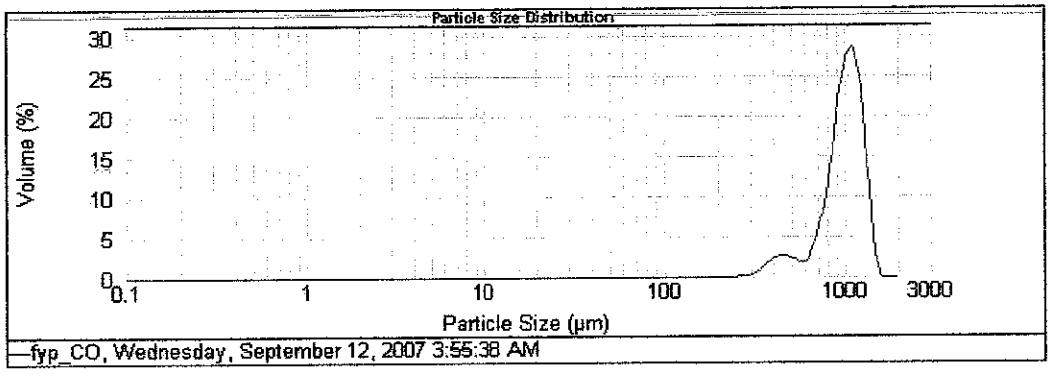


Figure 4-1: Carbon particle size distribution before grinding

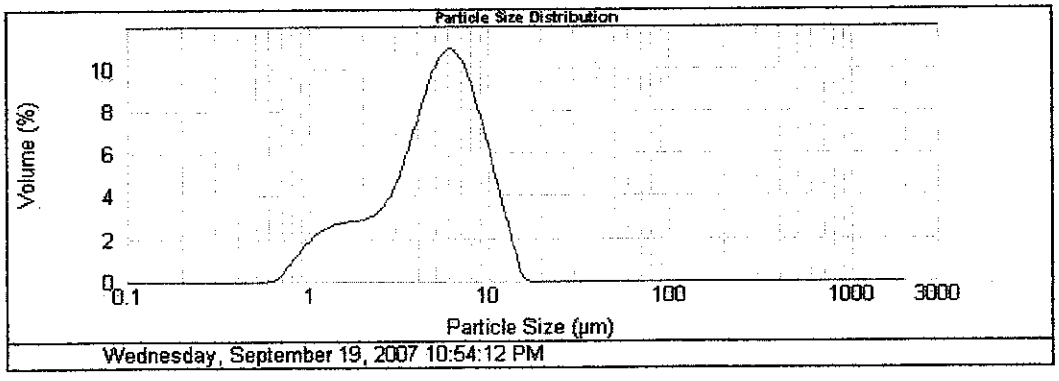


Figure 4-2: Carbon particle size distribution after grinding

For aluminium powder, the average particle size is 25.651 μm. This size is smaller compare to normal particle size for powder metallurgy process which is at ~150 μm [3] and there is no further grinding process needed for this powder. The particle size distribution of aluminium powder is shown in **Figure 4-3.**

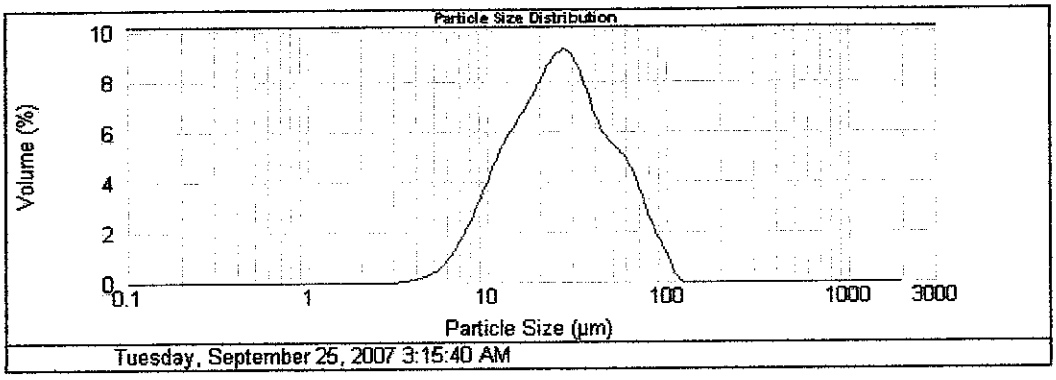
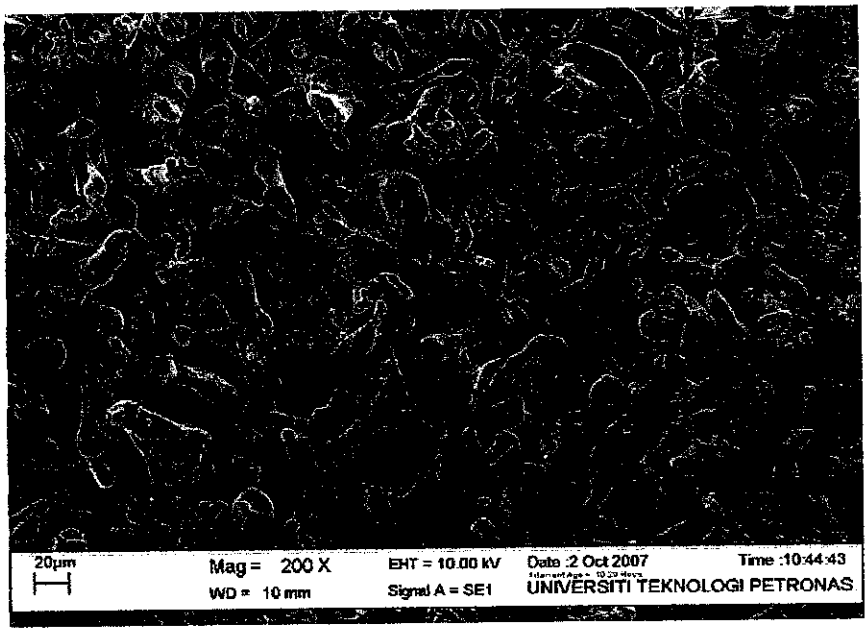


Figure 4-3: Particle size distribution for aluminium.

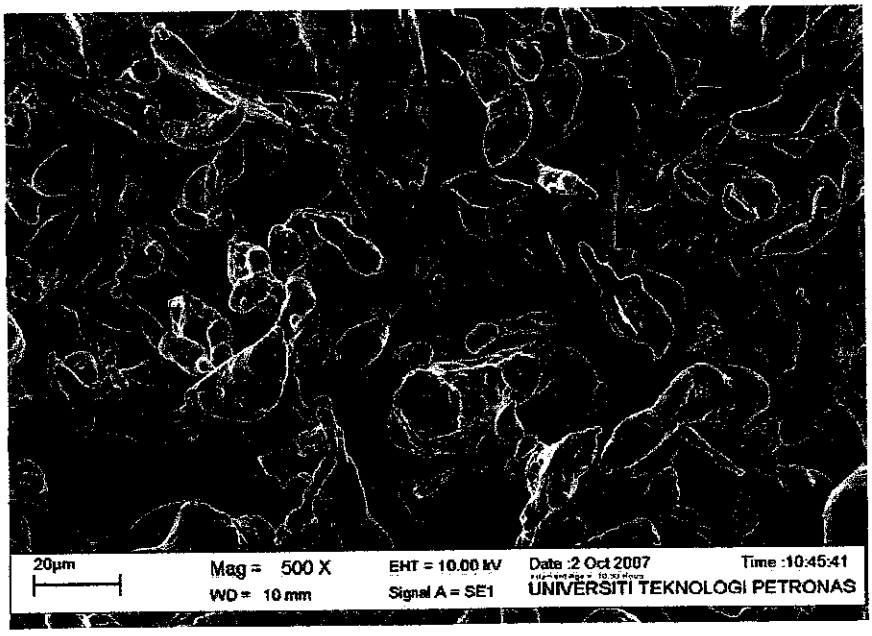
From the results above, it shows that the particle size of carbon powder is greatly reduced after grinding process. Actually before grinding process, the carbon powder exists as small particles but it merges with each other to form bigger particle size due to long storage time. Then, by applying small force with 5 minutes grinding time, the size of carbon particles reduces significantly from 1018 μm to 5.221 μm.

4.3 Microstructure Observation

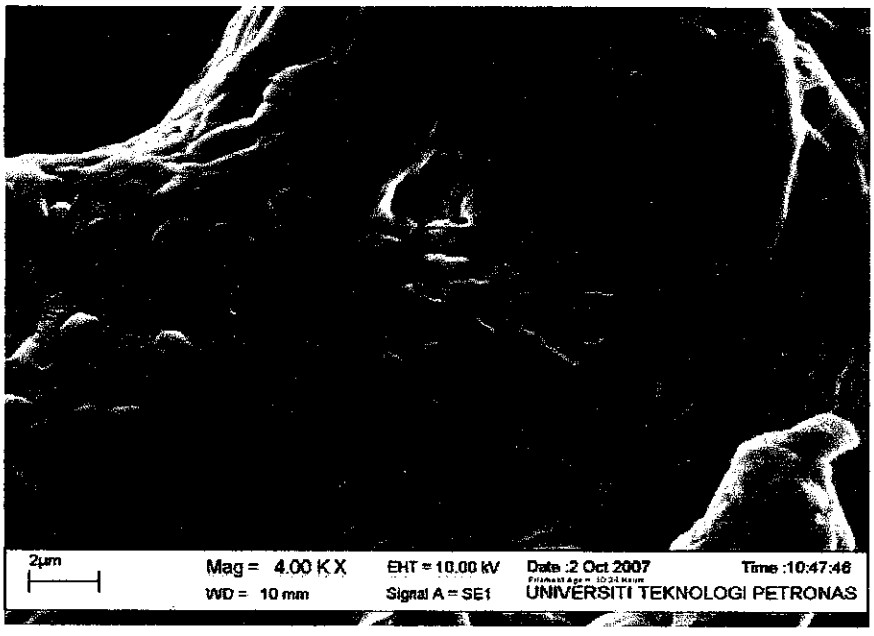
Microstructure observations for both powders were conducted using Variable Pressure Scanning Electron Microscope (VPSEM). From the observations, three kinds of microstructure images were captured using various magnification values. The results are shown in figures below.



a) 200 X Magnification

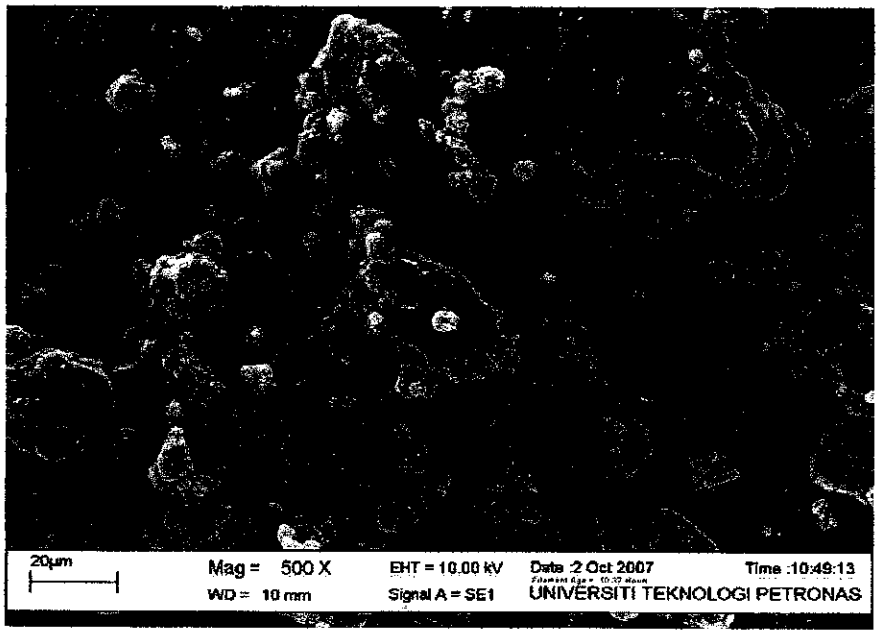


b) 500 X Magnification

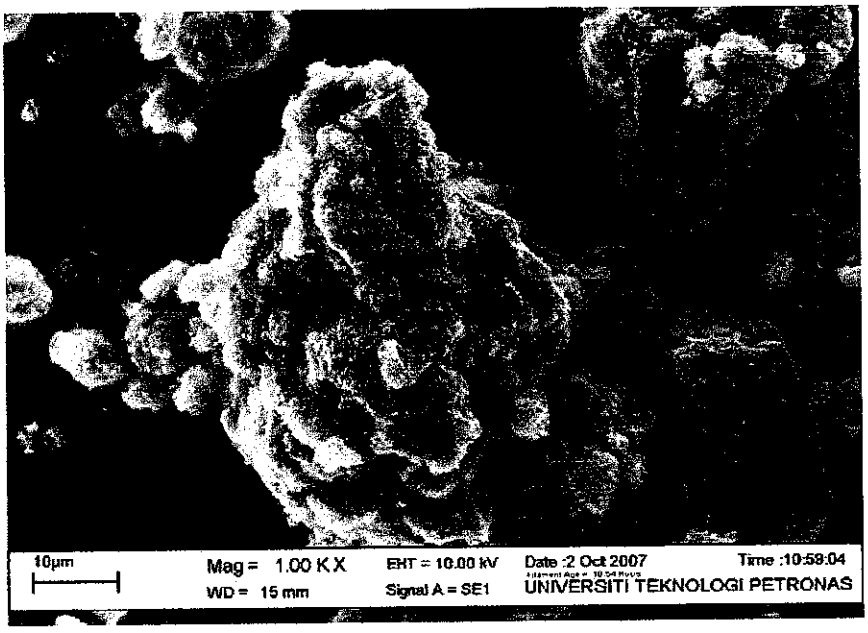


c) 4000 X Magnification

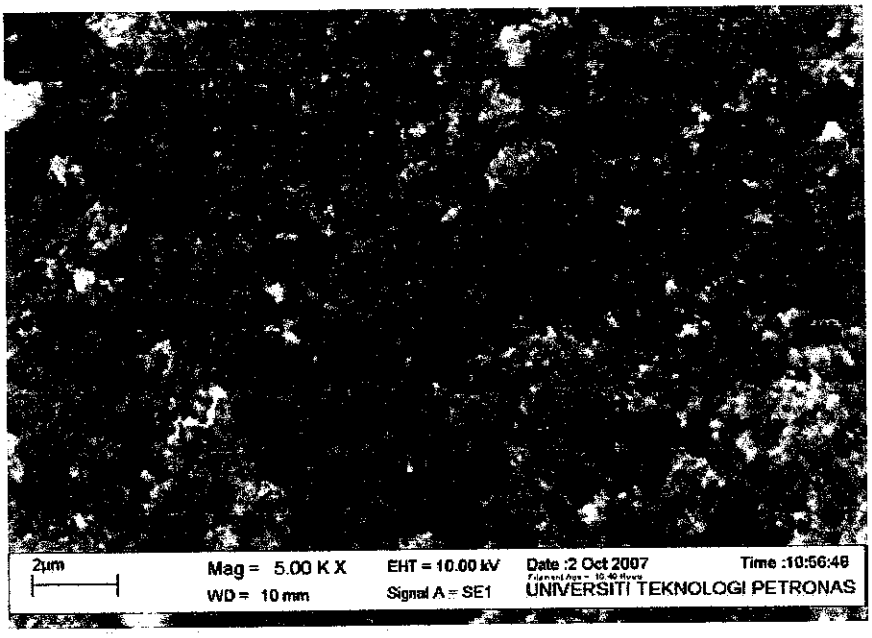
Figure 4-4 (a) (b) (c): Morphology of Aluminium particle



a) 500 X Magnification



b) 1000 X Magnification



c) 5000 X Magnification

Figure 4-5 (a) (b) (c): Morphology of Carbon particle

From **Figure 4-4 (a), (b) and (c),** it shows that the aluminium powder particles are granules rounded and irregular in shape. The possible method of producing this powder is water atomization. If using gas atomization, the shape of powder particle tends to be spherical.

4.4 Pure Aluminium Powder Compaction

Aluminium powder was compacted by using Autopallet Press Machine. Two samples of aluminium disk are produced in this project. **Table 4-3** below shows the properties of aluminium during compaction.

Table 4-3: Properties of aluminium during compaction

	Sample 1	**Sample 2**
Weight of powder	1.5622 g	1.360 g
Pressure applied	580 MPa	464 MPa
Diameter of disk	1.27 cm	1.27 cm
Height of disk	0.47 cm	0.41 cm
Theoretical density of Al	2.698 g/cm^3	2.698 g/cm^3
Measured density	2.624 g/cm^3	2.618 g/cm^3
Percent of theoretical density	97.25%	92 %

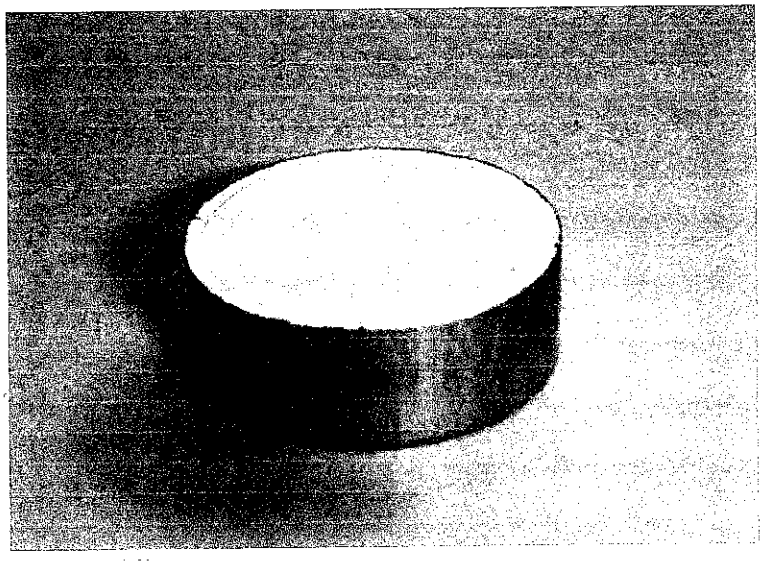


Figure 4-6: Green compact of aluminium

Based on the result, it shows that the density of green compact for **Sample 1** is about 97% of theoretical density of aluminium. This value is slightly higher than other people's work which is about 90-95% theoretical density. What I observed that the green compact was not perfectly in cylinder shape. So, it was very hard to determine the volume of the green compact exactly. By the way, the green compact was in good shape and strong enough to withstand normal handling without chipping and breaking as in **Figure 4-6.**

For the second sample, the weight of aluminium powder was reduced to form a smaller green compact. The pressure applied to form the compact was also reduced from 580 MPa to 464 MPa. As result, the second sample has theoretical density about 92% which is in range of other people's work.

4.5 Sintering of Aluminium Compact

After successful compaction process, the green compact was transferred to sintering furnace. The properties of compact and furnace atmosphere for both samples are as the following table.

Table 4-4: Sintering properties

	Sample 1	**Sample 2**
Initial weight of compact, m_1	1.5622 g	1.3600 g
Final weight of compact, m_2	1.5626 g	1.3653 g
Furnace atmosphere	Argon gas	Nitrogen gas
Sintering time	3 hours	15 minutes
Maximum Sintering temperature	620°C	630°C

From **Table 4-4**, the final weight of compacts is slightly higher than initial weight although it was not obvious. Sintering of compacts will expand the size of powder particles and increase the weight. There are certain modifications made in sintering procedures between both samples. The usage of argon gas in sintering furnace the

first sample is not suitable because argon has low dew point temperature which can reduce properties of aluminium compact. Because of that, nitrogen gas is used in furnace atmosphere for the second sample with 15 minutes sintering time 630°C maximum sintering temperature. Normal dew point temperature used in common aluminium sintering process is between -40 to -60°F [12].

4.6 Aluminium Compact Test

4.6.1 Sample 1

To determine whether the strength of compact has improved or not, the aluminium compact was cut at the centre of the disk into two pieces by using diamond cutter. Unfortunately, the time for cutting for the sample is less than 3 minutes and it broken into unexpected shape as in **Figure 4-7** below.

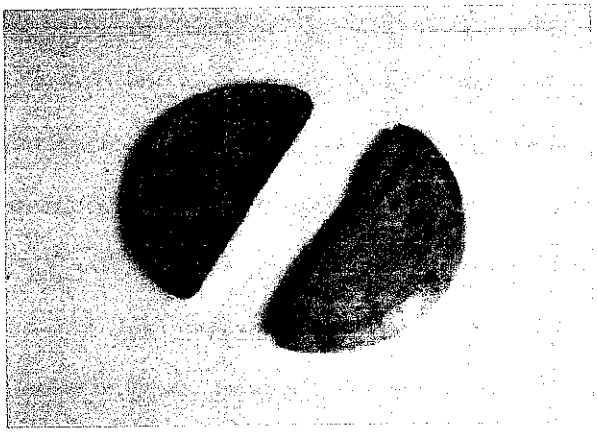


Figure 4-7: Sintered disk in argon at 620°C

To further analyze the compact, SEM was conducted to see how the powder particle changed after sintering at the surface of disk. The result is shown in the **Figure 4-8**. From the figure, it can be seen that the powder is still not well sintered. There is no significant bonding or densification occurred between the powder particles that cause low in strength.

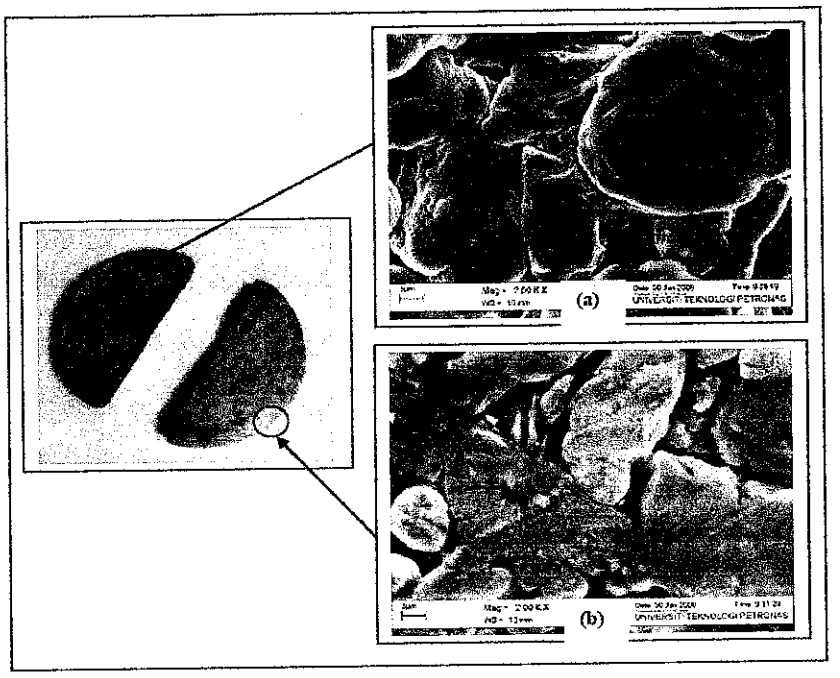


Figure 4-8: SEM Result a) at broken surface
b) at normal surface

To determine whether oxide layer has disturb sintering process or not, Energy Dispersive X-ray (EDX) analysis was conducted to see particles content in the original powder and also in the sintered disk.

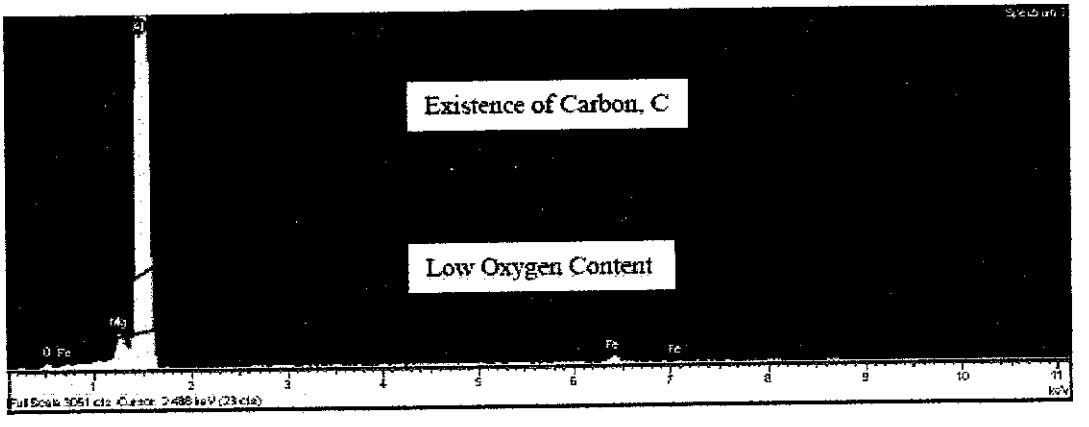


Figure 4-9: EDX on sintered aluminium part

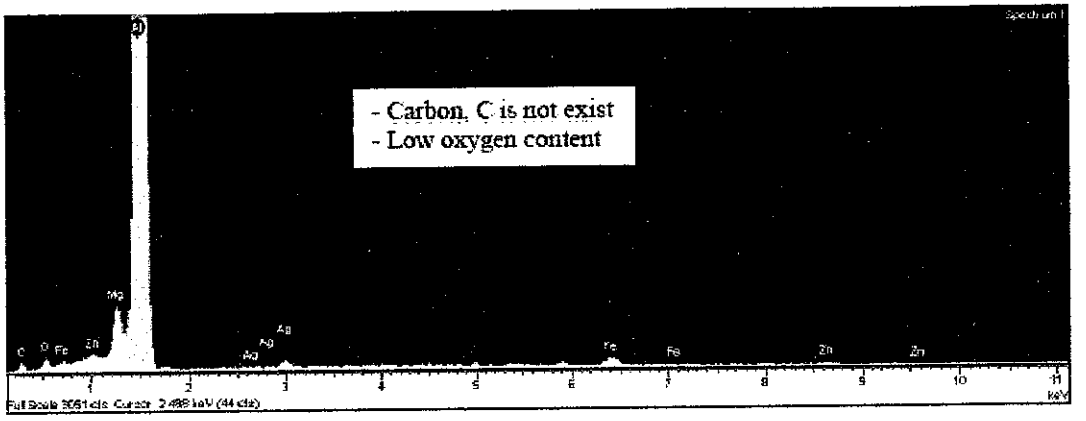


Figure 4-10: EDX on aluminium powder before sintering

4.6.2 Sample 2

This sample also was cut by using diamond cutter at the centre of the disk and it breaks during halfway of the cutting process as shown in **Figure 4-11.** Based on my observation, the cutting time for this sample is higher than the first sample by using same load. These indicate that under nitrogen atmosphere, harder material can be produced.

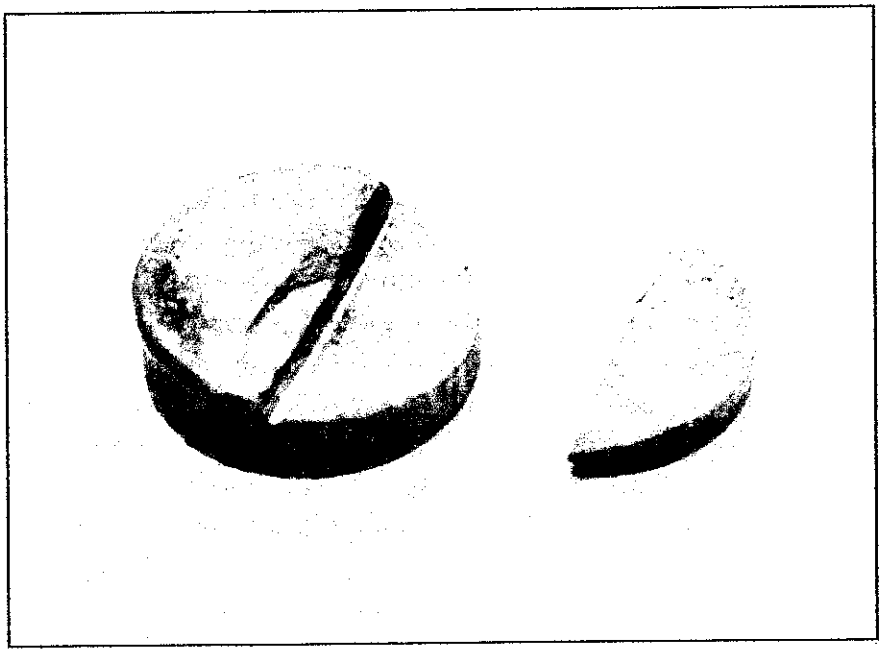


Figure 4-11: Sintered disk in nitrogen at 630°C

To analyze what exactly occurred between the powder particles, SEM and EDX was performed to the sample. Before that, the sample was polished, mounted and etched using Kellers etch which is suitable for aluminium alloys for 10-30 seconds immersion. The purpose of etching is optically enhancing microstructural features such as grain size and phase features.

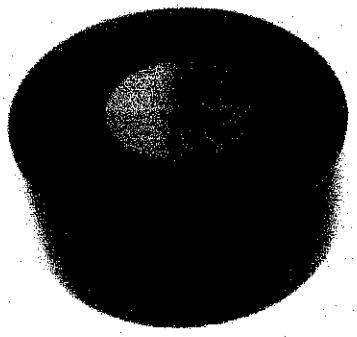


Figure 4-12: Polished surface of sintered disk before SEM analysis

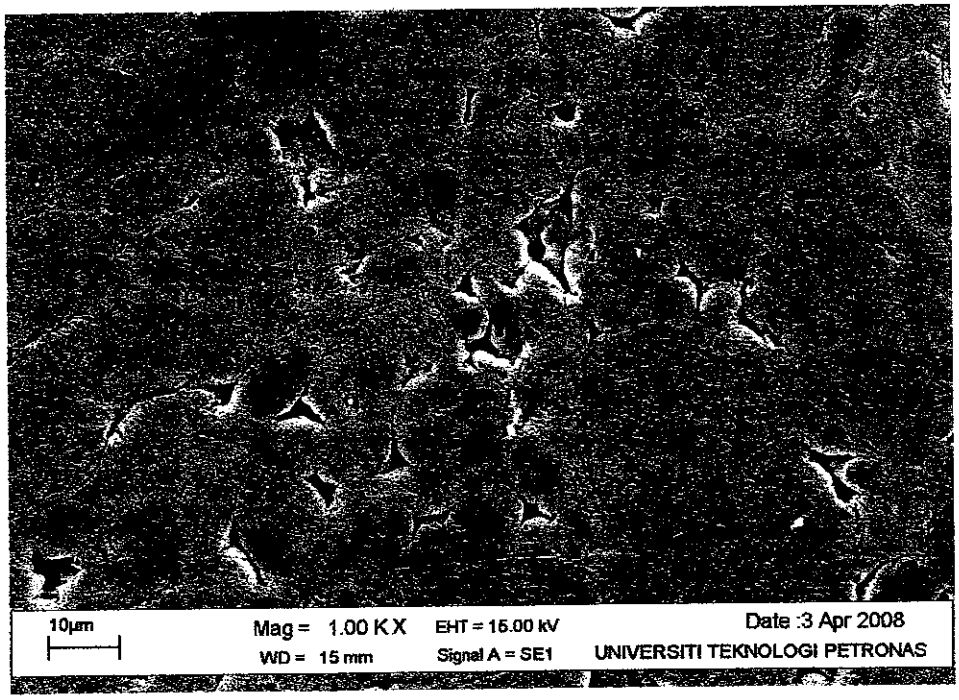


Figure 4-13: SEM image of microstructure of sintered disk in nitrogen at 630°C

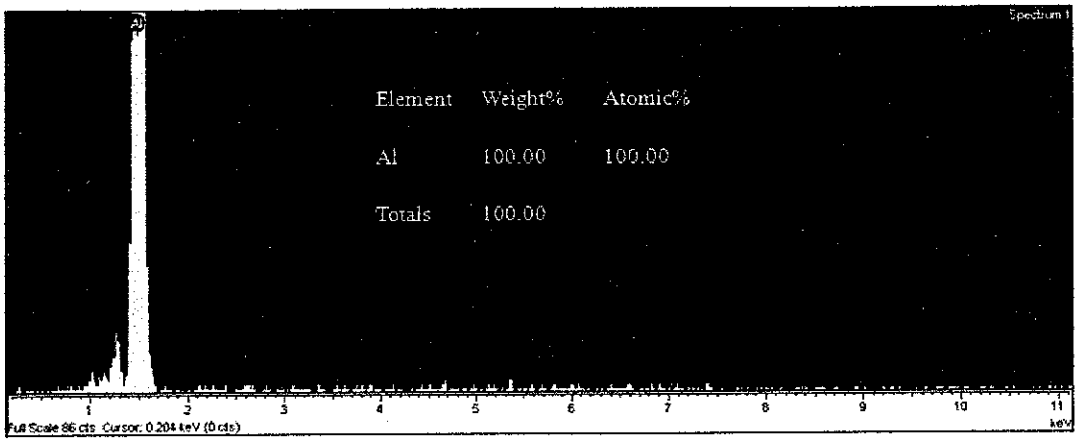


Figure 4-14: EDX result of sintered disk in nitrogen

From the SEM result, it shows that there are many pore spaces between the powder particles. Yet, some of the particles react and make significant bond between each other. Beside of that, oxide layer on aluminium particles were totally reduced by using nitrogen as sintering atmosphere and can be seen in **Figure 4-14**. So, nitrogen gas is the best sintering atmosphere to overcome problem with alumina film.

4.7 Composite Powder Compaction

Aluminium and carbon powder are mixed together in different portion. There are three samples prepared for the composite mixture and measured in term of volume percent. For this compaction, wax is used as a binder to produce better green compact. Compaction pressure of 255 MPa was applied to yield a green compact. **Table 4.5** below shows the properties of composite powder for compaction and the result after compaction.

Table 4.5: Properties of composite powder in compaction process

	Sample 1 (3% C)	**Sample 2 (4% C)**	**Sample 3 (7% C)**
Weight of aluminium	1.4152 g	1.4156 g	1.4225 g
Weight of carbon	0.0283 g	0.0424 g	0.0711 g
Weight of wax	0.0141g	0.0141 g	0.0141 g
Diameter of disk	1.27 cm	1.27 cm	1.27 cm
Height of disk	0.5 cm	0.52 cm	0.54 cm
Weight after compaction	1.406 g	1.447 g	1.489 g
Theoretical density	2.67 g/cm^3	2.66 g/cm^3	2.63 g/cm^3
Measured density	2.22 g/cm^3	2.20 g/cm^3	2.17 g/cm^3
Percent of theoretical density	83 %	82.7 %	82.5 %

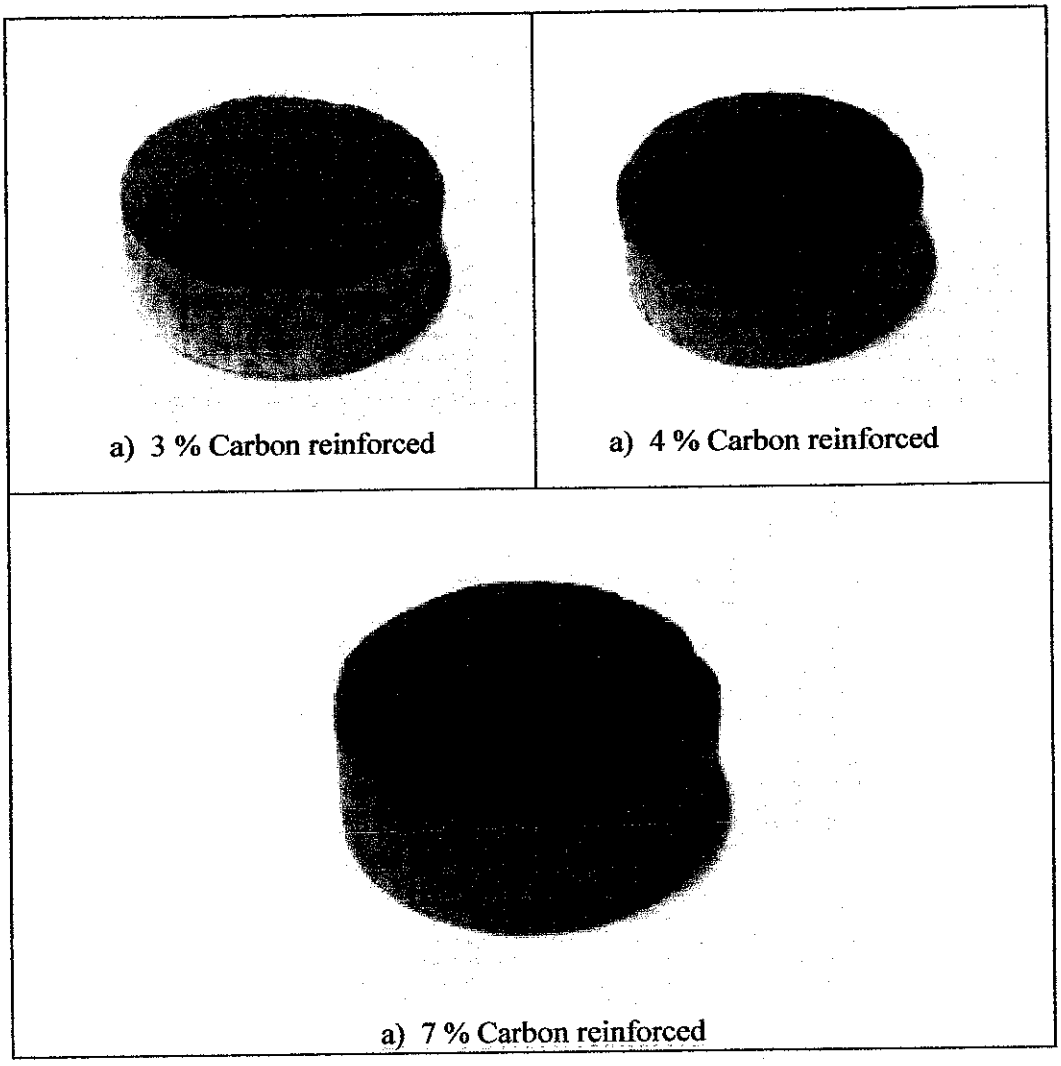


Figure 4.15: Green compact of Carbon-Aluminium composite

From **Table 4.5**, densities of green compacts for composite powder are reduced apparently compared to pure aluminium compact. For example, by adding 3 % of carbon to aluminium, the density is reduced from 2.618 g/cm^3 to 2.22 g/cm^3 which is decreased about 15 %. The huge different is maybe due to the pressure applied during compaction. For the pure aluminium compaction, 464 MPa of pressure was used while for the composite powder, only 255 MPa was applied. According to Young [13], above 300MPa bulk compression stage, poor sinterability was expected owing to formation of closed pore. Therefore, 255MPa which was starting point of homogeneous deformation stage was decide as the optimum compacting pressure.

From **Figure 4-15**, only composite with 3% of carbon can produce good shape of green compact and can handle easily. But for the 4% and 7% of carbon composite, the green compacts must be handled carefully because it can easily break with small force applied on it.

4.8 Sintering of Composite Compact

After compaction process, the three composite compacts were then transferred to sintering furnace. Different approach was applied during sintering in order to deal with binder which is wax. Burn off process to remove the wax was performed at 350°C for 1 hour [13] under nitrogen atmosphere. Then, sintering was carried out at temperature up to 620°C for 1 hour also under nitrogen. The properties of compacts and sintering atmosphere are as the following table:

Table 4-6: Sintering properties of composite compact

	3% Carbon	**4% Carbon**	**7% Carbon**
Initial weight of compact, m_1	1.406 g	1.447 g	1.489 g
Final weight of compact, m_2	1.398 g	1.433 g	1.483 g
Furnace atmosphere	Nitrogen		
Sintering time	1 hour		
Sintering temperature	620°C		

From **Table 4-6** above, the final weight of compact is slightly lower than initial weight although it is not obvious which is opposite to the result for pure aluminium. This is maybe due to the reaction between carbon and aluminium particles during sintering. Beside of that, the weight of compact reduced because of burn off process of wax.

4.9 Composite Compact Test

For the composite's mechanical properties evaluation, different approach was applied compared to pure aluminium compact. The composite compact was evaluated by using Micro hardness Test Machine. The test was performed using Vickers's scale with 300g load. Unfortunately, only composite compact with 3% of carbon can be evaluated because the other samples cannot be mounted during mounting process. The result is shown in **Table 4-7** below.

Table 4-7: Result for micro hardness test. (Vickers: 0.3kgf)

Sample	Test			Average
	1	2	3	
Pure Aluminium	65.8	64.5	63.9	64.7HV0.3
Aluminium + 3% Carbon	25.8	27.0	29.7	27.5HV0.3

From **Table 4-7**, the average hardness for the pure aluminium is decreased from 64.7HV0.3 to 27.5HV0.3 if the aluminium is added with 3% of carbon. The addition of the carbon makes the composite become softer because carbon is a soft material. EDX and SEM result for 3% carbon of composite compact are shown in **Figure 4-16** and **Figure 4-17** respectively.

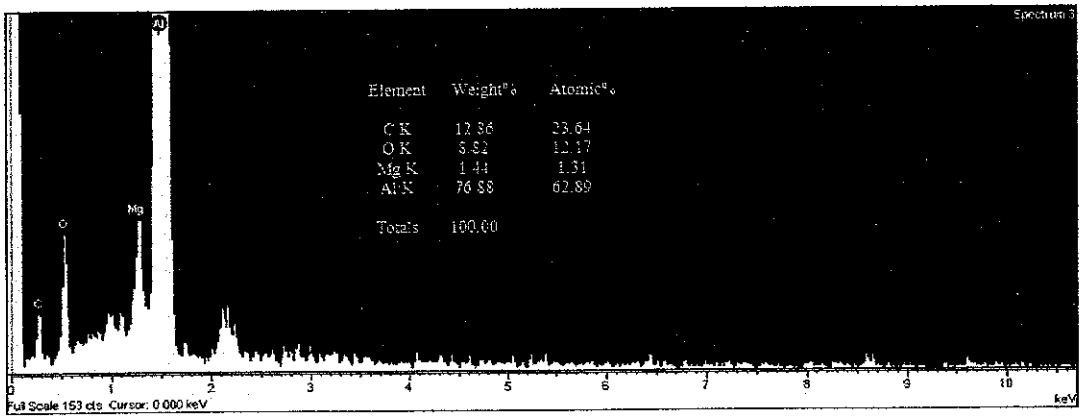
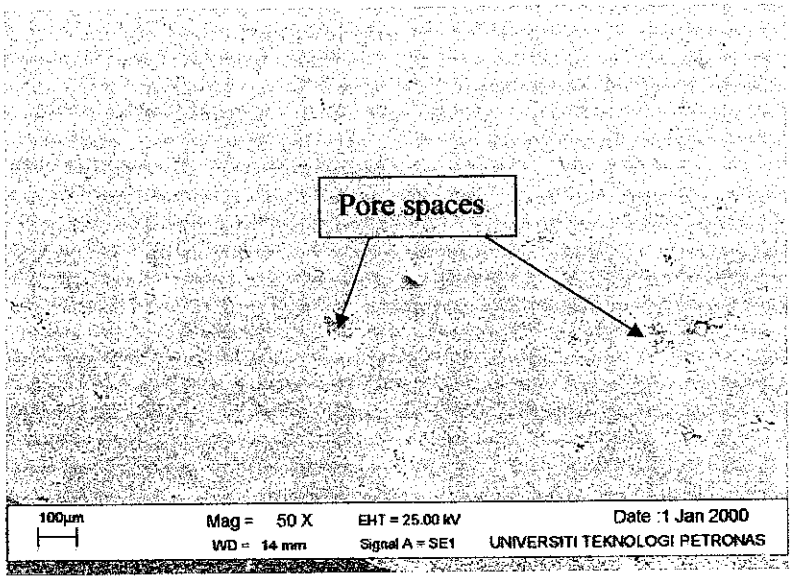
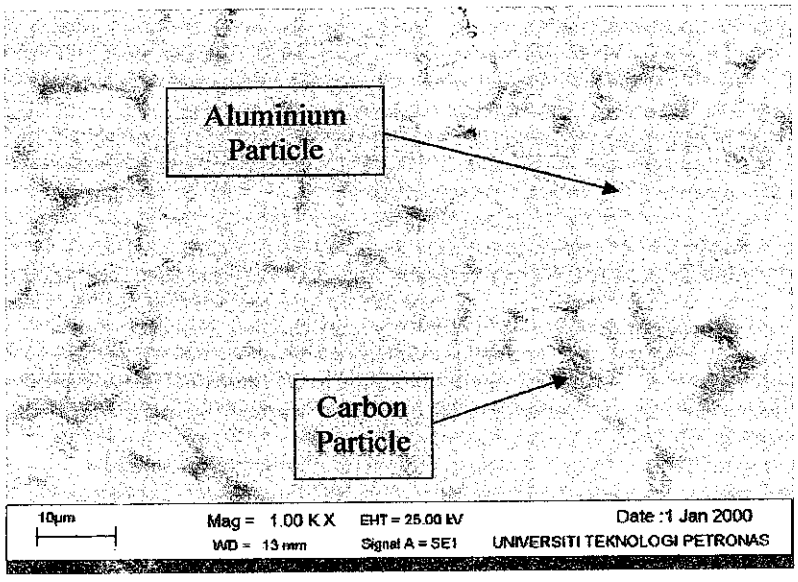


Figure 4-16: EDX result on 3% carbon reinforced aluminium composite



a) 50X magnification



b) 1000X magnification

Figure 4-17: SEM images on 3% carbon reinforced aluminium composite

From **Figure 4-17(a)**, we can see that a lot of pore spaces in 3% of carbon composite material under 50X magnification. By that, the density of material becomes lower because of high porosity of the material. In terms of powder particle bonding, it can be seen that carbon powder fills the empty spaces between aluminium particles perfectly as in **Figure 4-17(b).** Densification also occurs among aluminium powder particles.

CHAPTER 5

5.0 CONCLUSION AND RECOMMENDATIONS

By performing pure aluminium compaction and sintering process first, the flow of this project much safer and can be controlled in term of material usage if failure occurs. Aluminium powder sintering is hard to achieve because the aluminium oxide is not reduced by common furnace atmospheres. It is very important to find the suitable furnace atmospheres in sintering. In this project, the usage of argon gas in furnace atmosphere failed to sinter pure aluminium powder to get desired result. After major modifications of compaction pressure, and sintering time sintering atmosphere of nitrogen instead of argon, the properties of pure aluminium compact improved and better than the first sample in term of hardness and strength.

For the carbon reinforced aluminium composite, only 3% of carbon in aluminium can produce high strength green compact. If the amount of carbon is higher than that, the green compact is not strong enough for handling and further analysis. From the result, the hardness of composite material is lower than pure aluminium compact probably due to alumina film on aluminium powder is not totally reduced during sintering and carbon also is a soft material. By the way, this reinforced composite material is suitable to use as self lubricating material.

However there are some aspects and areas in the in this project must be studied to a greater extent. One of the major problems in this project is to determine sintering behaviour of aluminium. There are still no deep studies about this topic in this university and it is recommended to conduct the deep study for benefits to the future.

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[12] Brochure by members of the Marketing and the Technical & Standards Committees of the Pigments and Powder Division of the Aluminum Association, Inc

[13] Young Do Kim, *"Sintering Behavior and Mechanical Properties of 7XXX Al Alloy with Variation of SiC Contents,"* Division of Materials Science and Engineering, Hanyang University, Seoul, Korea.

2. **Others**

- http://en.wikipedia.org/wiki/Powder_metallurgy
- http://www.mpif.org
- Video form Society of Manufacturing Engineers, *Powder Metallurgy.*

APPENDICES

APPENDIX A: Project Gantt chart

Semester 1

No	Progress Work	Week													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of Project Topic														
2	Preliminary Research Work														
3	Submission of Preliminary Report				X										
4	Analyze Particle Size														
5	Mixing Particles														
6	Submission Of Progress Report								X						
7	Compaction Work														

Semester 2

No	Progress Work	Week													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Compaction of pure aluminium														
2	Sintering of pure aluminium														
3	Perform test to aluminium compact - Cutting - SEM - EDX														
4	Mixing composite powders														
5	Compaction of composite														
6	Sintering of composite														
7	Perform test to composite compact - Hardness test - SEM - EDX														

APPENDIX B

Particle size analysis results

MASTERSIZER 2000

Result Analysis Report

Sample Name:
fyp_Si

SOP Name:

Measured:
Wednesday, September 19, 2007 10:54:28 PM

Sample Source & type:
Works = utp

Measured by:
User

Analysed:
Wednesday, September 19, 2007 10:54:32 PM

Sample bulk lot ref:
01

Result Source:
Measurement

Particle Name:
Silica 0.0

Accessory Name:
Scirocco 2000 (A)

Analysis model:
Single narrow mode

Sensitivity:
Enhanced

Particle RI:
1.544

Absorption:
0

Size range:
0.020 to 2000.000 um

Obscuration:
2.85 %

Dispersant Name:
Dry dispersion

Dispersant RI:
1.000

Weighted Residual:
6.741 %

Result Emulation:
Off

Concentration:
0.0002 %Vol

Span :
1.873

Uniformity:
0.598

Result units:
Volume

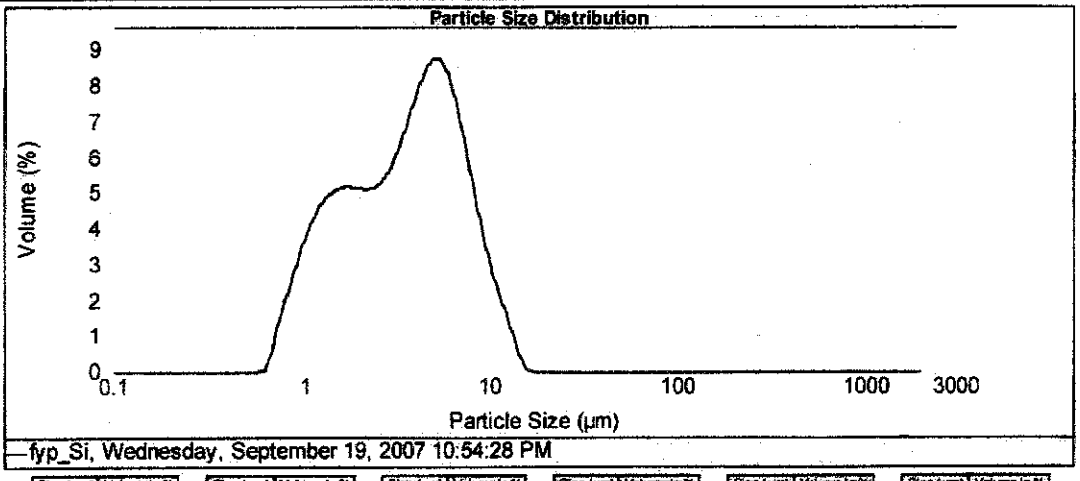
Specific Surface Area:
2.35 m²/g

Surface Weighted Mean D[3,2]:
2.555 um

Vol. Weighted Mean D[4,3]:
4.193 um

---

d(0.1): 1.174 um **d(0.5):** 3.667 um **d(0.9):** 8.041 um



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.063	0.00	0.366	0.00	2.462	3.78	15.676	0.00	98.656	0.00
0.011	0.00	0.070	0.00	0.441	0.00	2.776	4.08	17.470	0.00	109.929	0.00
0.012	0.00	0.078	0.00	0.492	0.00	3.094	4.51	19.468	0.00	122.491	0.00
0.014	0.00	0.087	0.00	0.548	0.00	3.447	5.05	21.690	0.00	136.488	0.00
0.015	0.00	0.097	0.00	0.610	0.09	3.841	5.58	24.169	0.00	152.084	0.00
0.017	0.00	0.108	0.00	0.680	0.82	4.280	6.00	26.931	0.00	169.462	0.00
0.019	0.00	0.120	0.00	0.758	1.41	4.769	6.21	30.008	0.00	188.826	0.00
0.021	0.00	0.134	0.00	0.844	2.00	5.314	6.08	33.437	0.00	210.403	0.00
0.024	0.00	0.150	0.00	0.941	2.55	5.921	5.62	37.258	0.00	234.446	0.00
0.026	0.00	0.167	0.00	1.048	2.99	6.598	4.89	41.515	0.00	261.235	0.00
0.030	0.00	0.185	0.00	1.168	3.31	7.351	4.01	46.259	0.00	291.088	0.00
0.033	0.00	0.207	0.00	1.302	3.52	8.191	3.14	51.545	0.00	324.348	0.00
0.037	0.00	0.231	0.00	1.451	3.62	9.127	2.37	57.435	0.00	361.411	0.00
0.041	0.00	0.257	0.00	1.616	3.65	10.170	1.74	63.998	0.00	402.708	0.00
0.045	0.00	0.286	0.00	1.801	3.63	11.333	1.24	71.311	0.00	448.725	0.00
0.051	0.00	0.319	0.00	2.007	3.61	12.628	0.70	79.459	0.00	500.000	0.00
0.056	0.00	0.355	0.00	2.238	3.63	14.071	0.19	88.539	0.00		
0.063		0.396		2.492		15.678		98.656			

Operator notes:

Malvern Instruments Ltd
Malvern, UK
Tel: +[44] (0) 1684-892456 Fax +[44] (0) 1684-892789

MASTERSIZER 2000

Result Analysis Report

Sample Name:
fyp2_Si

SOP Name:

Measured:
Tuesday, September 25, 2007 3:15:40 AM

Sample Source & type:
Works = utp

Measured by:
Unknown

Analysed:
Tuesday, September 25, 2007 3:15:44 AM

Sample bulk lot ref:
01

Result Source:
Measurement

Particle Name:
Aluminium

Accessory Name:
Scirocco 2000 (A)

Analysis model:
Single narrow mode

Sensitivity:
Enhanced

Particle RI:
2.500

Absorption:
3

Size range:
0.020 to 2000.000 um

Obscuration:
0.00 %

Dispersant Name:
Dry dispersion

Dispersant RI:
1.000

Weighted Residual:
1.661 %

Result Emulation:
Off

Concentration:
0.0000 %Vol

Span :
1.968

Uniformity:
0.591

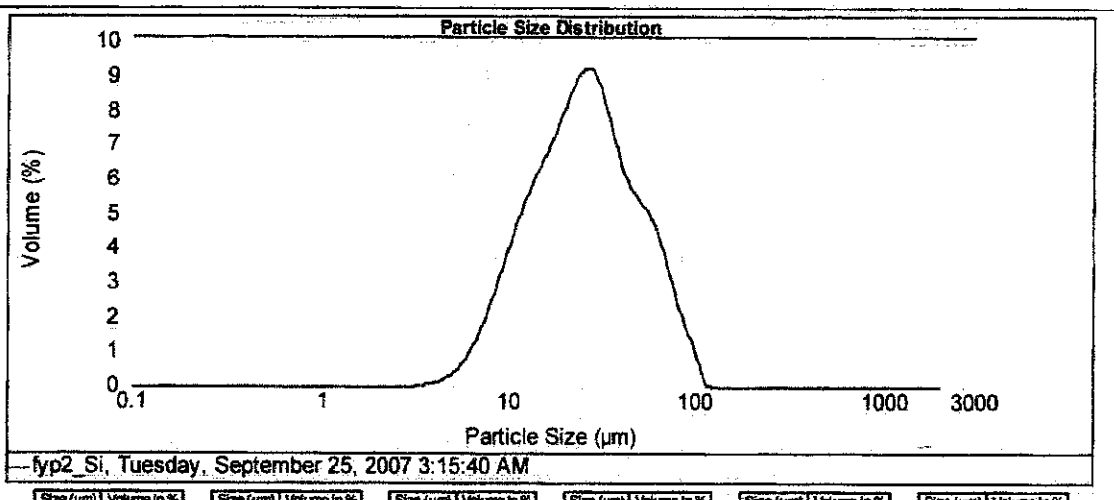
Result units:
Volume

Specific Surface Area:
0.293 m²/g

Surface Weighted Mean D[3,2]:
20.478 um

Vol. Weighted Mean D[4,3]:
31.093 um

d(0.1): 10.583 um **d(0.5):** 25.651 um **d(0.9):** 61.053 um



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.053	0.00	0.386	0.00	2.492	0.00	15.678	4.88	98.656	0.63
0.011	0.00	0.070	0.00	0.441	0.00	2.776	0.00	17.470	5.29	109.929	0.04
0.012	0.00	0.078	0.00	0.492	0.00	3.094	0.00	19.466	5.76	122.491	0.00
0.014	0.00	0.087	0.00	0.548	0.00	3.447	0.00	21.690	6.20	136.488	0.00
0.015	0.00	0.097	0.00	0.610	0.00	3.841	0.05	24.169	6.50	152.094	0.00
0.017	0.00	0.108	0.00	0.680	0.00	4.280	0.08	26.931	6.51	169.492	0.00
0.019	0.00	0.120	0.00	0.758	0.00	4.769	0.15	30.008	6.20	188.826	0.00
0.021	0.00	0.134	0.00	0.844	0.00	5.314	0.26	33.437	5.57	210.403	0.00
0.024	0.00	0.150	0.00	0.941	0.00	5.921	0.44	37.258	4.66	234.446	0.00
0.026	0.00	0.167	0.00	1.048	0.00	6.598	0.71	41.515	4.29	261.235	0.00
0.030	0.00	0.186	0.00	1.168	0.00	7.351	1.07	46.259	3.94	291.086	0.00
0.033	0.00	0.207	0.00	1.302	0.00	8.191	1.51	51.545	3.71	324.348	0.00
0.037	0.00	0.231	0.00	1.451	0.00	9.127	2.04	57.435	3.42	361.411	0.00
0.041	0.00	0.257	0.00	1.616	0.00	10.170	2.60	63.998	2.94	402.708	0.00
0.045	0.00	0.286	0.00	1.801	0.00	11.333	3.16	71.311	2.27	448.725	0.00
0.051	0.00	0.319	0.00	2.007	0.00	12.628	3.68	79.459	1.59	500.000	0.00
0.056	0.00	0.355	0.00	2.238	0.00	14.071	4.11	88.539	1.09		
0.063	0.00	0.396	0.00	2.492	0.00	15.678	4.49	98.656	1.09		

Operator notes: