Study on Sintering Behavior of Alumina Particle Reinforced Aluminum Matrix Composites and Its Properties.

by

Mohammad Faiz b Moktar

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

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CERTIFICATION OF APPROVAL

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Approved by,

(AP Dr Faiz Ahmad)
Project Supervisor

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
July 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.

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MOHAMMAD FAIZ BIN MOKTAR

ABSTRACT

Aluminium matrix composites (AMC) are very attractive due to high strength to weight ratio and have potential for automotive and aerospace industries. In this study, alumina particles reinforced aluminium were developed using powder metallurgy technique. The alumina particles were reinforced in the ranges of 2.5% to 10% of volume fraction. The materials were characterized using particle size analyzer and scanning electron microscope for particle size, distribution, shape and surface. A homogenous mixture of aluminium and alumina were prepared and compacted at 500MPa. The compacted parts were sintered at the temperature ranging from 550°C to 650°C under nitrogen atmosphere for two hours to study the behaviour of materials density, shrinkage, microstructure and hardness. In order for hardness test, micrograph and energy dispersive X-ray (EDX) to be conducted, surface preparation for the sintered parts is required such as mounting, grinding and polishing. The study yielded that the optimum sintering temperature is 580°C. The composites sintered at 580°C achieved the highest theoretical density and hardness value. The composites also produced the least shrinkage with below 1% controllable shrunk. The micrograph taken by SEM of the composites sintered at 580°C showed smooth microstructure produced, large pore spaces or void is eliminated and also with greater interparticle bonding. The results also showed that all density of the composites were increased after sintering process and the composite produced shrinkage with no impurities presented throughout the experiment.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1

INTRODUCTION

1.1 Background of study

Research and effort to develop, characterize and design structures with high temperature composite materials are underway across the globe. However, only in the past few years have these composites become realistic competence as engineering materials.

Today approximately 100,000 types of engineering materials are represented in the market and this figure is rapidly increasing. Mass market products, like automobiles, now contain metal matrix composite components. The application of MMCs is being explored in many other applications including aerospace and sporting goods. The first MMCs were developed during the 1960s, but because of problems with manufacturing processes and finding fibers that could be compatible with the matrix; no real attention was paid to these materials [1].

The high costs of MMC preclude them from achieving their full application potential. However the prices are expected to fall as the number of applications increases [1].

Hence, this study aimed at achieving an optimum sintering temperature that fit with combination of properties mention above. Towards realizing this study, the powder metallurgy processing route has been chosen as the fabrication method for the AMCs.

1.2 Problem Statement

The demand for light weight components are increased that caused from the need to reduce energy consumption in a variety of applications. These issues have led the increasing usage of aluminum alloy due to its competitive properties. High operational and capital costs associated with intricate machining operations that restricted the Aluminum Matrix Composites (AMCs) parts applications. The AMCs usually reinforced by Al_2O_3, SiC, C but SiO_2, B, BN, B_4C, AlN may also considered [2]. The unique characteristics of aluminum such as strength, weight, corrosion resistance and machinability can make the aluminum parts economically viable.

Powder Metallurgy method used in fabricating the MMC will result in high material utilization, cost effective and reduces energy consumption. By using Powder Metallurgy method, it is anticipated that the mechanical and physicals properties of Aluminum Matrix Composite to be superior to the MMC using other fabrication method and will lead to the development of new light weight metal composites.

1.3 Objectives and Scope of Study

The main objective of this study is to identify the optimum sintering temperature of alumina particle reinforced aluminum matrix composites. The composite is developed using the powder metallurgy route. The composite is sintered at four different temperatures under nitrogen atmosphere. Further than that, it is valuable to know and record the properties such as hardness and microstructure and study the behavior of this composite material during green state and after sintering process.

The scope of study is to investigate the sintering behavior of the composite restructuring sintered density, relative theoretical density, microstructure and hardness of the composite during green state and after sintering process.

1.4 Significance of the Study

This study is significant because of the need to develop low cost, light weight and high performance aluminium metal matrix composites using alumina particle as reinforcement. The main concern of this project is to search the optimum sintering temperatures for AMC in particular atmosphere in a particular composition. The behaviour of the samples in different sintering temperatures will evaluate their performance based on microstructure and also will investigate the mechanical and physical properties of the AMCs. Therefore the aim of optimize the sintering temperature will lead to develop a light weight sintered composite material.

CHAPTER 2

LITERATURE REVIEW

2.1 Aluminium Matrix Composite (AMC)

In metal matrix composite, one component is a metal or alloy forming at least one percolating network. The other component is embedded in this metal matrix and usually serves as reinforcement. Metal matrix is the percolating metal or alloy into which the reinforcement is embedded. The reinforcement is a constituent of MMC originating from the ingredient material which is combined with a metal or alloy e.g alumina fibres, silicon carbide whiskers and steel fibres. Reinforcement is characterised by its chemical composition, its shape and dimensions, its properties as ingredient material and its volume fraction and spatial distribution in the matrix [3].

The market value of AMC is the highest among the different MMCs, due to its small production compared to the total production of MMCs [4]. The reinforced materials for AMC can be classified in 3 different forms and are shown in Figure 2-1:

- Particulates
- Whiskers or discontinuous fibres that are polycrystalline.
- Continuous fibres

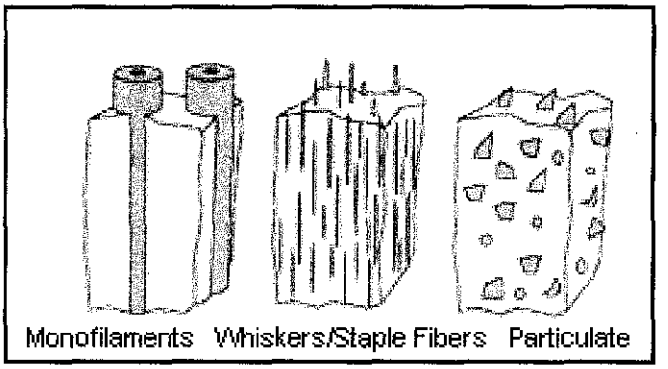


Figure 2-1: Monofilaments, whiskers/staple fibers and particulate
(Source; Website: http://www.metal-matrix.htm.,access 5 Feb 2008)

In this project, the particulate based composite is considered as the reinforcement. The particulate based composite systems offer low cost with significant improvement in stiffness.

2.2 Powder metallurgy process

The Powder Metallurgy (PM) route is the most commonly used method for preparation of discontinuous reinforced MMCs [5]. Powder metallurgy has become competitive to other manufacturing process because of it great advantages i.e. produces good surface finishes, provides materials which may be heat-treated for increased strength or increased wear resistance, provides controlled porosity for self-lubrication or filtration, offers long-term performance reliability in critical applications and is cost-effective [2]. The basic steps in the production of sintered engineering components are those of powder production; the mechanical compaction of the powder into a handleable preform; and the heating of the preform to a temperature below the melting point of the major constituent for a sufficient time to permit the development of the required properties[6]. The flow chart for composite process route by powder metallurgy is illustrated in Figure 2-2;

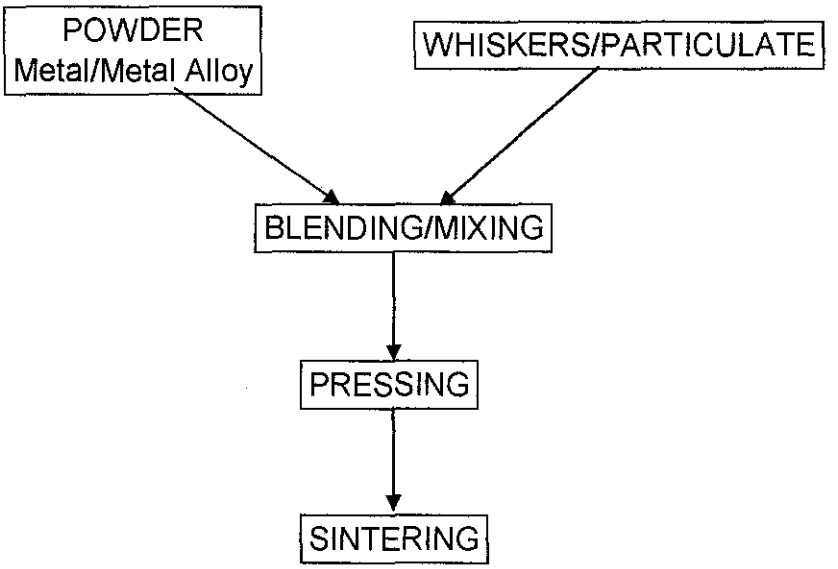


Figure 2-2: Flow chart for composite process route by powder metallurgy.

2.2.1 Powder Characterization

2.2.1.1 Particle Size

For packing composed of large particles, the particles size is not important to the density. If the mean particle size is below 100μm, then interparticle friction and particle bridging is likely to occur. The decreasing packing density with smaller particles is due to an increase in the surface area, a lower particle mass and weak forces such as electrostatic fields, moisture and surface adsorption [7]. Since interparticle cohesion increases with a smaller particle size, there is more agglomeration and inhibited packing and thus lower the packing density.

2.2.1.2 Particle Shape and Surface Texture

The greater the surface roughness or more irregular the particle shape results lower the packing density. This is due to the bridging of the particles. In powder mixing, an irregular particle shape will interfere with the mixing, but also

maintain a homogeneous mixture by interfering with demixing [7]. Density can be improved by mixing different sizes of particles.

2.2.2 Mixing

2.2.2.1 Lubricant

The metal powder is mixed with lubricant and optional alloying elements to form a homogenous blend. The main function of the lubricant in the powder mix is to reduce the friction between die wall and powder particle during compaction. There are two methods in applying the lubricant to the powder mix,

- Lubricant is applied all over the die cavity and top face of punch.
- Lubricant is added into the mixture of the powder mix.

The lubricant applied in powder mix can result in higher density of the powder through increased effective pressure on the powder hence improved the mechanical strength of the composite. It is also can reduce the ejection force apply after the compaction. But there will be a drawback in the method is added into the mixture of the powder mix. According to Abolfazl Babakhani, Ali Haerian and Mohammad Ghambari(2006)

> Due to the low density of the lubricant (around 1 g/cm^3), at higher amounts of binder, the green density is lowered. Maximum density is achieved when no lubricant is mixed with the powder, but the die wall is properly lubricated. The changes are more or less linear [8].

So it is not advisable to add the lubricant into the powder mix. By using lubricant or release agent over the die wall and top punch it will generate maximum density of the composite and hence increase the strength of the materials.

2.2.2.2 Volume Fraction

One of the most important factors determining the properties of composites is the relative proportions of the matrix and reinforcing materials. The relative proportions can be given as the weight fractions or the volume fractions [9]. In this study, alumina reinforced aluminium composite density is determined using volume fraction method.

Volume fraction of the alumina is ranging from 2.5%, 5%, 7.5% and 10%. The overall fraction of the composite is 100 cm^3. It is important to know the density of both powders and reinforcement in order to determine the density of the composite.

2.2.3 Compaction process

The behaviour of powders on pressing depends on many factors such as particle size, shape and composition, the plasticity of the solid and the effects of surface films.

There are two compaction techniques identified throughout this present study. Cold compaction and more recent uses hot compaction technique use warm powder in heated dies to increase green density and hence improve mechanical properties of the composite. The external pressure repacks and deforms the particles into a higher density [7]. It is important to understand the compaction of particles occurred during the compaction process. There are four stages in densification of the powder, illustrated in **Figure 2-3**.

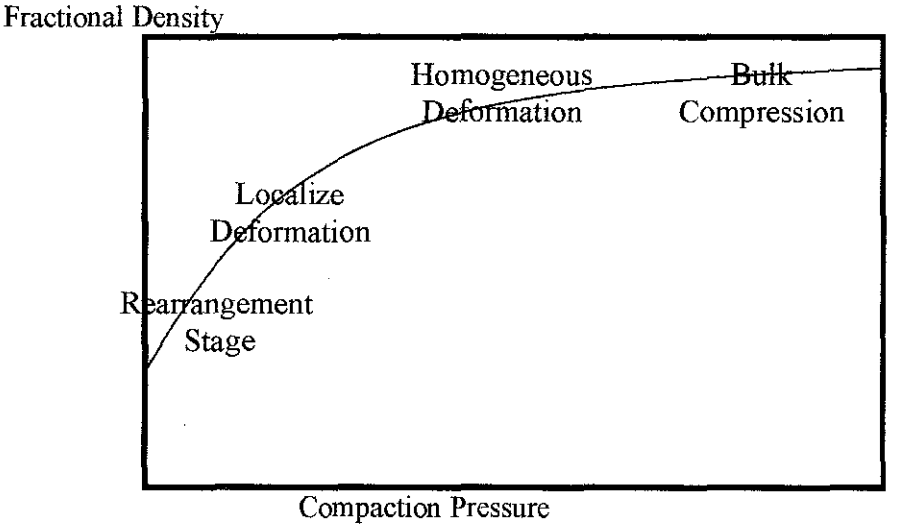


Figure 2-3: Fractional density versus pressure for particle compaction showing the four overlapping stages.

The ejection of the composite part, based on the past study concluded that to avoid any damage to the compacted part in ejection, the ejection pressure should be decreased immediately after the maximum pressure is obtained [10].

2.2.4 Sintering process

Sintering is a method to form objects from powder compacts by heating the material (below its melting point) until its particles adhere to each other. During sintering, pores between the starting particles are removed together with the growth of particle and develop strong bonding between adjacent particles [11].

According to J. L. Estrada, V. M. Carreno, H. Balmori and J. Duszcyk (1996), noted that the influence of different atmospheres (air and nitrogen atmosphere) during sintering showed that sintering powders with cold isostatic pressing at 408MPa and at different temperatures (300 to 530°C) in air under ideal condition (1 atm pressure), the oxygen in the compacts and the oxygen in the air react with the metal forming aluminum oxide.

This aluminum oxide hinders the diffusion of aluminum atoms through the oxide layer which increases the compact volume. In this case, sintering is produced only by diffusion through the interparticle contact points. On the other hand, sintering in nitrogen avoids the formation of aluminum oxide permitting more Al atoms to diffuse through the oxide layer [12].

CHAPTER 3

METHODOLOGY

3.1 Experimental Procedure

Beginning of this project is about researching and understanding on the powder metallurgy process concept and metal matrix composites fundamentals. A thorough literature review will be done through reference books, internet and journals for further understanding. All the works, effort and procedures used in this project will closely follow the provided Gantt chart. The flow of the research processes is illustrated in **Figure 3-1**.

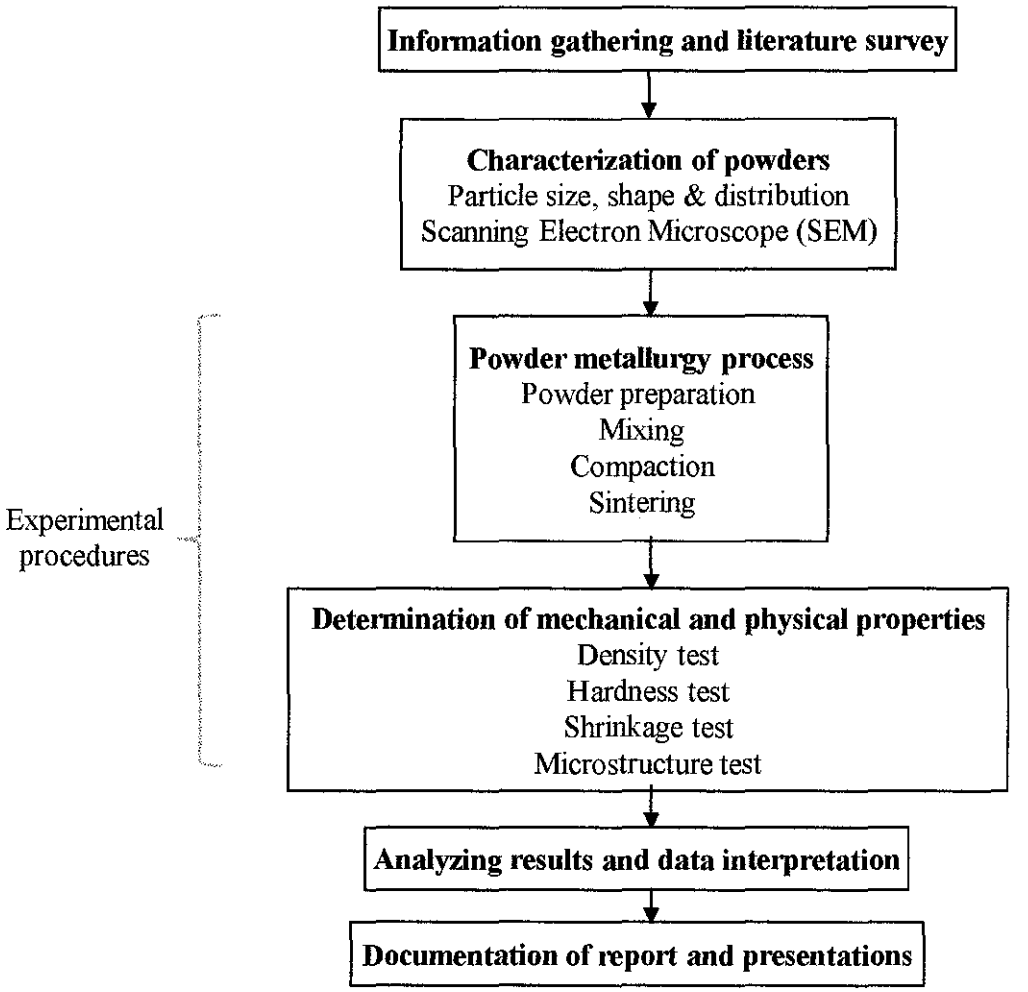


Figure 3-1: Flow chart of research process.

Appendix B and Appendix C illustrated the Gantt chart of this project in Final Year Project I and Final Year Project II.

3.2 Experimental Work

3.2.1 Powder Characterization

3.2.1.1 Particle Size Distribution

Particle size distribution 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.

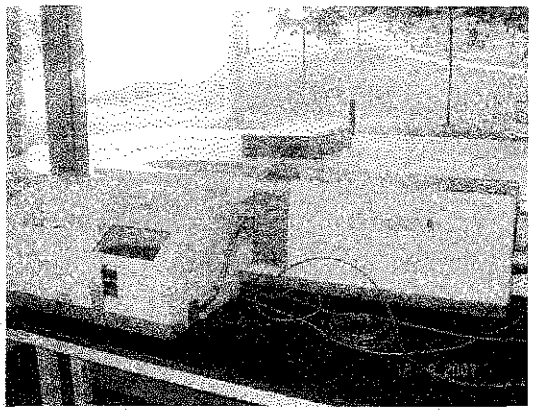


Figure 3-2: Mastersizer® 2000 with Scirocco dry powder dispersion unit

3.2.1.2 Particle Shape and Energy Dispersive X-ray

Particle shape analysis and chemical characterization or elemental analysis is performed by using Scanning Electron Microscope.

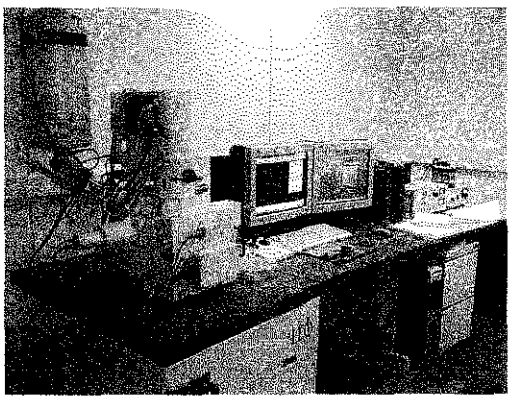


Figure 3-3: Scanning Electron Microscope

SEM works by scanning the surface of the poured specimen with electron beam, and the reflected (or back-scattered) beam of electrons is collected, then displayed at the same scanning rate on a cathode ray tube (similar to a TV screen). The image on the screen represents the surface features of the specimen. For non conductive specimen, a very thin surface metallic coating must be applied. The coating used is gold coating.

3.2.2 Composite Powder Volume Fraction and Mixing

Both aluminum and alumina powders are mixed with certain volume fraction. This is the most important factors in determining the properties of composite relative to proportions of the matrix and reinforcing materials. The properties for both materials are as follows:

Table 3-1: Properties of alumina and aluminum

Alumina		**Aluminum**	
Purity	99%	Density	2.7g/cm^3
Bulk Density	800-1000kg	Melting Temperature	660°C
Density	3.98g/cm^3		

In this project, 2.5%, 5%, 7.5% and 10% of alumina powder are mixed with aluminum powder. Table 3-1 shows the relative volume fraction for the composite.

Table 3-2: Composite Relative Volume Fraction

	Sample Dimension (cm)			Aluminum			Alumina			Composite	
	h	d	vol	%vol	vol (cm3)	mass (g)	%vol	vol (cm3)	mass (g)	mass (g)	Density
AMC-1	0.6657	1.30	0.8836	97.5	0.8615	2.3261	2.5	0.0221	0.0879	2.4140	2.732
AMC-2	0.6657	1.30	0.8836	95.0	0.8394	2.2664	5.0	0.0442	0.1758	2.4423	2.764
AMC-3	0.6657	1.30	0.8836	92.5	0.8173	2.2068	7.5	0.0663	0.2638	2.4705	2.796
AMC-4	0.6657	1.30	0.8836	90.0	0.7952	2.1471	10	0.0884	0.3517	2.4988	2.828
Total Usage					3.3135	8.9465		0.2209	0.8792		

Meanwhile the mixing of each composite is accomplished in a small vessel using spatula for at 15 – 20 minutes to assure the uniform dispersion of particles.

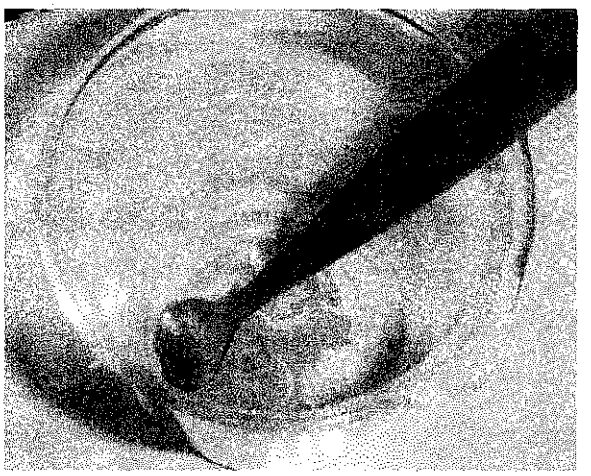


Figure 3-4: Mixing of Composites

3.2.3 Composite Compaction

After the mixing process, the composite mixes are then compacted by using mechanical press to produce a green compact. This cold pressing process is performed by pressing machine. Release agent is applied on walls of the die and the top punch of the compaction press. Table 3-2 shows the compaction parameters employed. The compaction pressure is achieved by using the Autopallet Press Machine.

Table 3-3: Compaction Parameters

Sample No.	AMC-0	AMC-1	AMC-2	AMC-3	AMC-4
Composition	Aluminum Powder	Al-97.5%vol; Alumina-2.5%vol	Al-95%vol; Alumina-5%vol	Al-92.5%vol; Alumina-7.5%vol	Al-90%vol; Alumina-10%vol
Compaction Pressure (MPa)	495.14	495.14	495.14	495.14	495.14
Holding Time (s)	60	60	60	60	60
Eject Force (kgf)	5000	5000	5000	5000	5000
Diameter (cm)	1.304	1.3	1.3	1.3	1.3
Height (cm)	0.602	0.73	0.846	0.846	0.72
Weight (g)	1.954	2.326	2.328	2.401	2.356

The sample dimension is determined by refer to the mould available in the laboratory. Figure 3-5 shows the size and dimension of the sample.

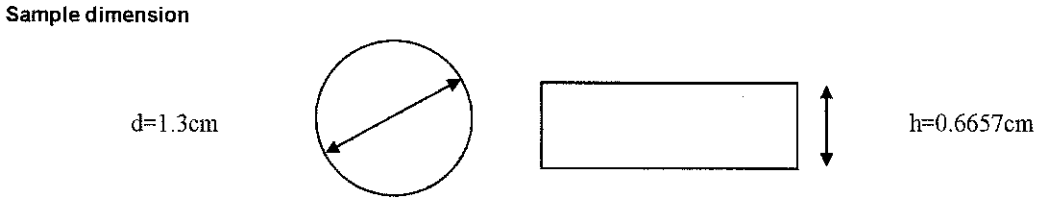


Figure 3-5: Sample Dimensions

Figure 3-6: Autopallet Press Machine

3.2.4 Sintering Process

Sintering is carry out under pure nitrogen atmosphere within the temperature range between 550 and 650°C using the tube furnace available in the laboratory. The green parts are heated below the melting point of the aluminum but high enough to bond the particle. Table 3-3 shows the sintering parameters employed.

Table 3-4: Sintering Parameters

	AMC-0	AMC-1	AMC-2	AMC-3	AMC-4
Composition	Aluminum Powder	Al-97.5%vol; Alumina-2.5%vol	Al-95%vol; Alumina-5%vol	Al-92.5%vol; Alumina-7.5%vol	Al-90%vol; Alumina-10%vol
Temperature (°C)	650	650	650	650	650
Holding Time (Hr)	2	2	2	2	2
Atmosphere	Pure Nitrogen	Pure Nitrogen	Pure Nitrogen	Pure Nitrogen	Pure Nitrogen
Rate of Heating (°C/min)	20	20	5	5	5
Rate of Cooling (°C/min)	20	20	5	5	5

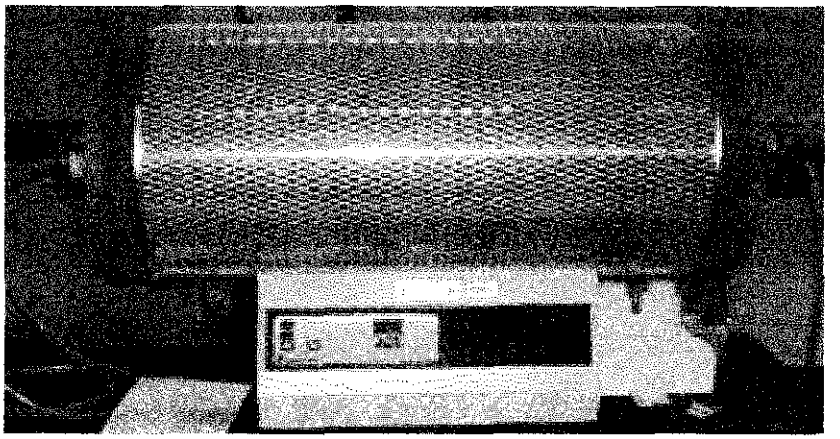


Figure 3-7: Carbolite tube furnace.

3.2.5 Density Test

Density test is performed to determine the degree of the particle pack together and it is given by the ratio of mass and volume.

$$\rho = \frac{m}{v}$$

where $\rho = density$, $m = mass$, $v = volume$

Meanwhile for relative theoretical density is the ratio of the experimental density to the calculated density.

$$\%TD = \frac{\rho_{experiment}}{\rho_{theory}}$$

3.2.6 Hot Mounting

After sintering process the samples is then mounted by mounting machine. Buehler, Simpliment 1000 is the mounting machine model. Thermosetting polymeric powder used is phenolic powder. The phenolic powder is placed in the mould with sample then is heated for 2min and cooled for 5min under the pressure of 3500psi

Figure 3-8: Auto Mounting Pressing Machine

3.2.7 Polishing and Grinding

Grinding and polishing is performed using Grinder and Polisher machine model Metaserv 2000. The sample is ground with SiC paper and water. The SiC paper used raging from 400grits to 1200grits. For polishing the sample is polish with the rough polish first which is 6micron and then polish with the 1 micron. The speed for both processes is 150 rpm.

3.2.8 Metallographic

In order to investigate microstructure of the sample, metallurgy optical microscope and scanning electron microscope is used. The model for metallurgy optical microscope is Zeiss and the magnification employed in this experiment is 20X. For scanning electron microscope the magnification employed is 1kX.

3.2.9 Energy Dispersive X-ray

EDX is an analytical technique used for the elemental analysis or chemical characterization of a sample.

3.2.10 Micro hardness Test

To measure the hardness of the material, micro hardness tester is used. The model for this instrument is Leco LM 247 AT. The unit of hardness given by the tester is Vickers Pyramid Number, HV. The load used in this experiment is 100gf with the magnification of 50X. The hardness reading for each sample is taken at 7 different locations of the sample's surface. Then the average of the readings is calculated.

3.2.11 Shrinkage Test

Shrinkage test is the measure of percentage of dimensional change after sintering process by taking the change in dimension and divide the before sintering dimension.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Powder Characterization

Alumina and aluminum powder are characterized using Scanning Electron Microscope, Energy Dispersive X-ray and Particle Size Analyzer. These powders are characterized to determine particle size distribution, particle shape and oxide layer on the aluminum surface.

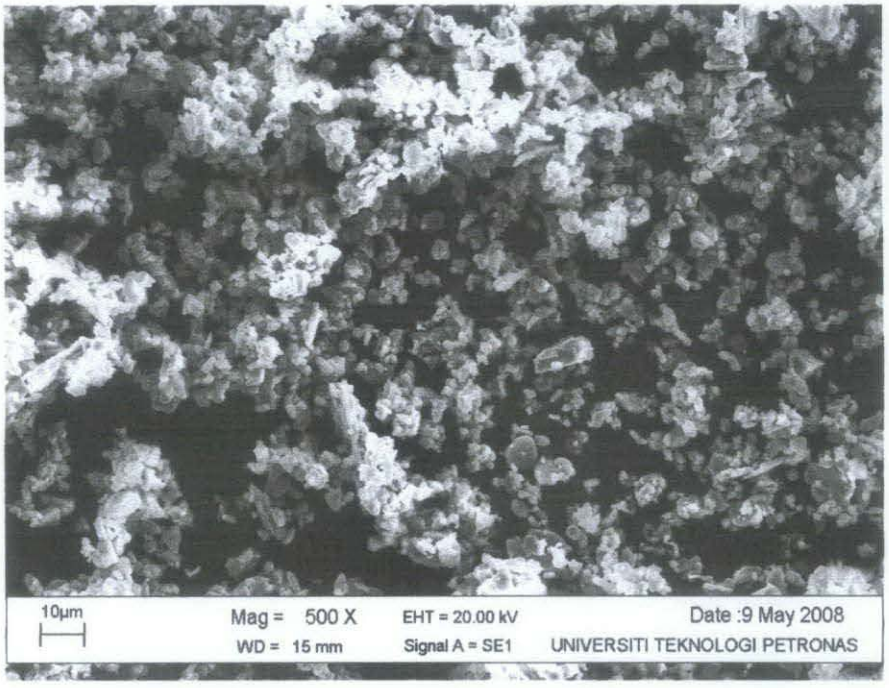


Figure 4-1(a): Particle shape of alumina powder with 500X Mag.

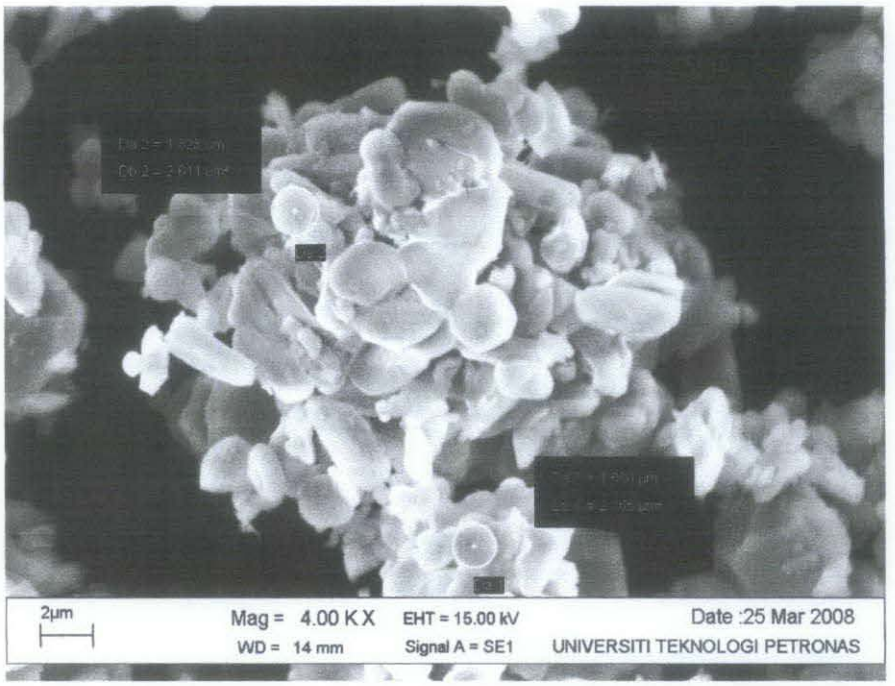


Figure 4-1(b): Particle shape of alumina powder with 4.0KX Mag.

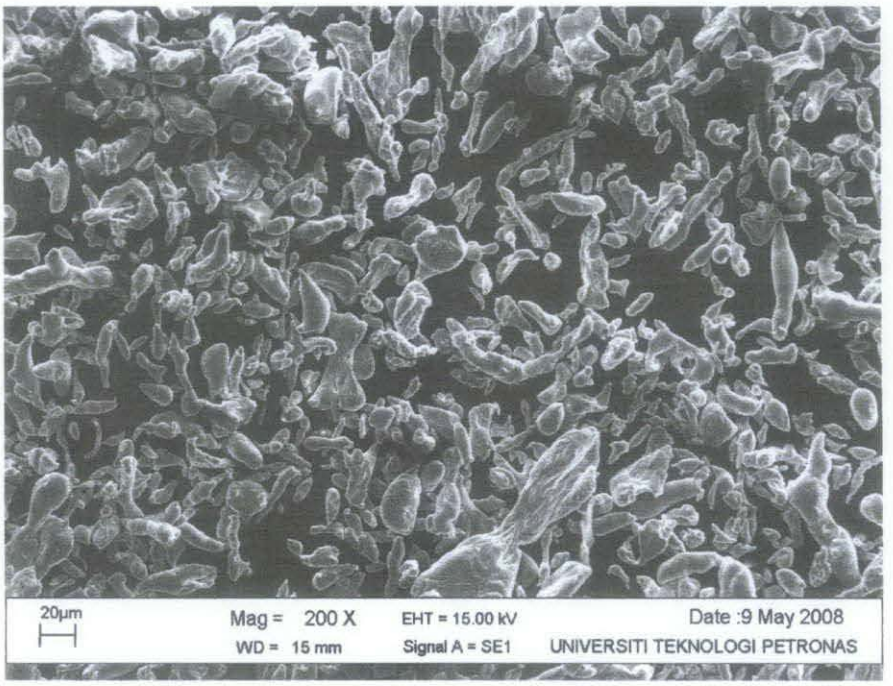


Figure 4-2(a): Particle shape of aluminum powder

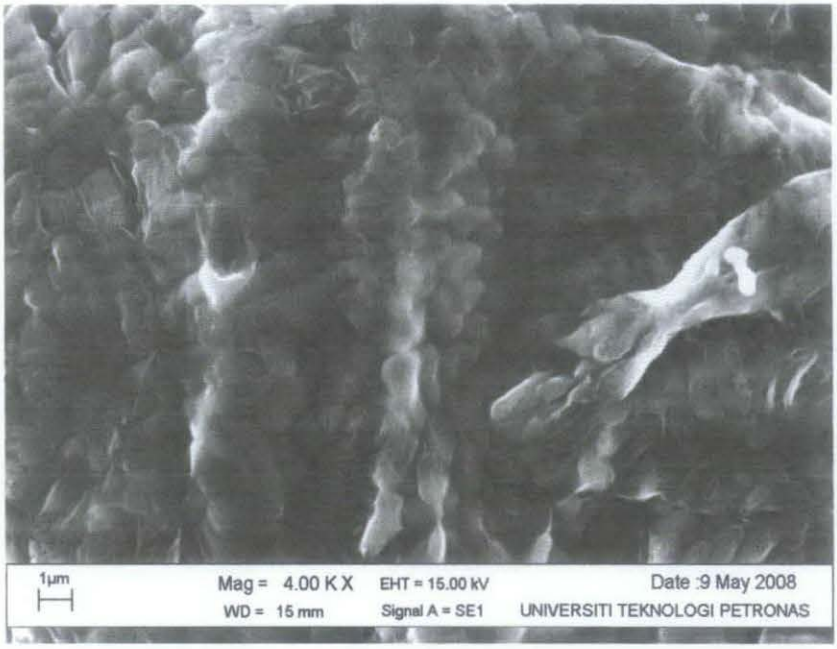


Figure 4-2(b): Morphology of aluminum powder

Referring to **Figure 4-1**, the shape of the alumina is the combination of spherical and irregular shape but mostly in spherical. As the alumina is spherical in shape, it is increased the packing density. The density improves as the particles approach a spherical shape and hence improve the mechanical properties of the composite [11].

It can be seen in **Figure 4-2** (a) (b) that the aluminum powder particle is the combination of irregular and round granule. The possible method of producing this powder is water atomization. If using gas atomization, the shape of powder particle tends to be spherical.

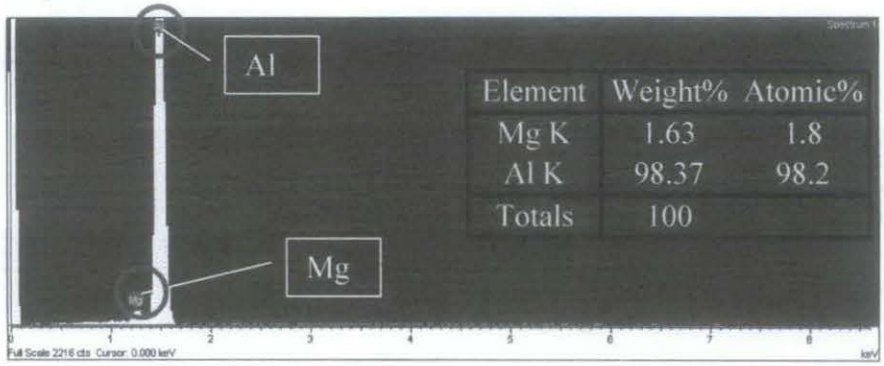


Figure 4-3: Chemical composition of aluminum powder

Figure 4-3 shows that the existence of aluminum element and magnesium after Energy Dispersive X-ray (EDX) analysis. This indicates that the aluminum powder is aluminum alloy because the present of magnesium element and also originally no oxide layer form on the aluminum surface.

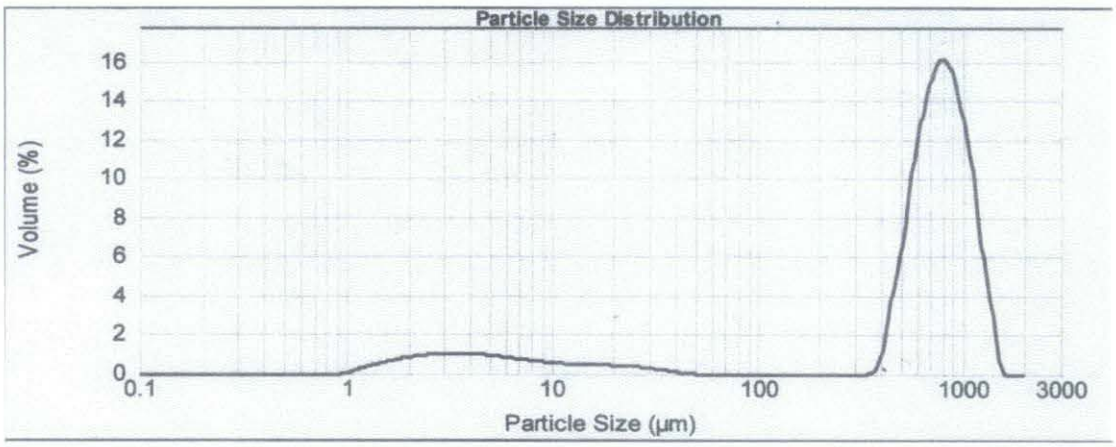


Figure 4-4: Alumina particle size distribution before grinding (as received).

Figure 4-4 shows the average size of as received alumina particles are distributed around 900µm and 1100µm but this size is quite large for powder metallurgy process. In order to reduce the alumina particle size, alumina powder was grinded using mortar grinder.

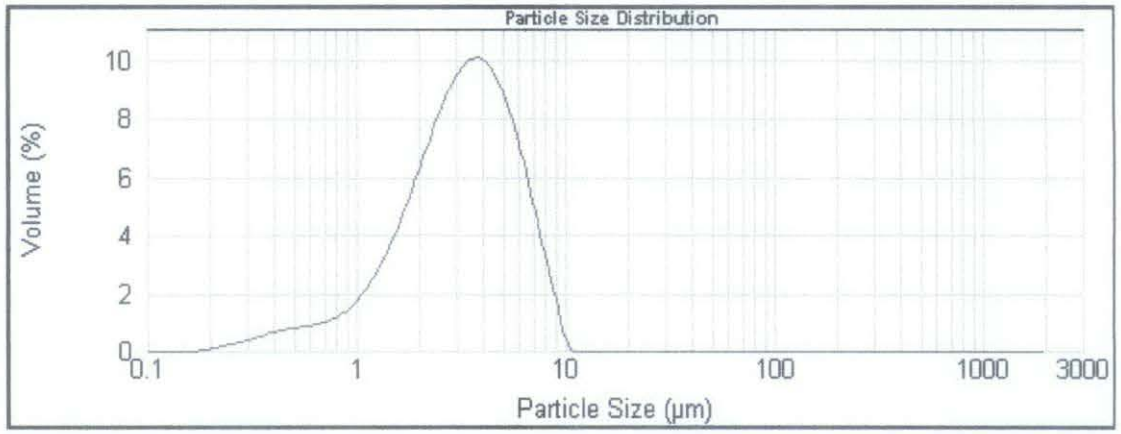


Figure 4-5: Alumina particle size distribution after grinding.

Figure 4-5 shows that alumina particle size is greatly reduced after underwent grinding process. The alumina particles are distributed around 1µm and 10µm.

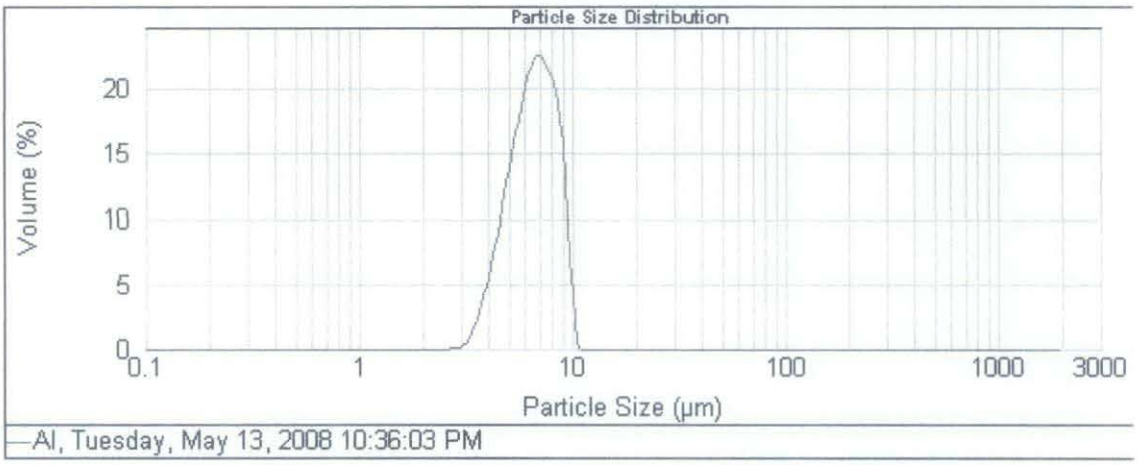
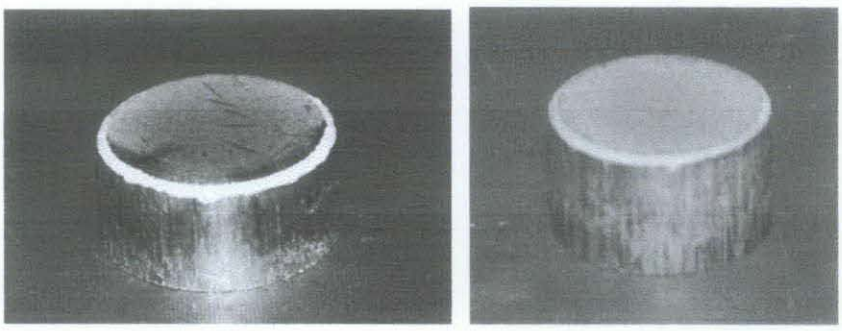


Figure 4-6: Aluminum particle size distribution

Figure 4-6 show the aluminum particle size distribution which the aluminum particles are distributed around 5µm and 12µm. This size is smaller compare to normal particle size for powder metallurgy process which is at ~150µm and there is no further grinding process needed for this powder.

4.2 Sintering of Composite Compact

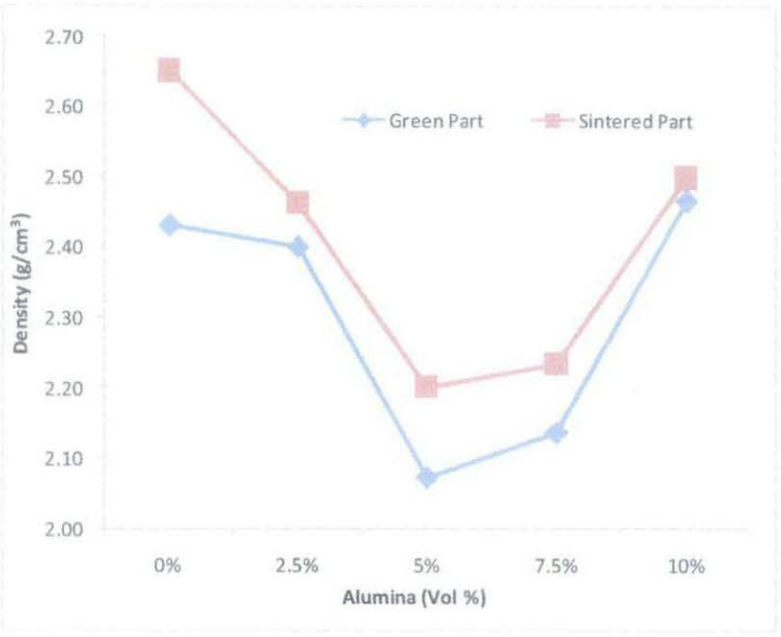
After compaction process, the samples were transferred to tube furnace. All formulations were sintered at temperature ranging 550°C to 650°C under nitrogen atmosphere for 2 hours. The nitrogen gas in the furnace is control by flow rate of 1100cm^3. Figure below are the green part and sintered part.



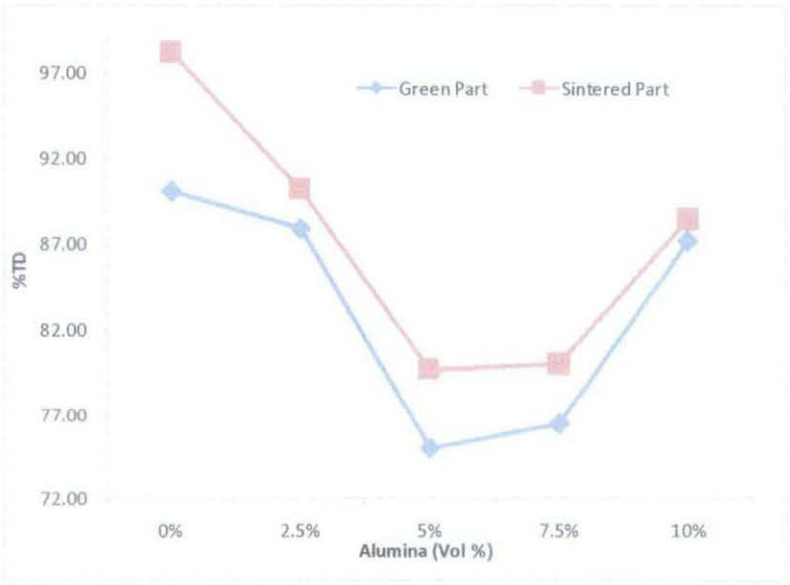
(a) (b)

Figure 4-7: Typical physical feature of (a) green part and (b) sintered part

4.2.1 Sintering at 650°C



(a)



(b)

Figure 4-8: Relationship between green and sintered density for sintered samples at 650°C for 2hr in Nitrogen atmosphere (a) Density vs. Alumina vol%, (b) Relative Theoretical Density vs. Alumina vol%.

The density and relative theoretical density of all sintered parts are increased after sintering process. Figure 4-8 shows that, at sintering temperature of 650°C pure aluminum powder achieved the highest density as well as the relative theoretical density. The theoretical density for pure aluminum powder achieved 98%. Sintered parts with 5% and 7.5% alumina volume fraction produced low density after sintering process. This indicated that these two sintered parts are the softest parts in this sintering temperature.

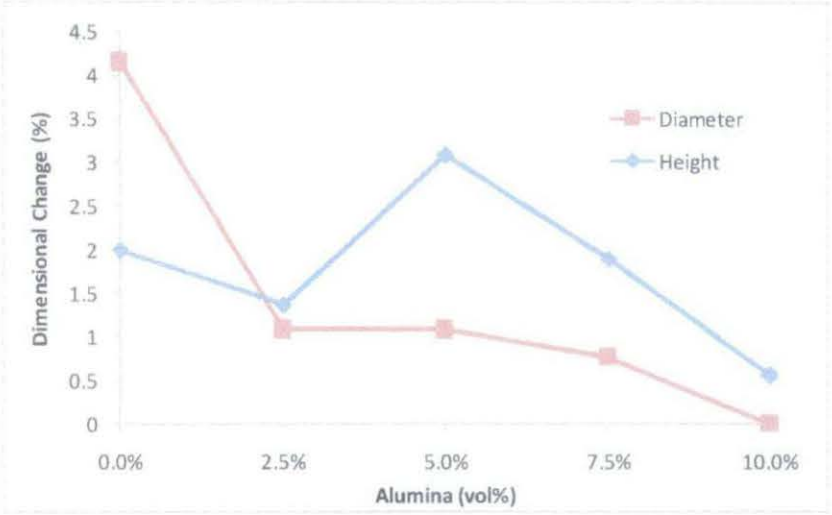


Figure 4-9: Shrinkage curve of the sintered part; sintering at 650°C for 2 hr under nitrogen atmosphere.

Figure 4-9 shows that shrinkage behavior of the sintered parts. As expected sintering under nitrogen atmosphere will produce shrinkage, all sintered parts indicated changes in dimension either the height or diameter. At 650°C, pure aluminum sintered part produced highest shrinkage both in diameter and height and the least producing shrinkage is sintered part with 10% alumina. The least shrinkage for sintered part with 10% alumina is due to the large reinforcing amount added.

Figure 4-10 shows the chemical element present in the sintered parts. From initial aluminum, magnesium and oxygen are presences. After sintering process EDX analysis shows that no impurities presences in this experiment. The highest concentration of

oxygen element indicated that large alumina particle distribution existed. Trend shows that high concentration of oxygen resulted least dimensional change to the composite.

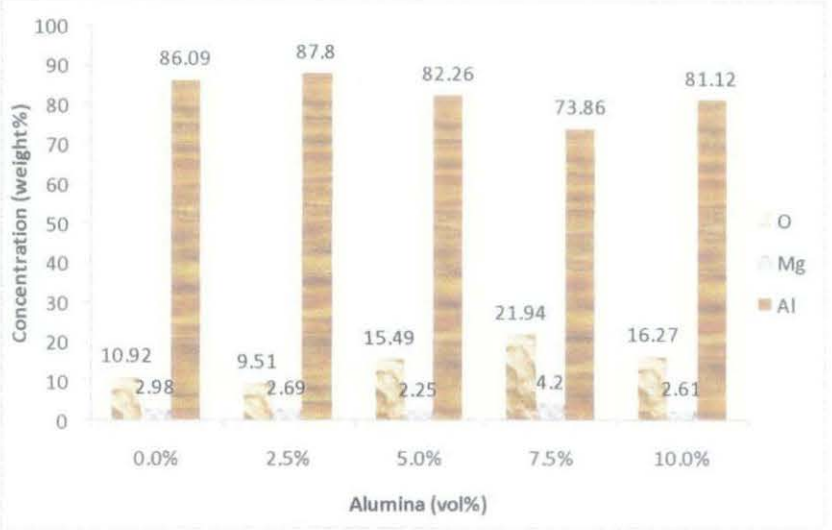


Figure 4-10: EDX analysis of sintered part sintering at 650°C for 2hr under nitrogen atmosphere. Aluminum, magnesium and oxygen content in investigated materials.

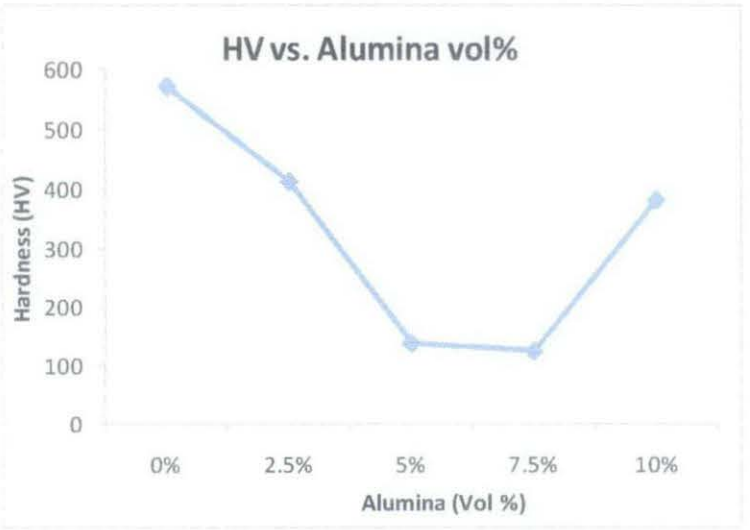


Figure 4-11: Relationship between Hardness value of sintered parts and Alumina vol%

Figure 4-11shows the hardness curve of the sintered parts. It shows that low density of sintered part can result in low hardness value. Sintered parts with 5% and 7.5% alumina volume result in low hardness value.

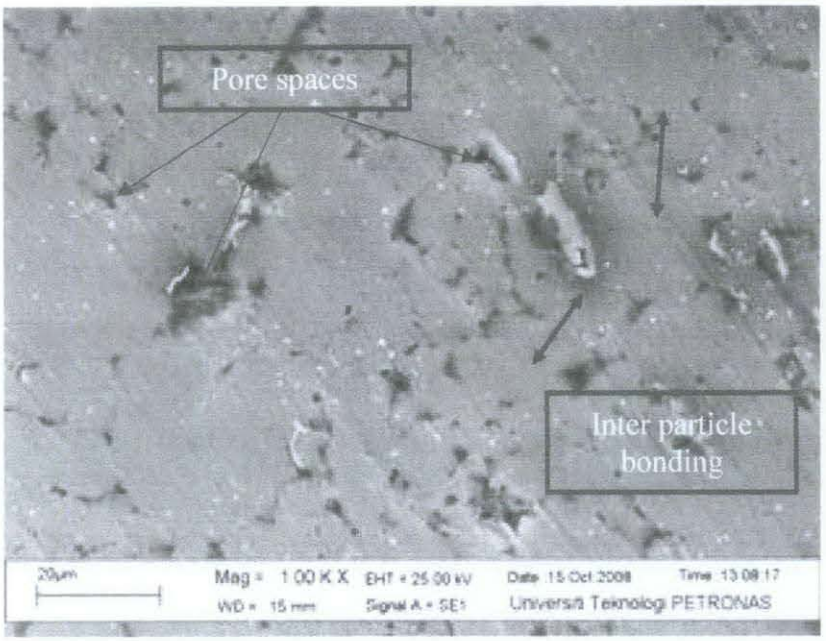


Figure 4-12: SEM Micrograph with 1.0KX magnification of pure aluminum sintered at 650°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

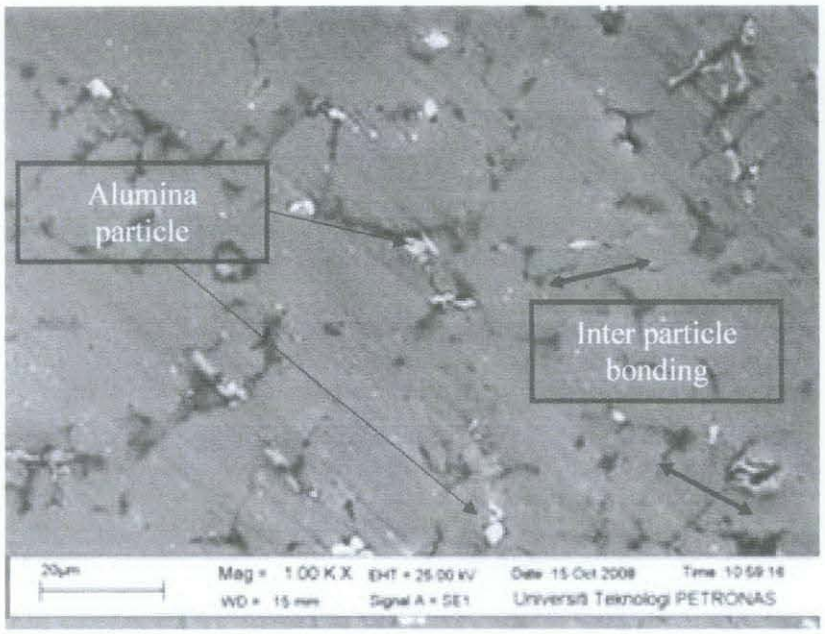


Figure 4-13 (a): SEM Micrograph with 1.0KX magnification of Al-97.5%vol; Alumina-2.5%vol sintered at 650°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

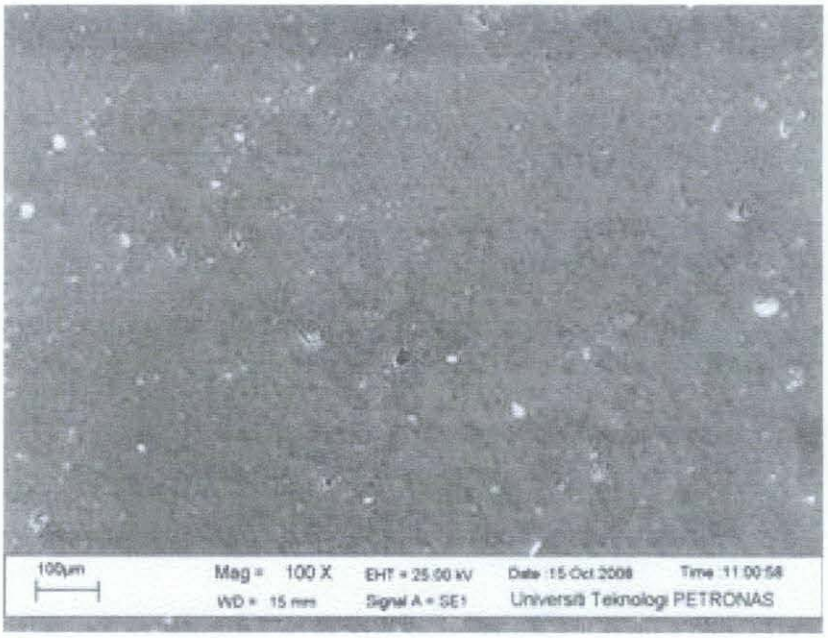


Figure 4-13 (b): SEM Micrograph with 100X magnification of Al-97.5%vol; Alumina-2.5%vol sintered samples at 650°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

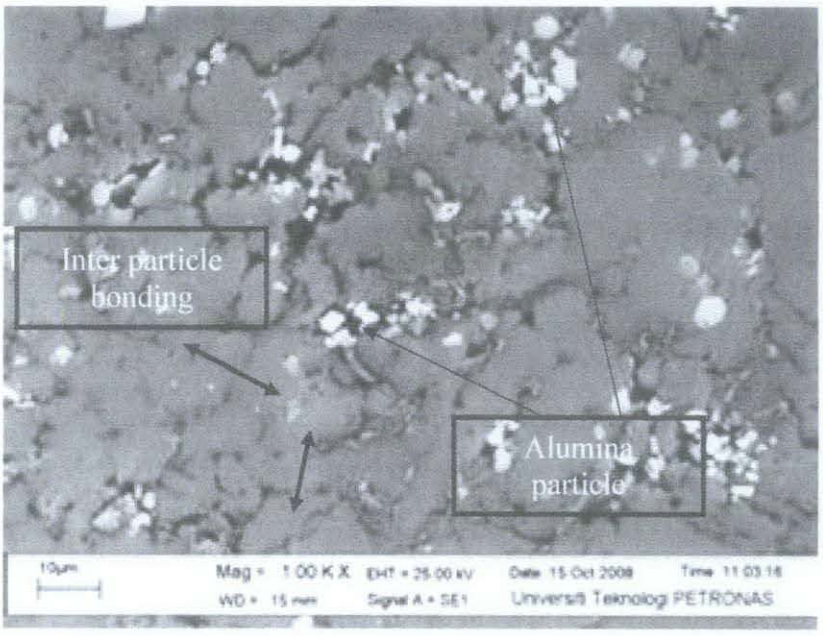


Figure 4-14 (a): SEM Micrograph with 1.0KX magnification of Al-95%vol; Alumina-5%vol sintered samples at 650°C for 2hr under nitrogen atmosphere shows porosity and particle bond

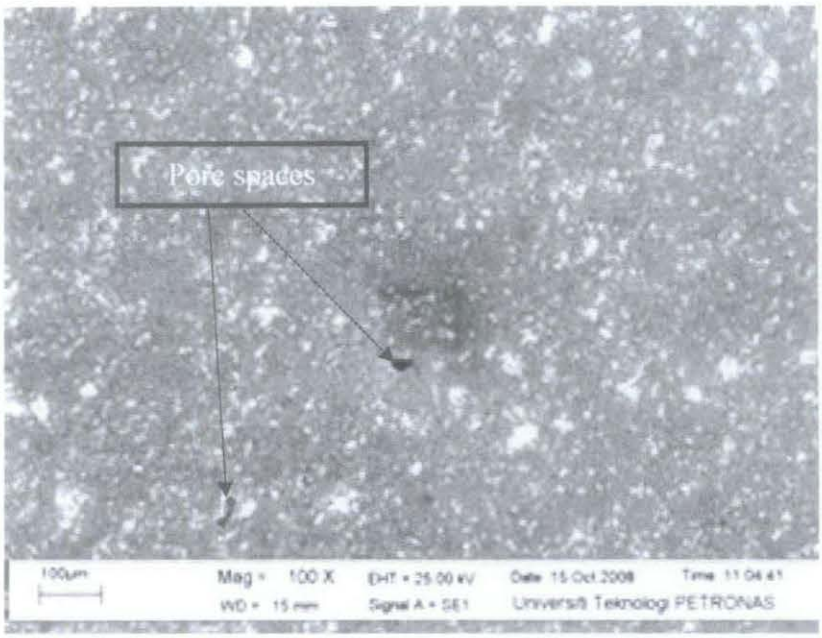


Figure 4-14 (b): SEM Micrograph with 100X magnification of Al-95%vol; Alumina-5%vol sintered samples at 650°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

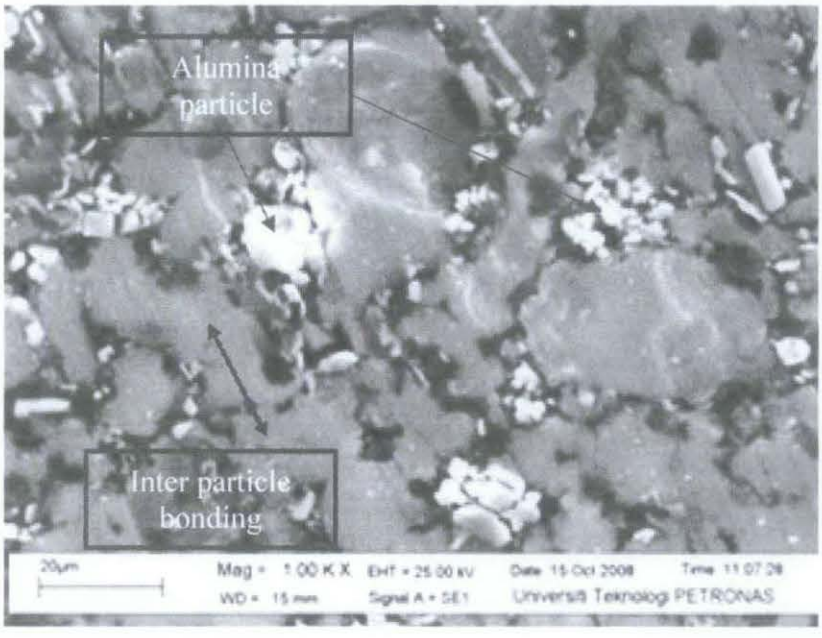


Figure 4-15 (a): SEM Micrograph with 1.0KX magnification of Al-92.5%vol; Alumina-7.5%vol sintered samples at 650°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

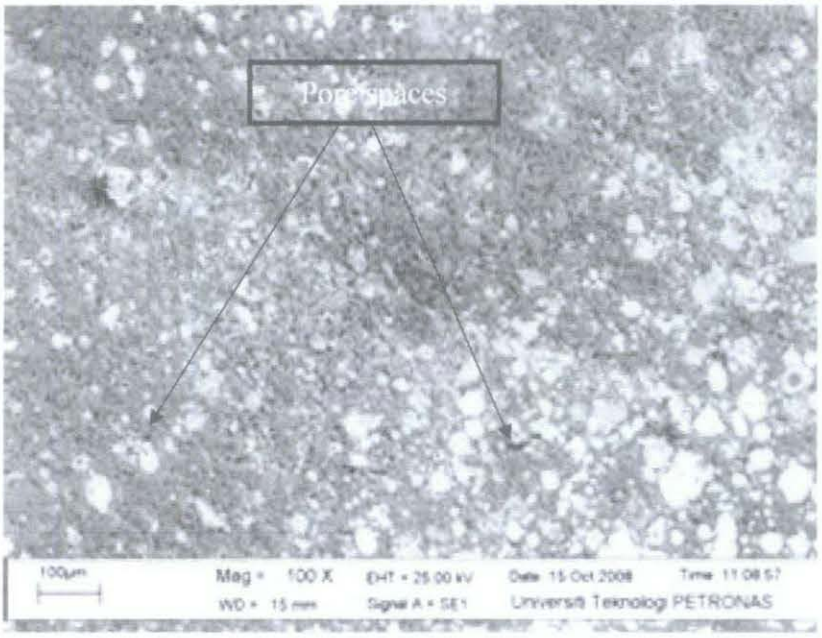


Figure 4-15 (b): SEM Micrograph with 100X magnification of Al-97.5%vol; Alumina-2.5%vol sintered samples at 650°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

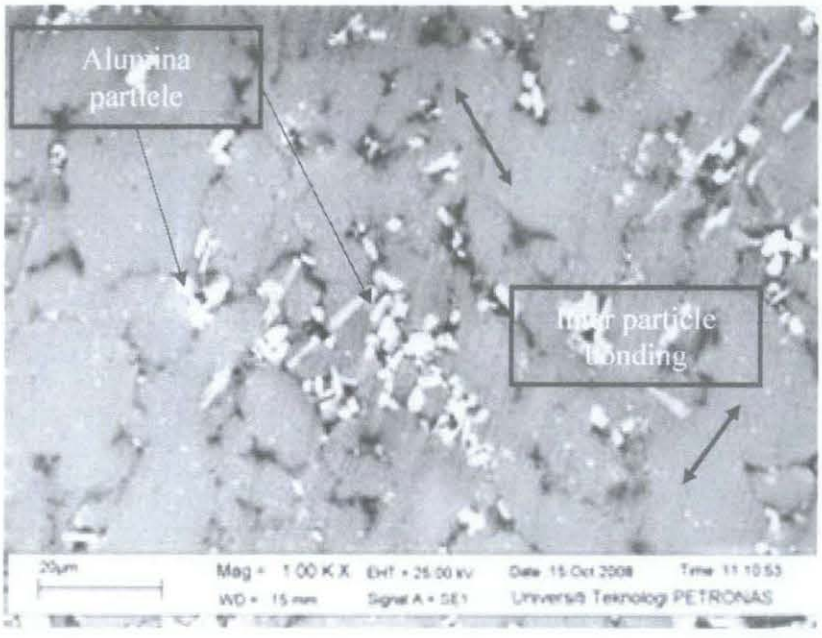


Figure 4-16 (a): SEM Micrograph with 1.0KX magnification of Al-90%vol; Alumina-10%vol sintered samples at 650°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

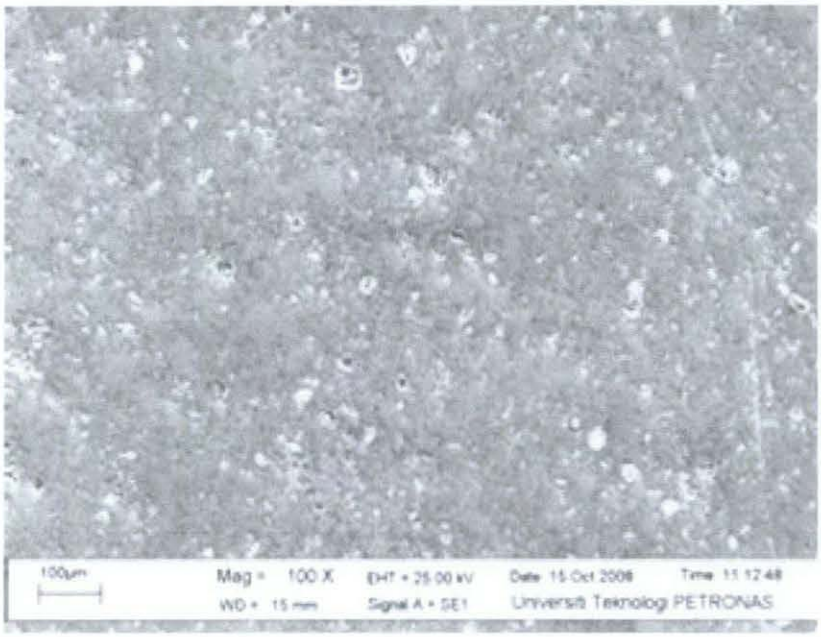
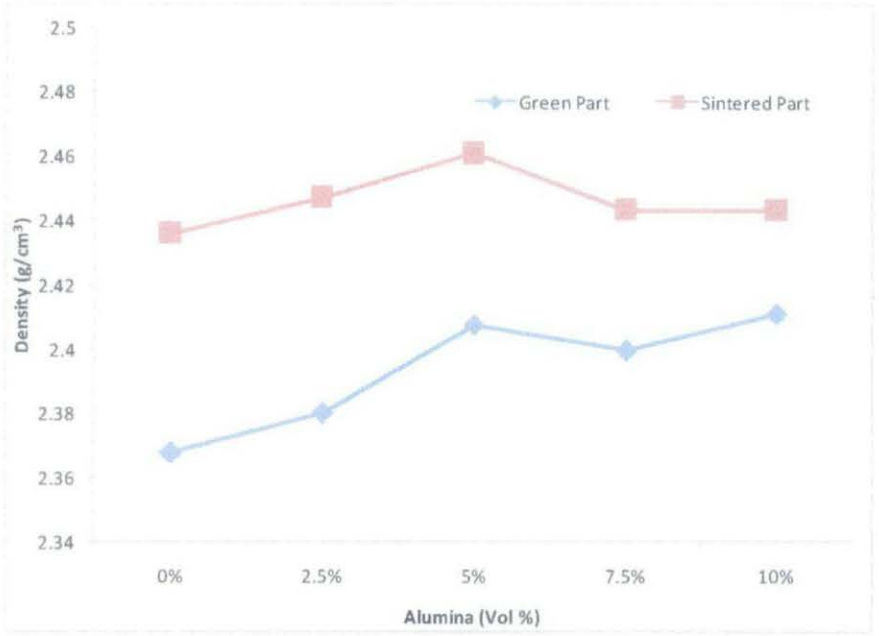


Figure 4-16 (b): SEM Micrograph with 100X magnification of Al-90%vol; Alumina-10%vol sintered samples at 650°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

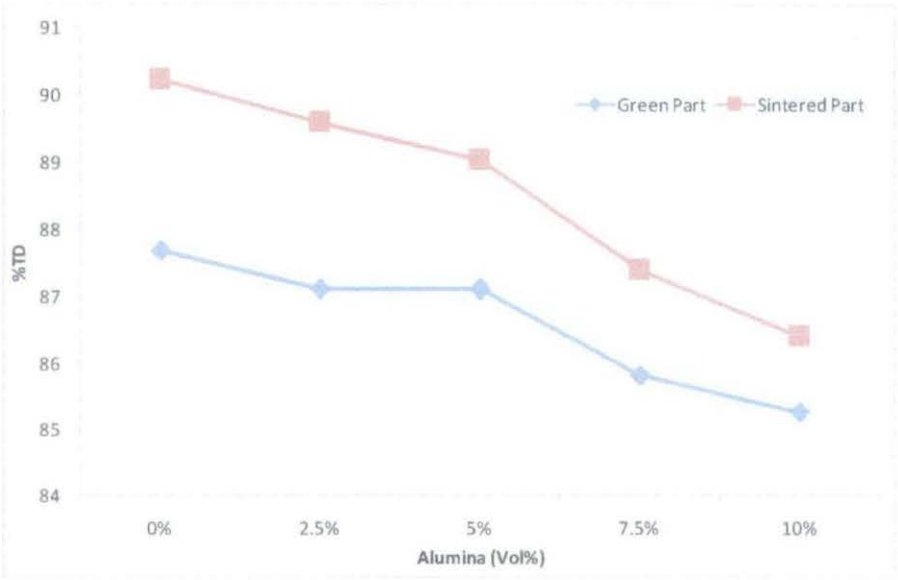
From the hardness curves, the reason for this behavior may lie in microstructure. Sintering at 650°C, sintered parts with 5% and 7.5% alumina microstructure tend to formed coarse microstructure. The micrograph also shows poor inter particle bonding between aluminum-aluminum particle and aluminum-alumina particle. Sintered parts with 2.5%, 10% and pure aluminum micrograph shows good inter particle bonding as well as fine microstructure.

Figure 4-12-4-16 shows the micrograph of sintered parts. As density increased large pore was eliminated and the distribution of alumina particle is filled up the pore spaces along the grain boundary of aluminum particle bonding. As the refer to sintered part of alumina 5% and 7.5% micrograph Figure 4-14 (a)(b) and 4-15 (a)(b), a lot of large pore spaces emerged.

4.3.2 Sintering at 625°C



(a)



(b)

Figure 4-17: Relationship between green and sintered density for sintered samples at 625 °C for 2hr in Nitrogen atmosphere (a) Density vs. Alumina vol%, (b) Relative Theoretical Density vs. Alumina vol%.

The density and relative theoretical density of all sintered parts are increased after sintering process. Figure 4-17 shows that, at sintering temperature of 625°C pure aluminum powder achieved the highest density as well as the relative theoretical density. The theoretical density for pure aluminum powder achieved 90%. Sintered parts with 10% alumina volume fraction produced the lowest density after sintering process. This indicated that this sintered part is the softest parts in this sintering temperature.

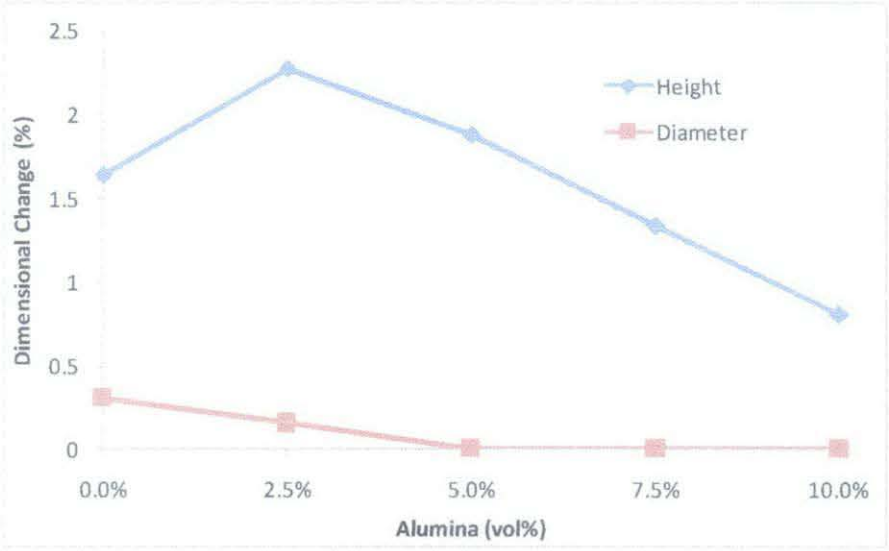


Figure 4-18: Shrinkage curve of the sintered part; sintering at 625°C for 2 hr under nitrogen atmosphere.

Figure 4-18 shows that shrinkage behavior of the sintered parts. As expected sintering under nitrogen atmosphere will produce shrinkage, all sintered parts indicated changes in dimension either the height or diameter. At 625°C, pure aluminum sintered part produced highest shrinkage both in diameter and height and the least producing shrinkage is sintered part with 10% alumina. The least shrinkage for sintered part with 10% alumina is due to the large reinforcing amount added.

Figure 4-19 shows the chemical element present in the sintered parts. From initial aluminum, magnesium and oxygen are presences. After sintering process EDX analysis shows that no impurities appeared in this experiment. The highest concentration of

oxygen element indicated that large alumina particle distribution existed. Trend shows that high concentration of oxygen resulted little dimensional change to the composite.

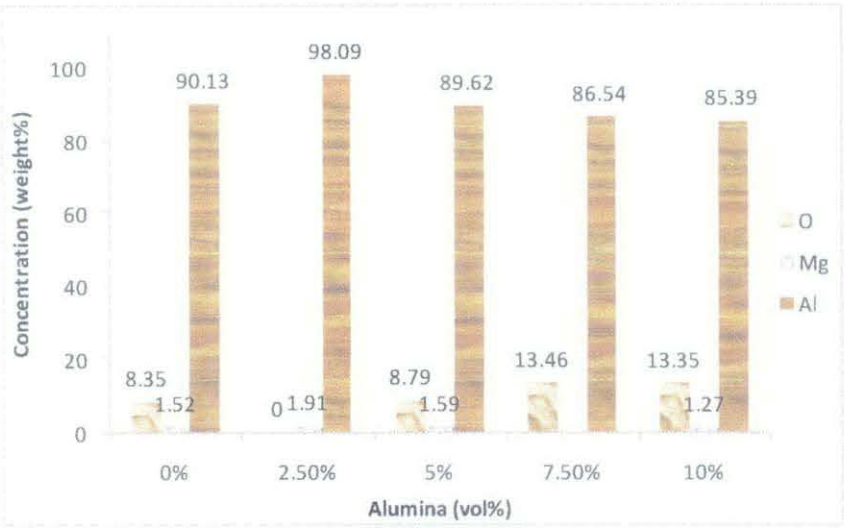


Figure 4-19: EDX analysis of sintered part sintering at 625°C for 2hr under nitrogen atmosphere. Aluminum, magnesium and oxygen content in investigated materials.

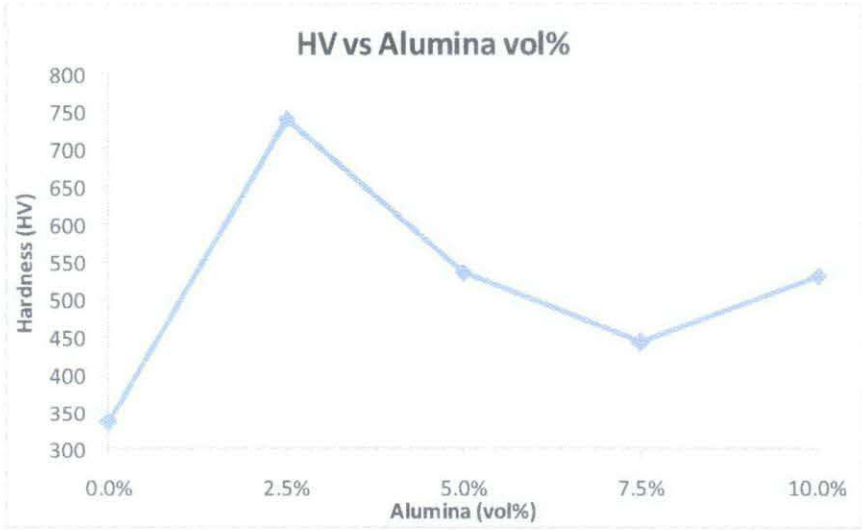


Figure 4-20: Relationship between Hardness value of sintered parts and Alumina %vol

Figure 4-20 shows the hardness curve of the sintered parts. It shows that low density of sintered part can resulted in low hardness value. Sintered parts with 7.5% alumina and pure aluminum result in low hardness value.

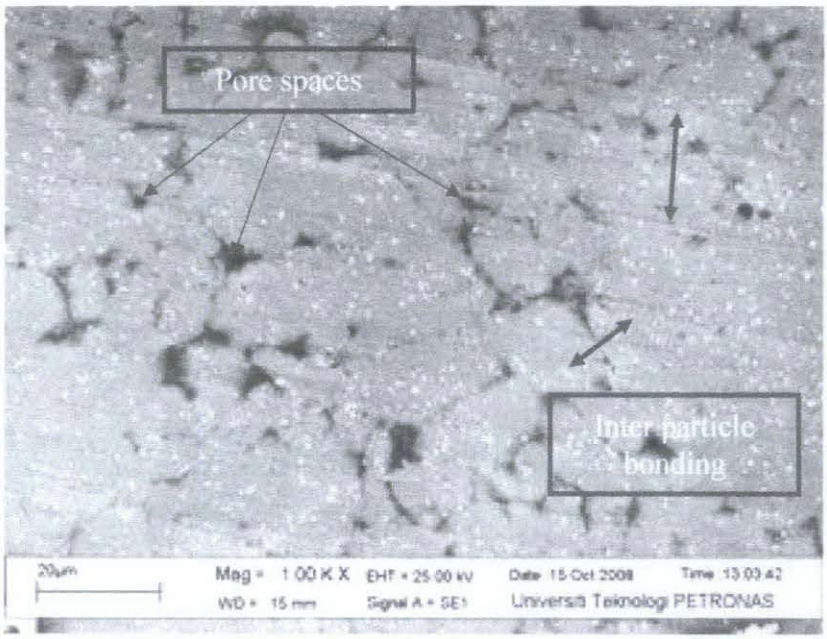


Figure 4-21: SEM Micrograph with 1.0KX magnification of pure aluminum sintered at 625°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

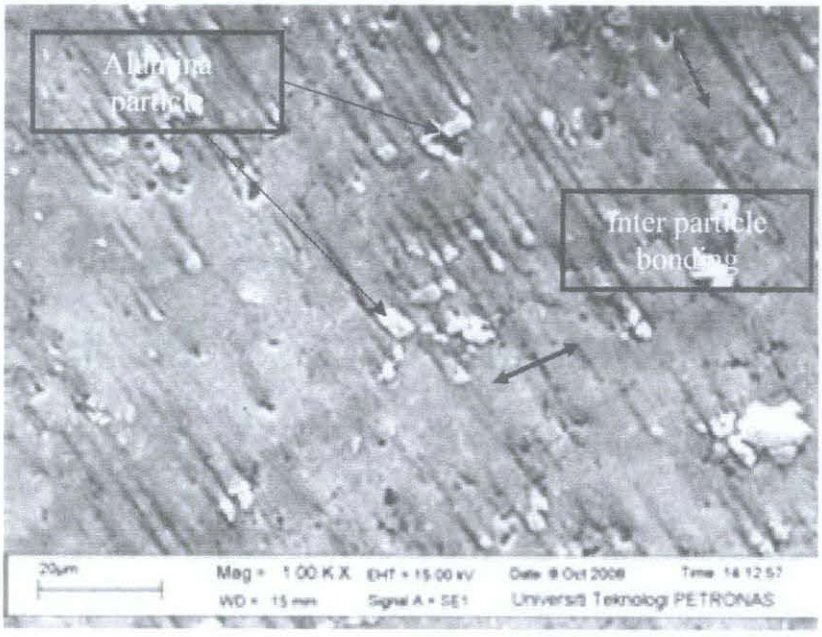


Figure 4-22 (a): SEM Micrograph with 1.0KX magnification of Al-97.5%vol; Alumina-2.5%vol sintered at 625°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

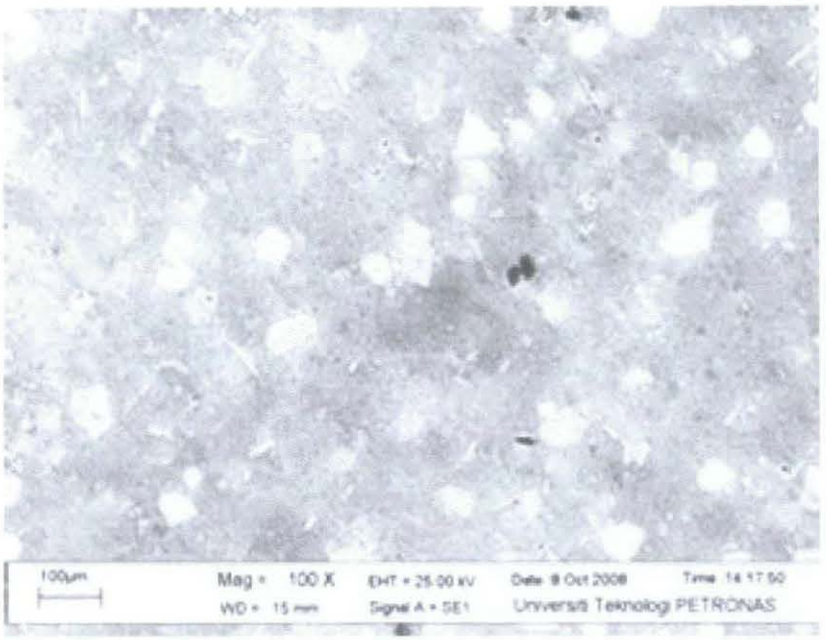


Figure 4-22 (b): SEM Micrograph with 100X magnification of Al-97.5%vol; Alumina-2.5%vol sintered samples at 625°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

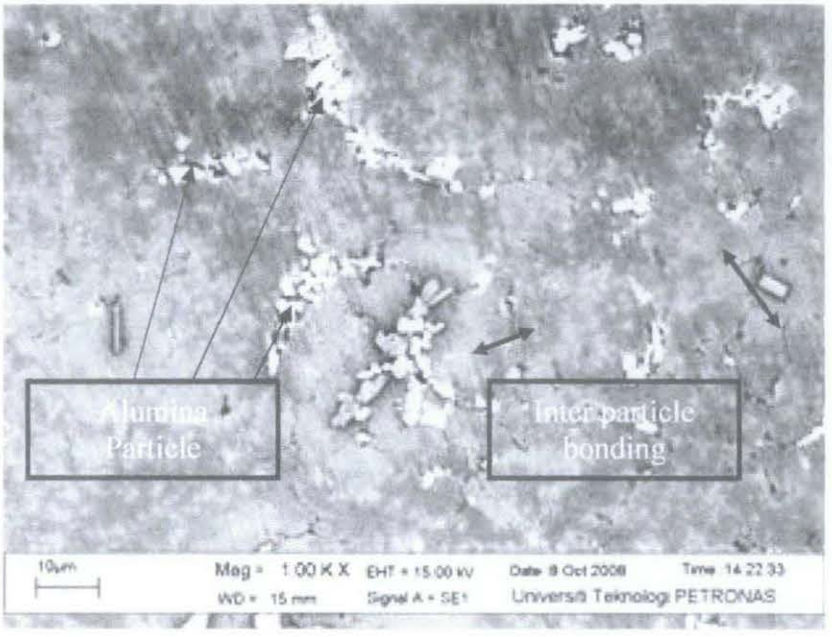


Figure 4-23 (a): SEM Micrograph with 1.0KX magnification of Al-95%vol; Alumina-5%vol sintered samples at 625°C for 2hr under nitrogen atmosphere shows porosity and particle bond

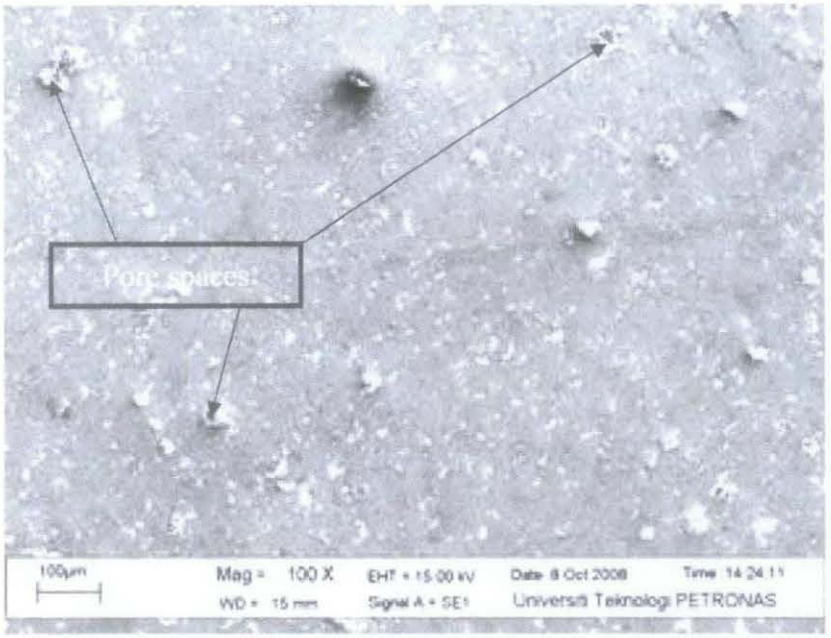


Figure 4-23 (b): SEM Micrograph with 100X magnification of Al-95%vol; Alumina-5%vol sintered samples at 625°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

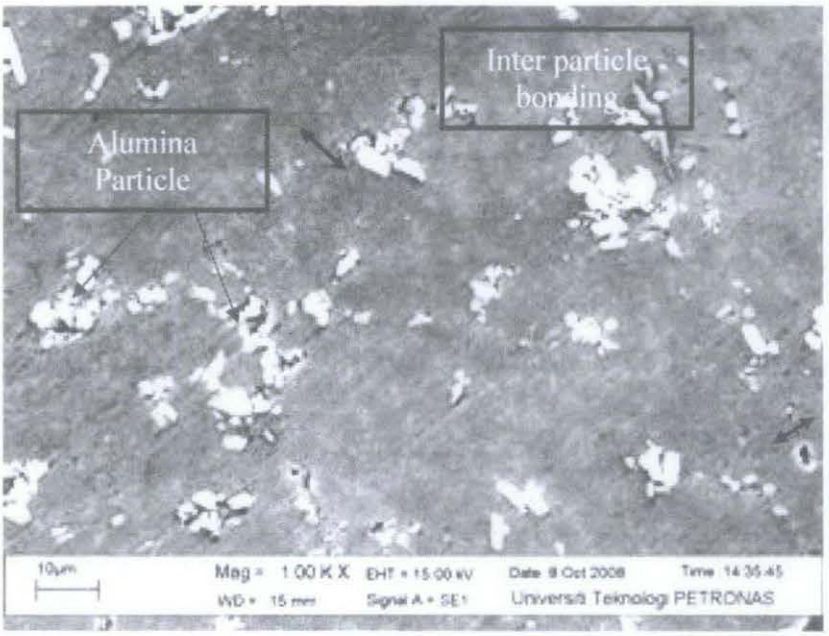


Figure 4-24 (a): SEM Micrograph with 1.0KX magnification of Al-92.5%vol; Alumina-7.5%vol sintered samples at 625°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

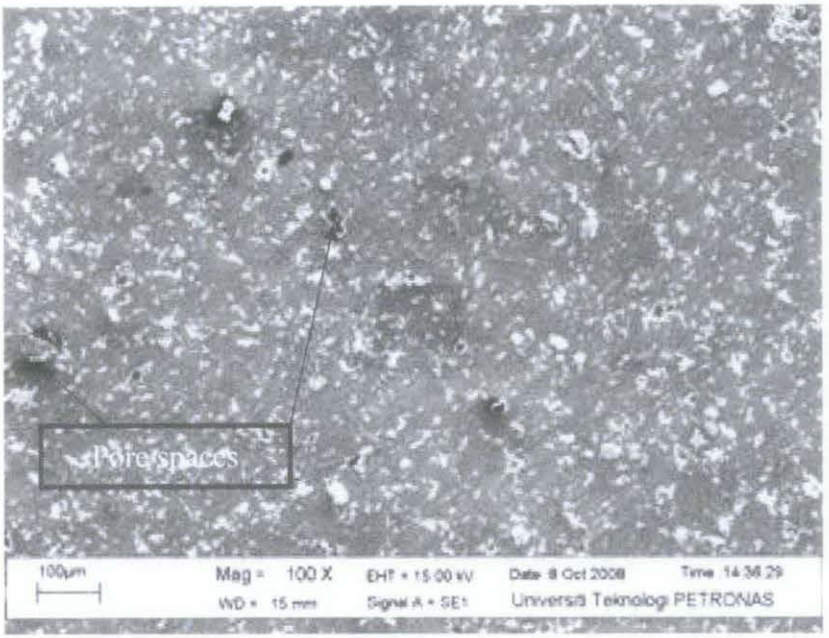


Figure 4-24 (b): SEM Micrograph with 100X magnification of Al-97.5%vol; Alumina-2.5%vol sintered samples at 625°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

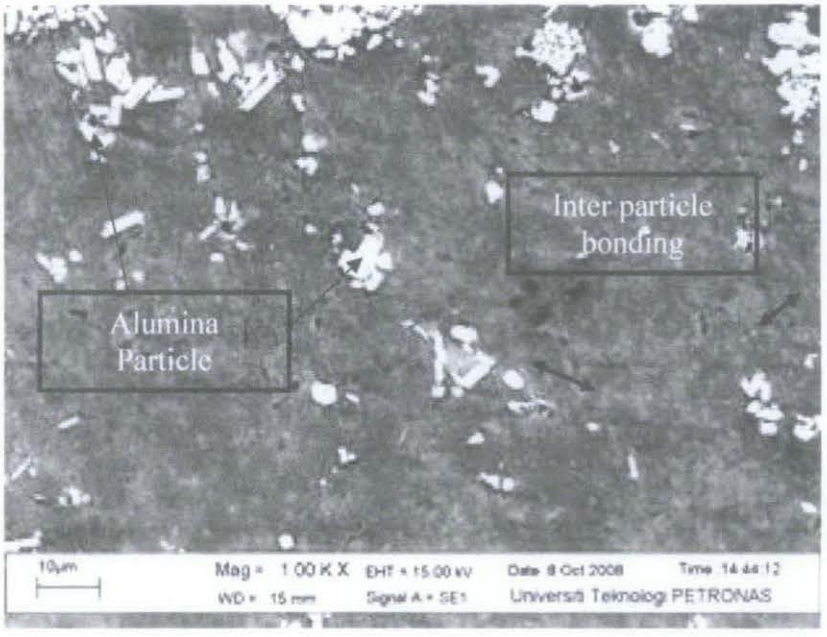


Figure 4-25 (a): SEM Micrograph with 1.0KX magnification of Al-90%vol; Alumina-10%vol sintered samples at 625°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

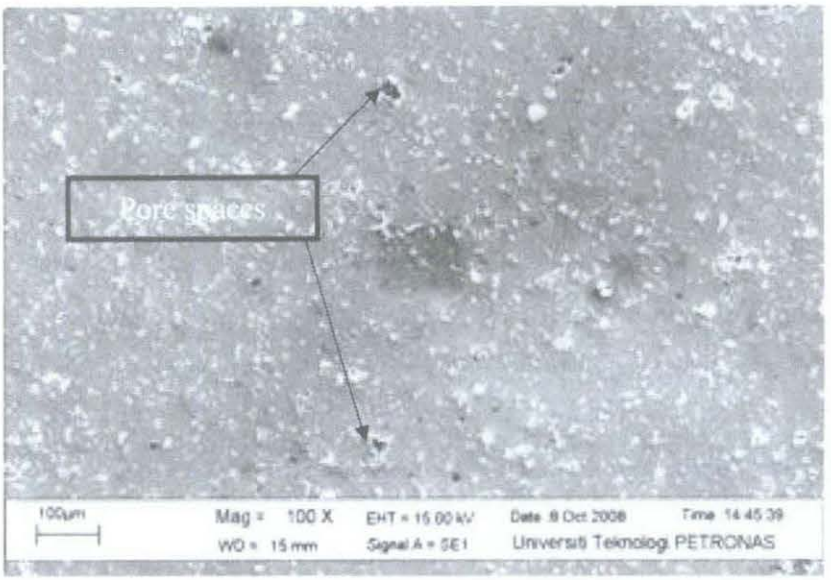
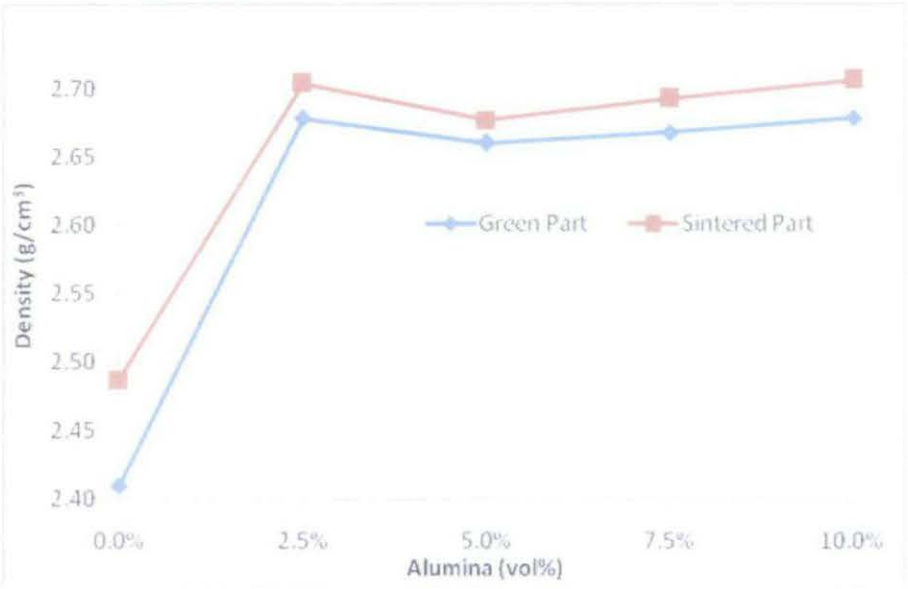


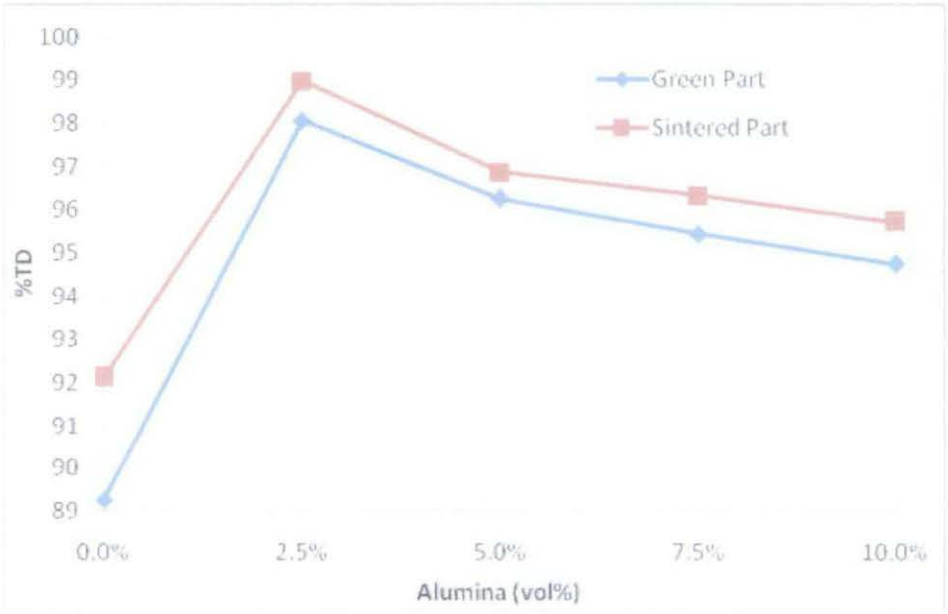
Figure 4-25 (b): SEM Micrograph with 100X magnification of Al-90%vol; Alumina-10%vol sintered samples at 625°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

From the hardness curves, the reason for this behavior may lie in microstructure. Sintered parts with 2.5% alumina shows that the alumina particle is well distributed and shows the good inter particle bonding between aluminum and alumina. Pure aluminum micrograph shows that the pore spaces present around the grain boundary. For the sintered part with 7.5% alumina micrograph shows that the alumina particles filled up the pore spaces and suppress surface particle contact to form bonding. It is also shows that large pores also appeared. All sintered parts shows fine microstructure and the bonding formed is better than at sintering temperature of 625°C that resulted high value of hardness.

4.2.3 Sintering at 580°C



(a)



(b)

Figure 4-26: Relationship between green and sintered density for sintered samples at 580 °C for 2hr in Nitrogen atmosphere (a) Density vs. Alumina vol%, (b) Relative Theoretical Density vs. Alumina vol%.

The density and relative theoretical density of all sintered parts are increased after sintering process. Figure 4-26 shows that, at sintering temperature of 580°C sintered part with 2.5% alumina achieved the highest density as well as the relative theoretical density. The theoretical density for sintered part with 2.5% achieved 98% TD. Pure aluminum sintered part produced low density after sintering process. This indicated that this sintered parts are the softest parts in this sintering temperature.

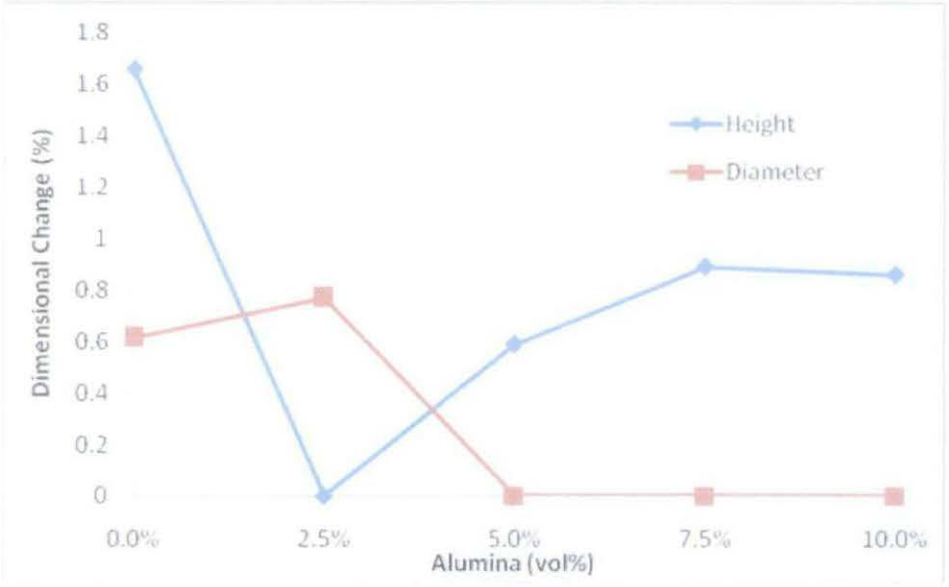


Figure 4-27: Shrinkage curve of the sintered part; sintering at 580°C for 2 hr under nitrogen atmosphere.

Figure 4-27 shows that shrinkage behavior of the sintered parts. As expected sintering under nitrogen atmosphere will produce shrinkage, all sintered parts indicated changes in dimension either the height or diameter. At 580°C, pure aluminum sintered part produced the highest shrinkage both in diameter and height and the least producing shrinkage is sintered part with 10% alumina. The least shrinkage for sintered part with 10% alumina is due to the large reinforcing amount added.

Figure 4-28 shows the chemical element present in the sintered parts. From initial experiment aluminum, magnesium and oxygen presence. After sintering process, EDX analysis shows that no impurities presences in this experiment. The highest

concentration of oxygen element indicated that large alumina particle distribution existed. Trend shows that high concentration of oxygen resulted least dimensional change to the composite.

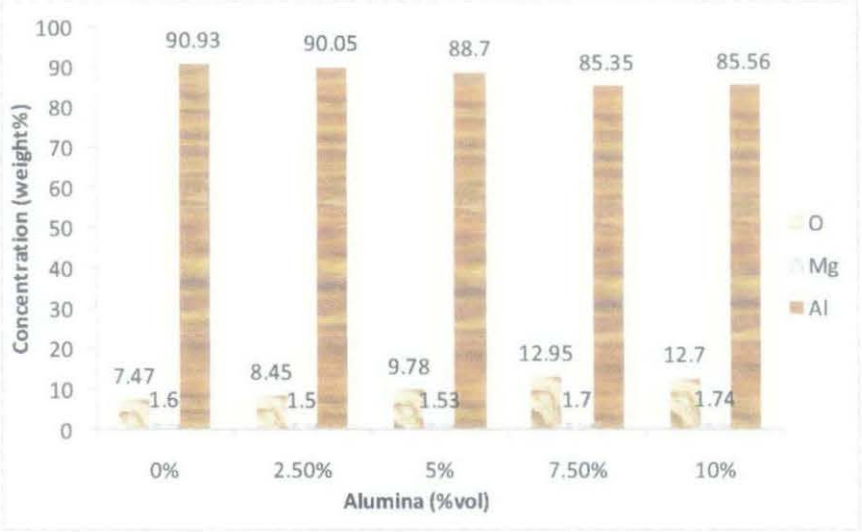


Figure 4-28: EDX analysis of sintered part sintering at 580°C for 2hr under nitrogen atmosphere. Aluminum, magnesium and oxygen content in investigated materials.

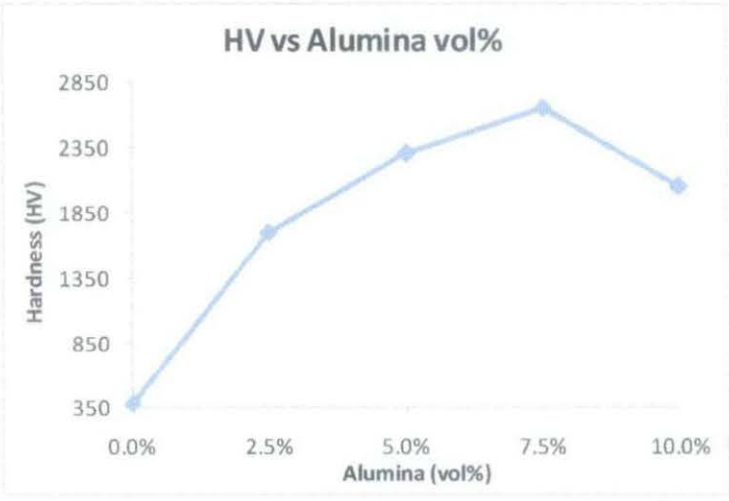


Figure 4-29: Relationship between Hardness value of sintered parts and Alumina %vol.

Figure 4-29 shows the hardness curve of the sintered parts. It shows that low density of sintered part can result in low hardness value. All Sintered part mixed with alumina powder shows high hardness value.

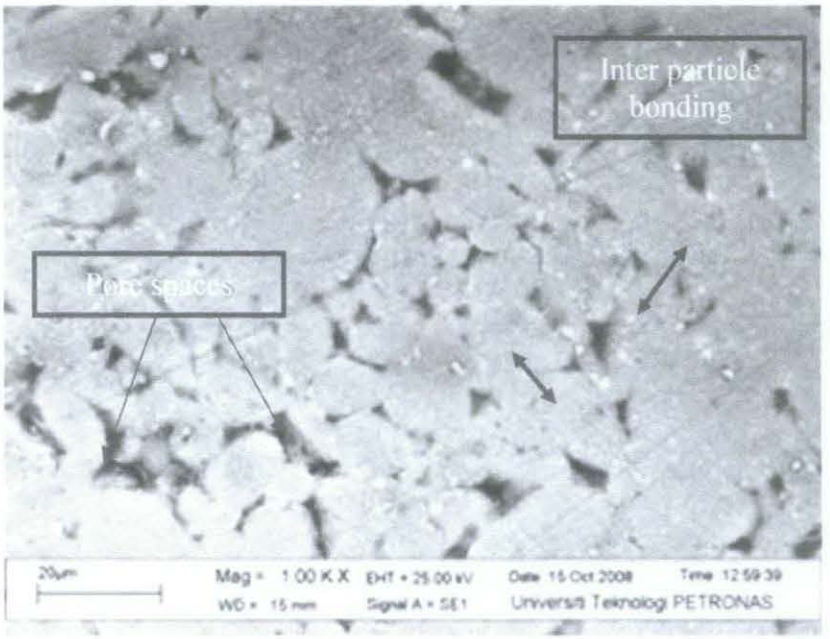


Figure 4-30: SEM Micrograph with 1.0KX magnification of pure aluminum sintered at 580°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

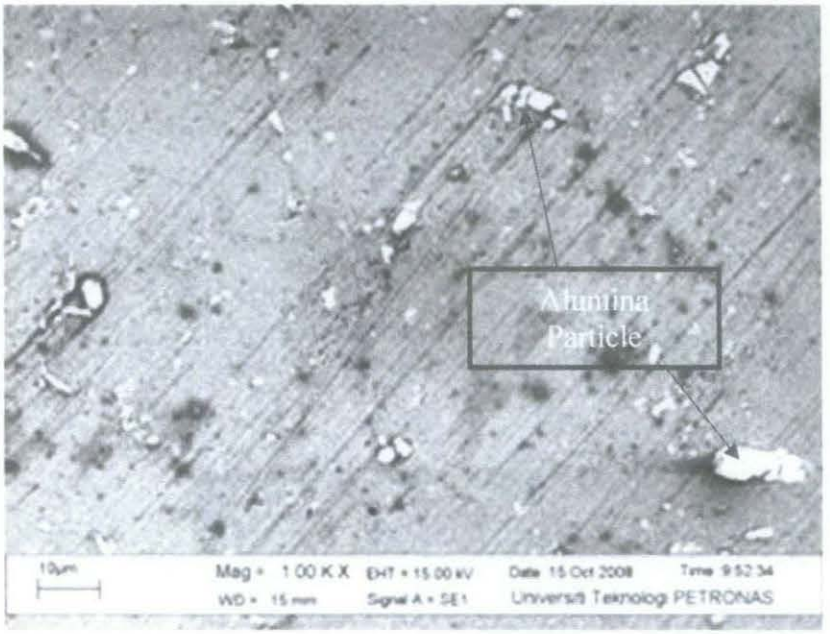


Figure 4-31 (a): SEM Micrograph with 1.0KX magnification of Al-97.5%vol; Alumina-2.5%vol sintered at 580°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

Figure 4-31 (b): SEM Micrograph with 100X magnification of Al-97.5%vol; Alumina-2.5%vol sintered samples at 580°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

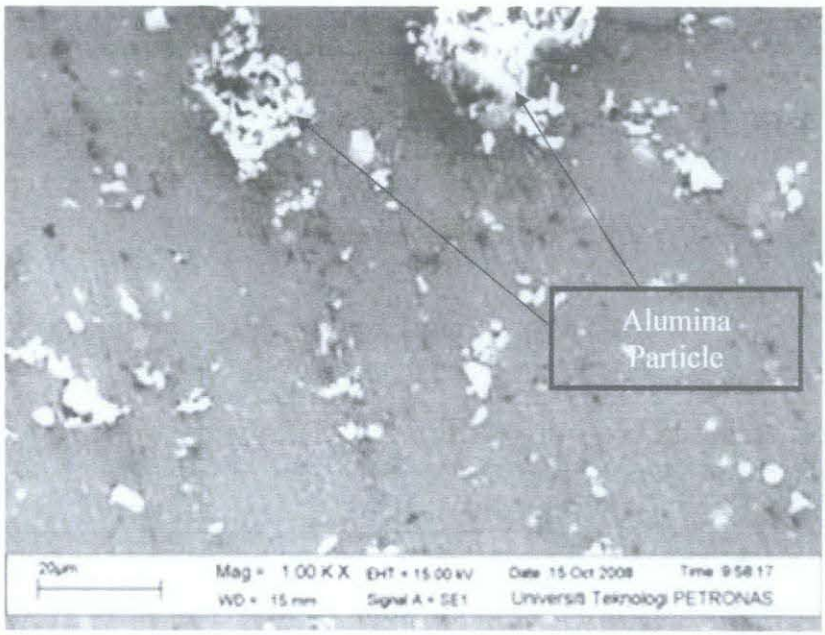


Figure 4-32 (a): SEM Micrograph with 1.0KX magnification of Al-95%vol; Alumina-5%vol sintered samples at 580°C for 2hr under nitrogen atmosphere shows porosity and particle bond

Figure 4-32 (b): SEM Micrograph with 100X magnification of Al-95%vol; Alumina-5%vol sintered samples at 580°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

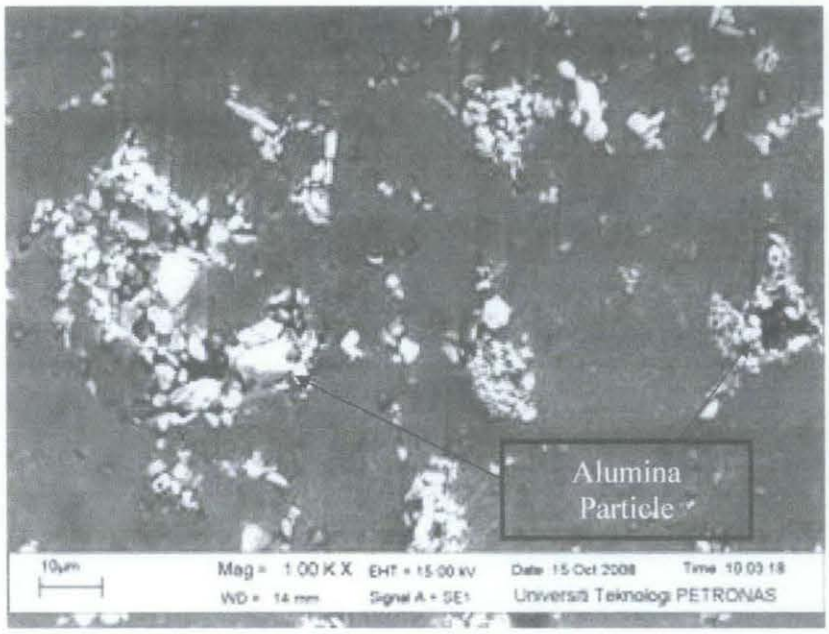


Figure 4-33 (a): SEM Micrograph with 1.0KX magnification of Al-92.5%vol; Alumina-7.5%vol sintered samples at 580°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

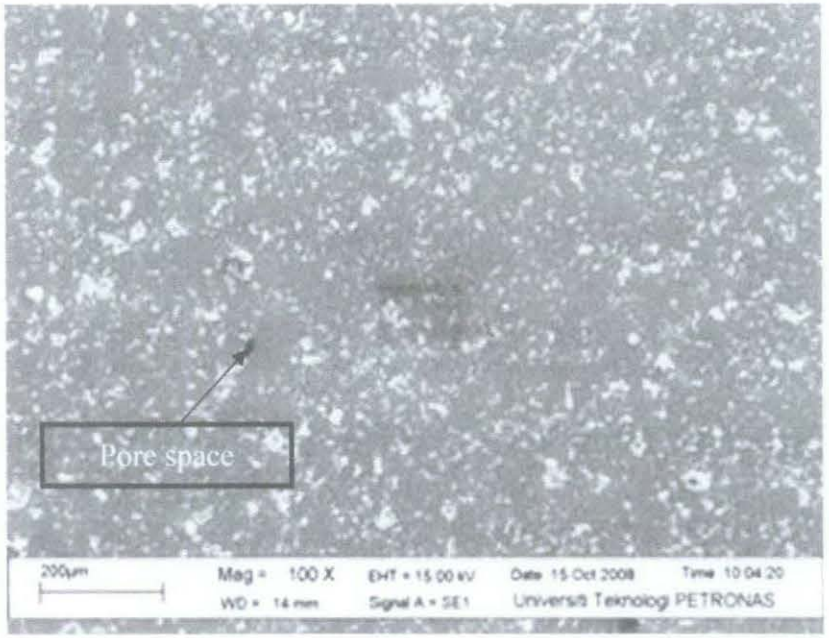


Figure 4-33 (b): SEM Micrograph with 100X magnification of Al-97.5%vol; Alumina-2.5%vol sintered samples at 580°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

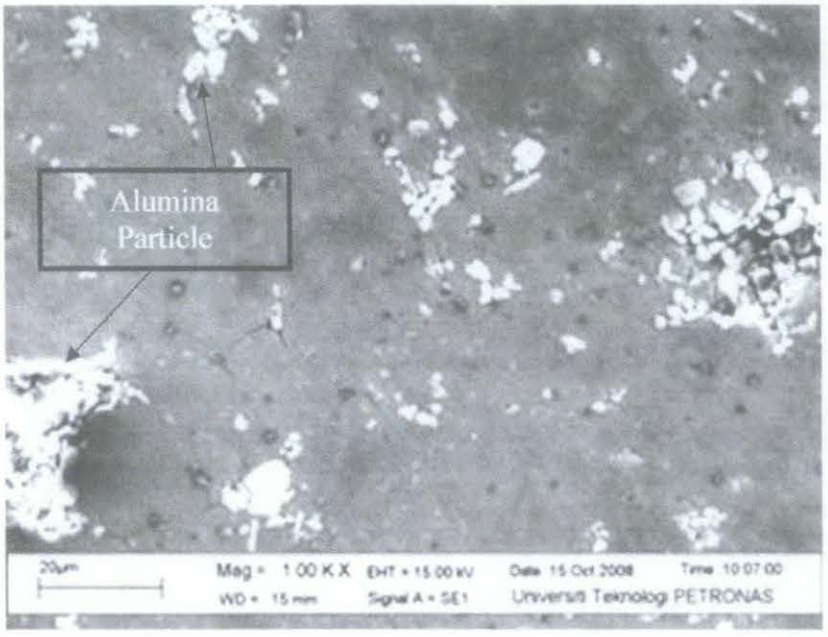


Figure 4-34 (a): SEM Micrograph with 1.0KX magnification of Al-90%vol; Alumina-10%vol sintered samples at 580°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

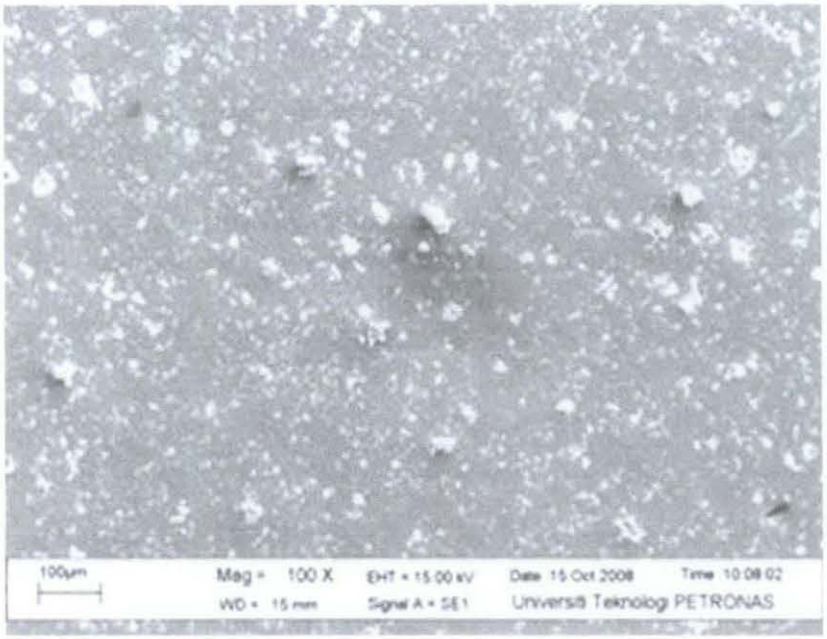
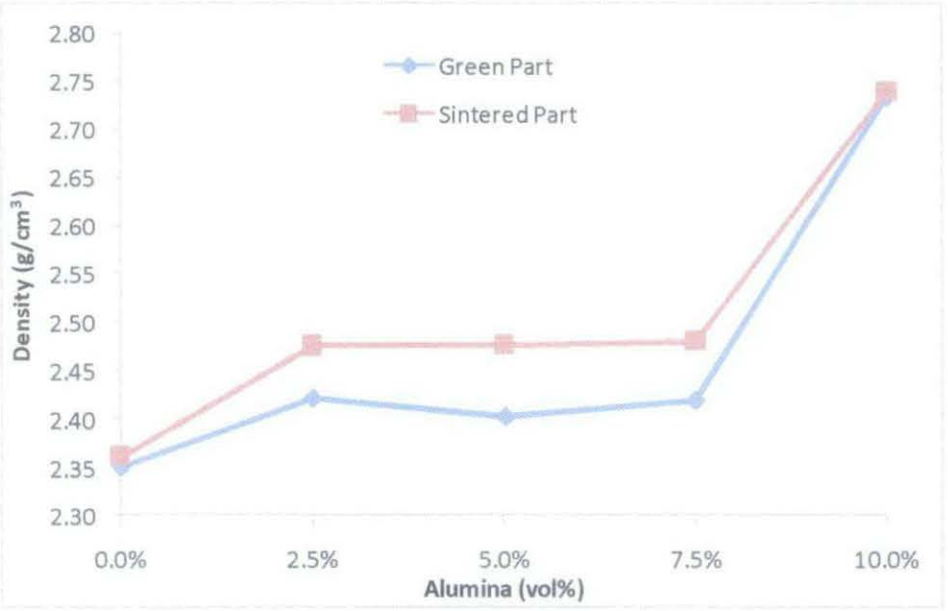


Figure 4-34 (b): SEM Micrograph with 100X magnification of Al-90%vol; Alumina-10%vol sintered samples at 580°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

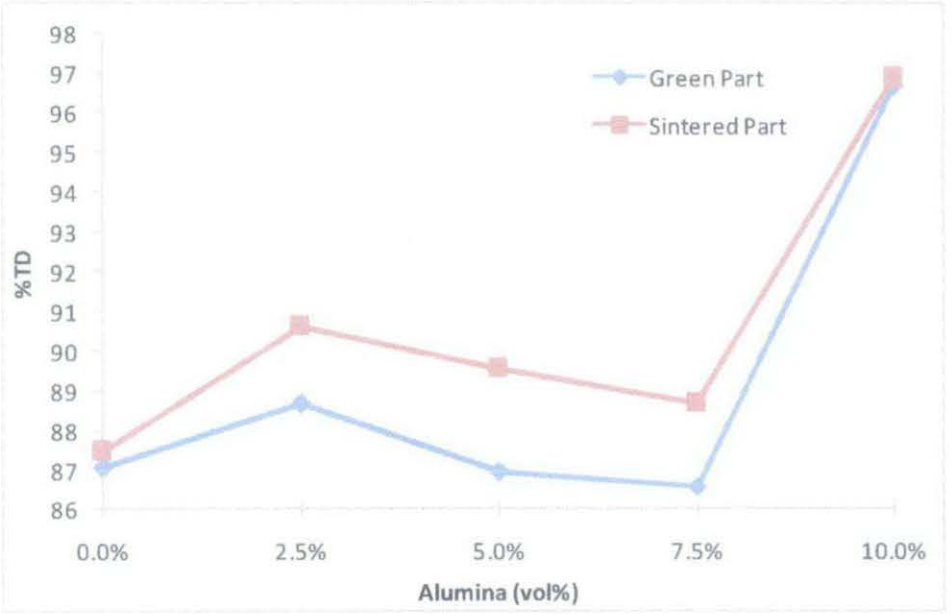
From the hardness curves, the hardness values are increased as the alumina volume fraction increased. Sintering at this temperature gives consistently high value of hardness which around 1000 to 2900 HV. The hardness curves indicated how strong the intermolecular bond between particles. All sintered parts producing the least dimensional change which is below 1% changes except for the pure aluminum powder.

Figure 4-30 to 4-34 shows the micrograph of sintered parts. As density increased large pore is eliminated and also the sintered parts achieved the highest theoretical density. The micrograph also shows the alumina particles are not well distributed. This is due to poor mix of the aluminum powder and alumina powder in the early stage of the experiment.

4.2.3 Sintering at 550°C



(a)



(b)

Figure 4-35: Relationship between green and sintered density for sintered samples at 550 °C for 2hr in Nitrogen atmosphere (a) Density vs. Alumina vol%, (b) Relative Theoretical Density vs. Alumina vol%.

The density and relative theoretical density of all sintered parts are increased after sintering process. Figure 4-35 shows that, at sintering temperature of 550°C, sintered part with 10% alumina achieved the highest density as well as the relative theoretical density. The theoretical density for sintered part with 10% achieved 97% TD. Pure aluminum sintered part produced low density after sintering process. This indicated that this sintered parts are the softest parts in this sintering temperature.

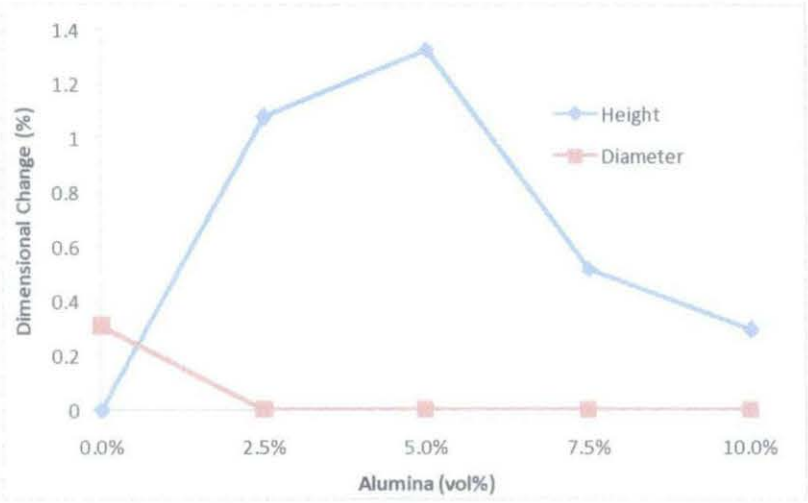


Figure 4-36: Shrinkage curve of the sintered part; sintering at 550°C for 2 hr under nitrogen atmosphere.

Figure 4-36 shows that shrinkage behavior of the sintered parts. As expected sintering under nitrogen atmosphere will produce shrinkage, all sintered parts indicated changes in dimension either the height or diameter. At 550°C, sintered part with 5% alumina, produced the highest shrinkage and the least producing shrinkage is sintered part with 10% alumina. The least shrinkage for sintered part with 10% alumina is due to the large reinforcing amount added.

Figure 4-37 shows the chemical element present in the sintered parts. From initial experiment aluminum, magnesium and oxygen presences. After sintering process EDX analysis shows that no impurities presences in this experiment. The highest concentration of oxygen element indicated that large alumina particle distribution

existed. Trend shows that high concentration of oxygen resulted least dimensional change to the composite.

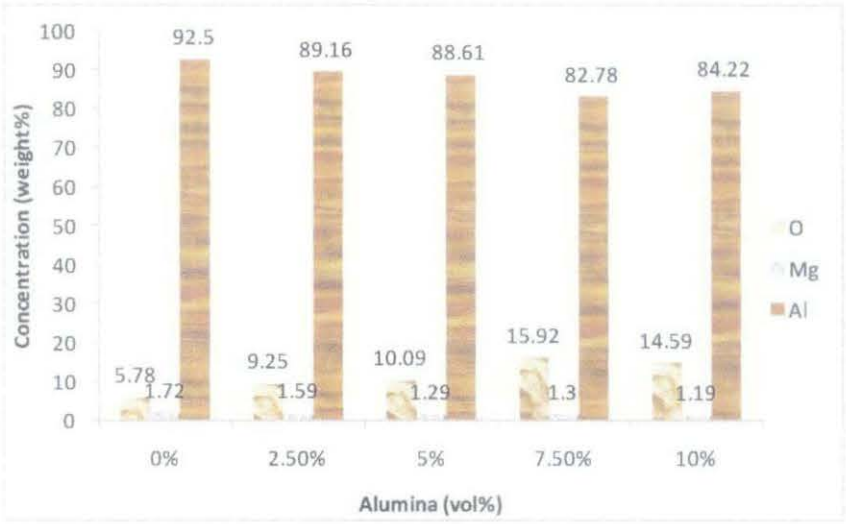


Figure 4-37: EDX analysis of sintered part sintering at 550°C for 2hr under nitrogen atmosphere. Aluminum, magnesium and oxygen content in investigated materials.

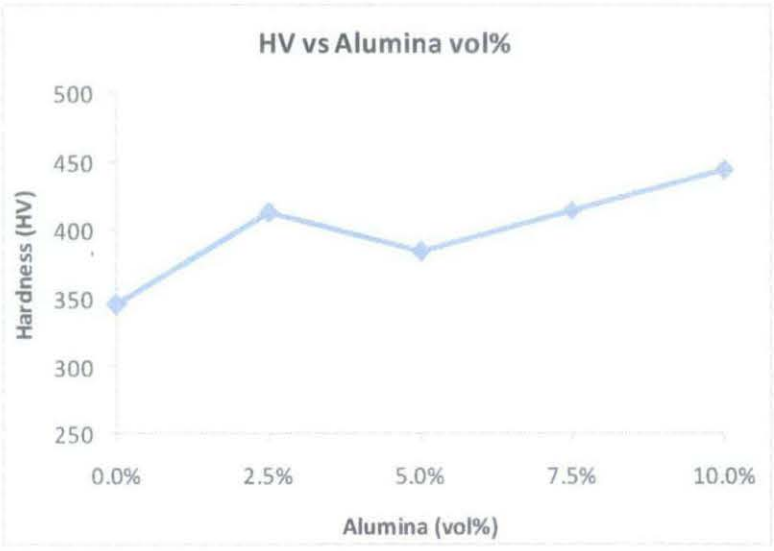


Figure 4-38: Relationship between Hardness value of sintered parts and Alumina %vol.

Figure 4-38 shows the hardness curve of the sintered parts. It shows that low density of sintered part can result in low hardness value. All Sintered part mixed with alumina powder shows high hardness value.

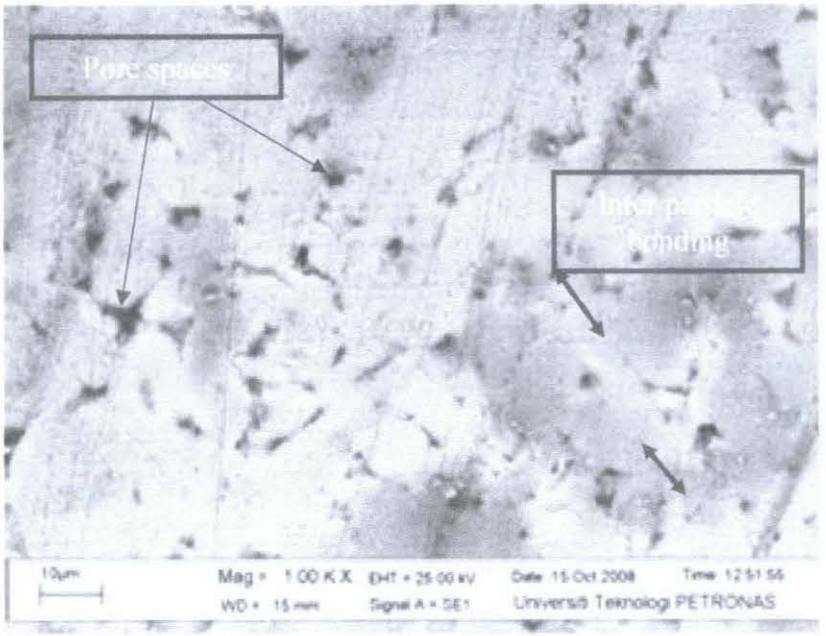


Figure 4-39: SEM Micrograph with 1.0KX magnification of pure aluminum sintered at 550°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

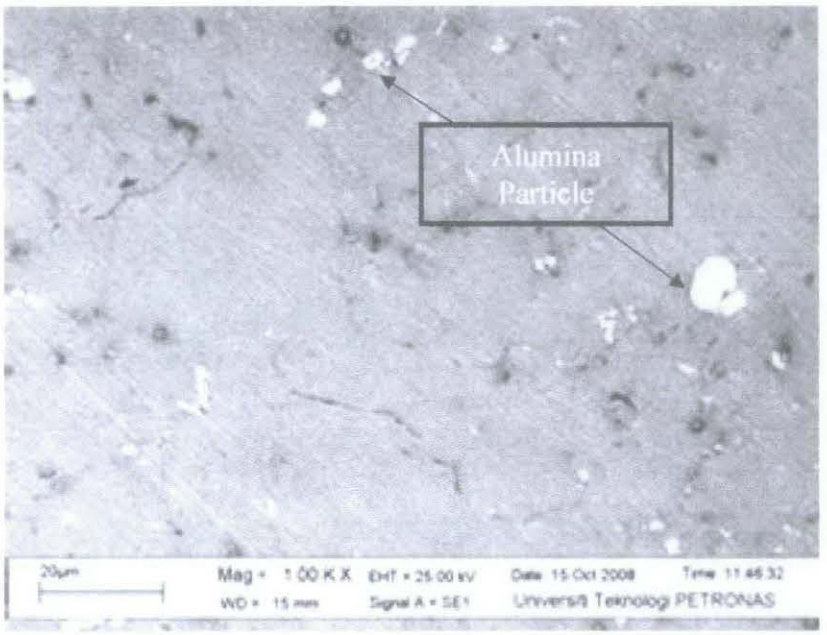


Figure 4-40 (a): SEM Micrograph with 1.0KX magnification of Al-97.5%vol; Alumina-2.5%vol sintered at 550°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

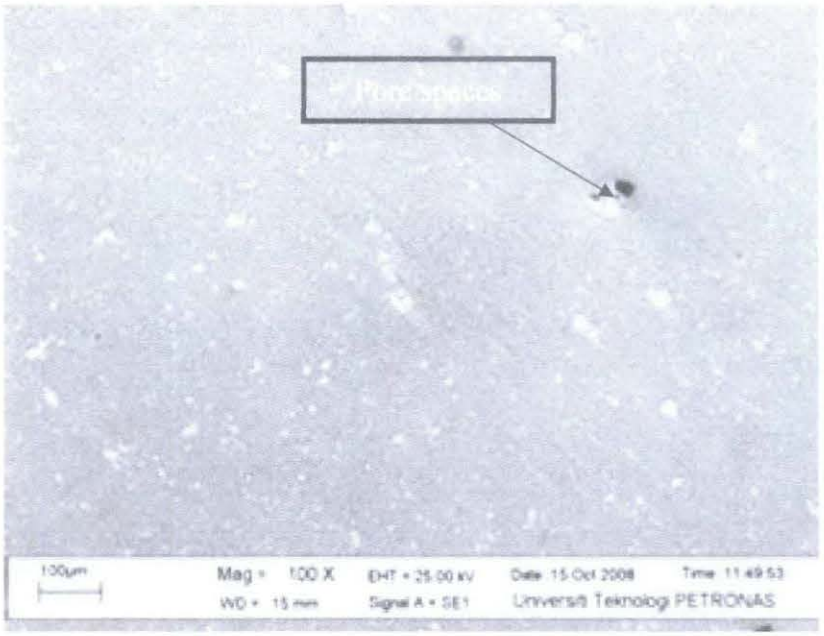


Figure 4-40 (b): SEM Micrograph with 100X magnification of Al-97.5%vol; Alumina-2.5%vol sintered samples at 550°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

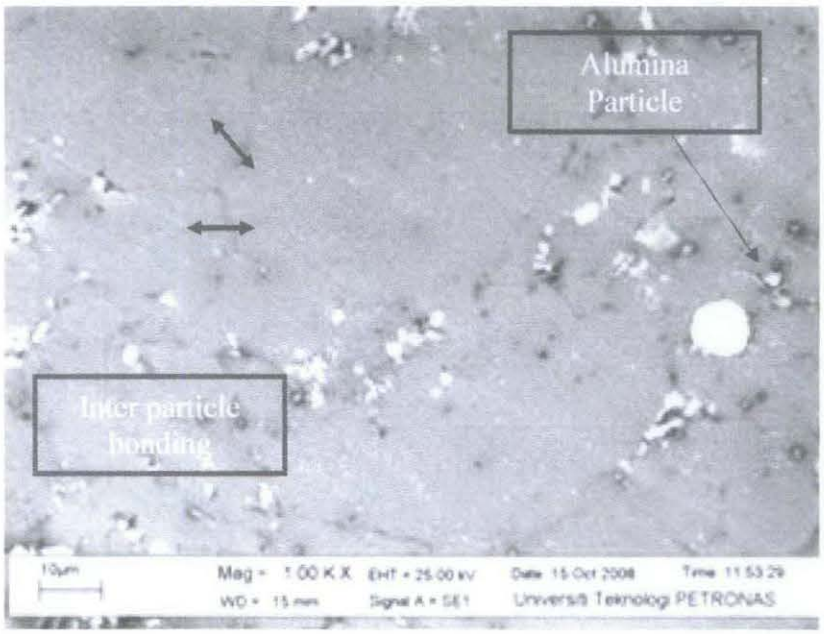


Figure 4-41 (a): SEM Micrograph with 1.0KX magnification of Al-95%vol; Alumina-5%vol sintered samples at 550°C for 2hr under nitrogen atmosphere shows porosity and particle bond

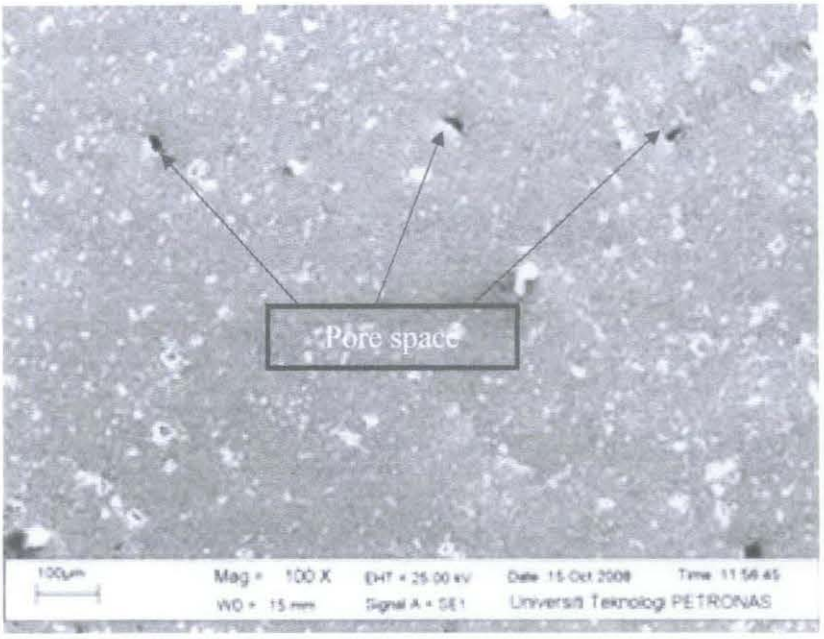


Figure 4-41 (b): SEM Micrograph with 100X magnification of Al-95%vol; Alumina-5%vol sintered samples at 550°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

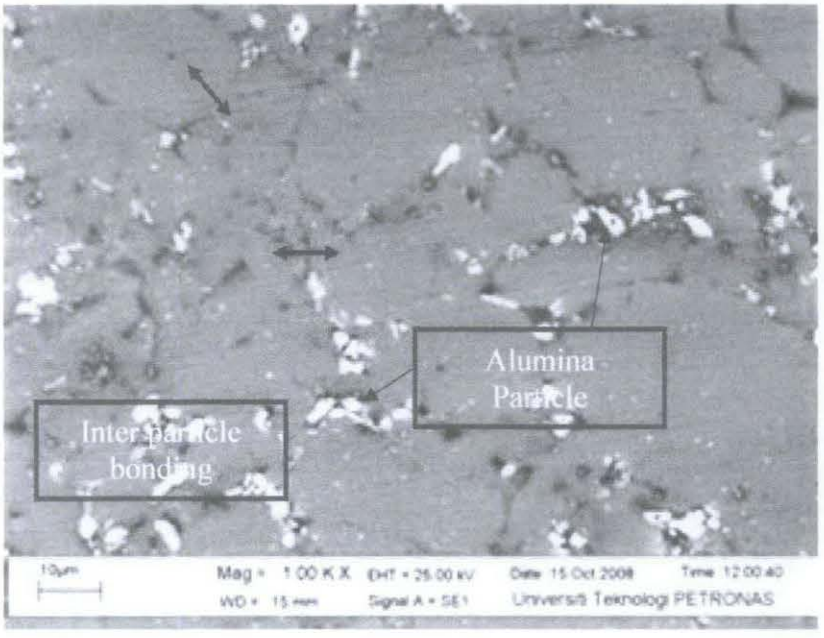


Figure 4-42 (a): SEM Micrograph with 1.0KX magnification of Al-92.5%vol; Alumina-7.5%vol sintered samples at 550°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

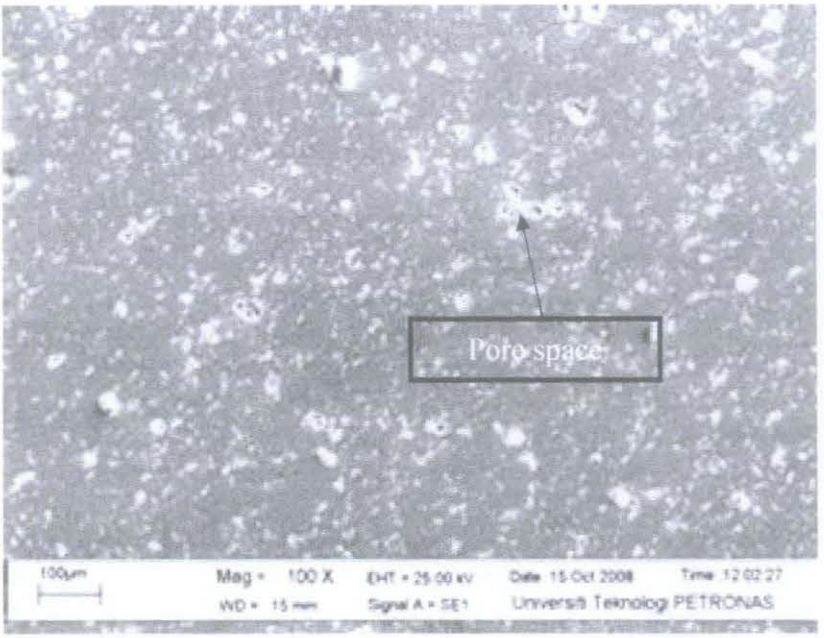


Figure 4-42 (b): SEM Micrograph with 100X magnification of Al-97.5%vol; Alumina-2.5%vol sintered samples at 550°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

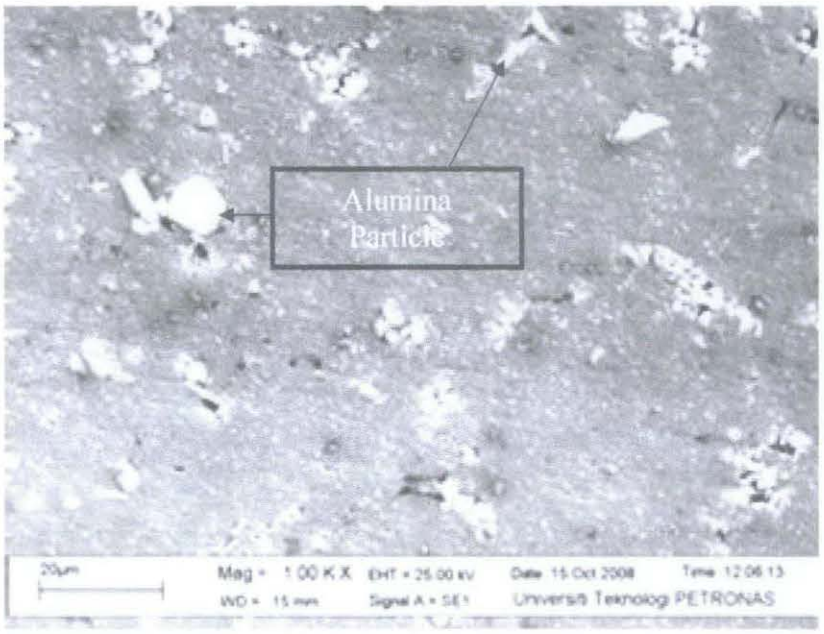


Figure 4-43 (a): SEM Micrograph with 1.0KX magnification of Al-90%vol; Alumina-10%vol sintered samples at 550°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

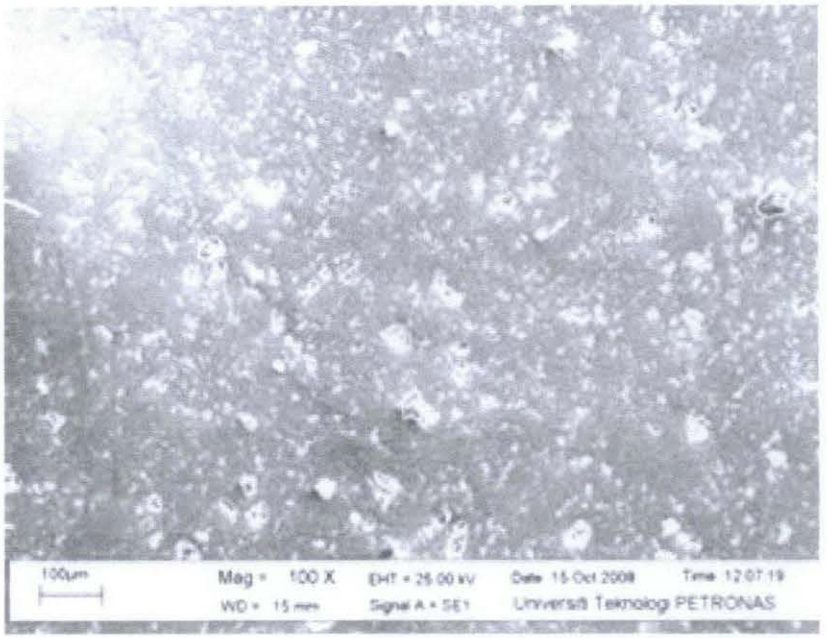


Figure 4-43 (b): SEM Micrograph with 100X magnification of Al-90%vol; Alumina-10%vol sintered samples at 550°C for 2hr under nitrogen atmosphere shows the porosity and particle bond

From the hardness curves, the hardness values are increased as the alumina volume fraction increased. The hardness curves indicated how strong the intermolecular bond between particles. As expected sintered part with 10% alumina producing the least shrinkage among the other sintered parts and the micrograph also shows that pores spaces is eliminated that lead to high packing density.

SEM micrograph for sintered part with pure aluminum and 7.5% alumina show that large pore spaces appeared and microstructure for both sintered parts are coarsening and resulted in low hardness value.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

Alumina particle reinforced aluminum matrix composites were successfully produced using powder metallurgy technique. The main concerned in this study is the sintering behavior of the composites where the composites were underwent four different temperatures under the nitrogen atmosphere in the tube furnace. Sintering behavior of alumina particle reinforced aluminum matrix composites were investigated and explained.

The optimum sintering temperature of this study is 580°C. At this temperature the composites achieved the highest densification with relative theoretical density ranging from 95% and 99% compared to the other temperatures. At this temperature also the composite produced less shrinkage with controllable shrunk less than 1%. Moreover due to high packing density achieved by the composites, resulted consistently produced higher value of hardness with hardness value ranging from 1700HV and 2700HV. The density and hardness of the composite reflected the microstructure behavior of the composite. Sintered at 580°C produced smooth microstructures and pore spaces are eliminated with great inter particles bonding shown. Based on the EDX analysis, there are no impurities presences in the sintered parts as there are only three chemical elements presented during green state and after sintering process which are aluminum, oxygen and magnesium. The high concentration of oxygen element in the composite is due to higher amount of alumina being added.

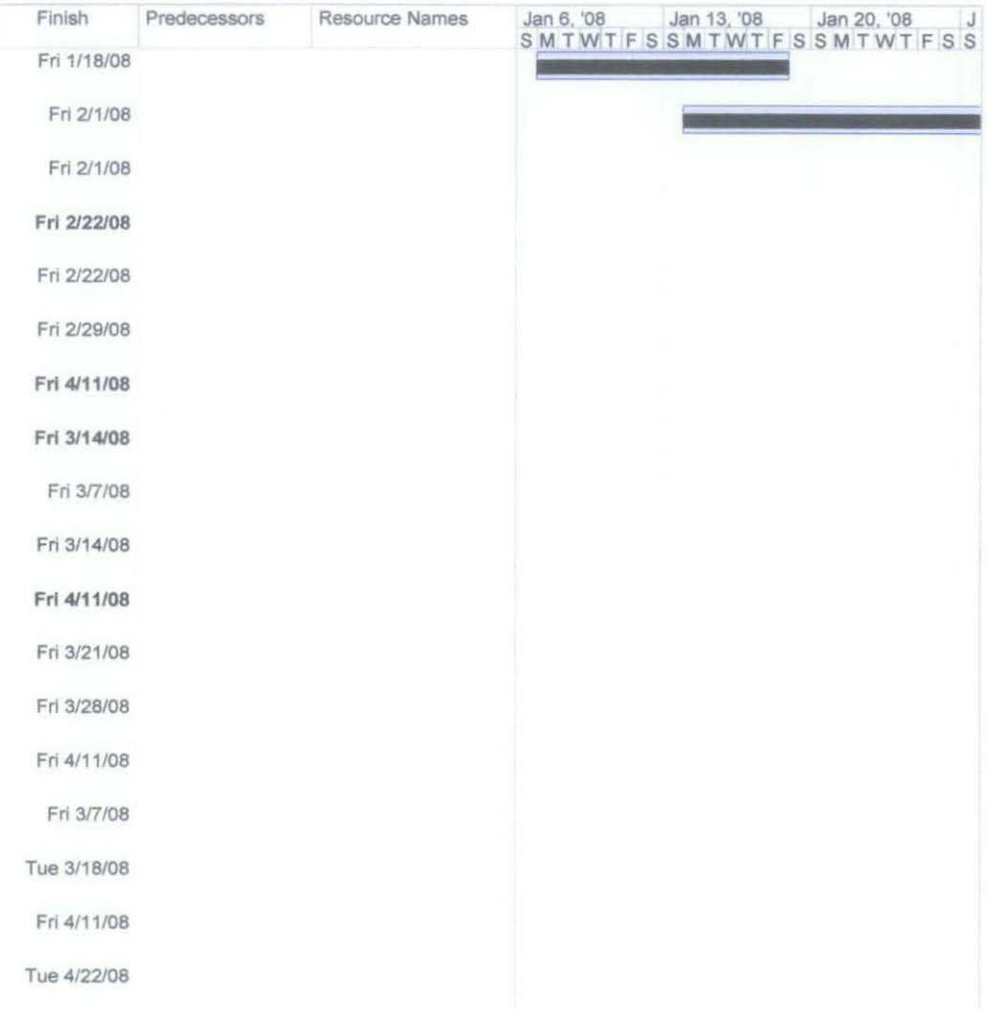
Extra care should be taken in dealing with aluminum powder. It is because the tendency of the aluminum powder to naturally form the oxide layer on the surface of the particles. This film layer act as a hindrance to sintering of aluminum reinforced alumina. High densification of aluminum reinforced alumina powder is hard to achieve when sintering is conducted under the air atmosphere in furnace or with the present of oxygen element in the furnace. Another improvement that should be considered is the mixing and blending process of the powders. As shown in the micrograph of the composites, the tiny alumina particles is clustered at each other. This is due to the behavior of tiny particles to agglomerate to each other.

REFERENCES

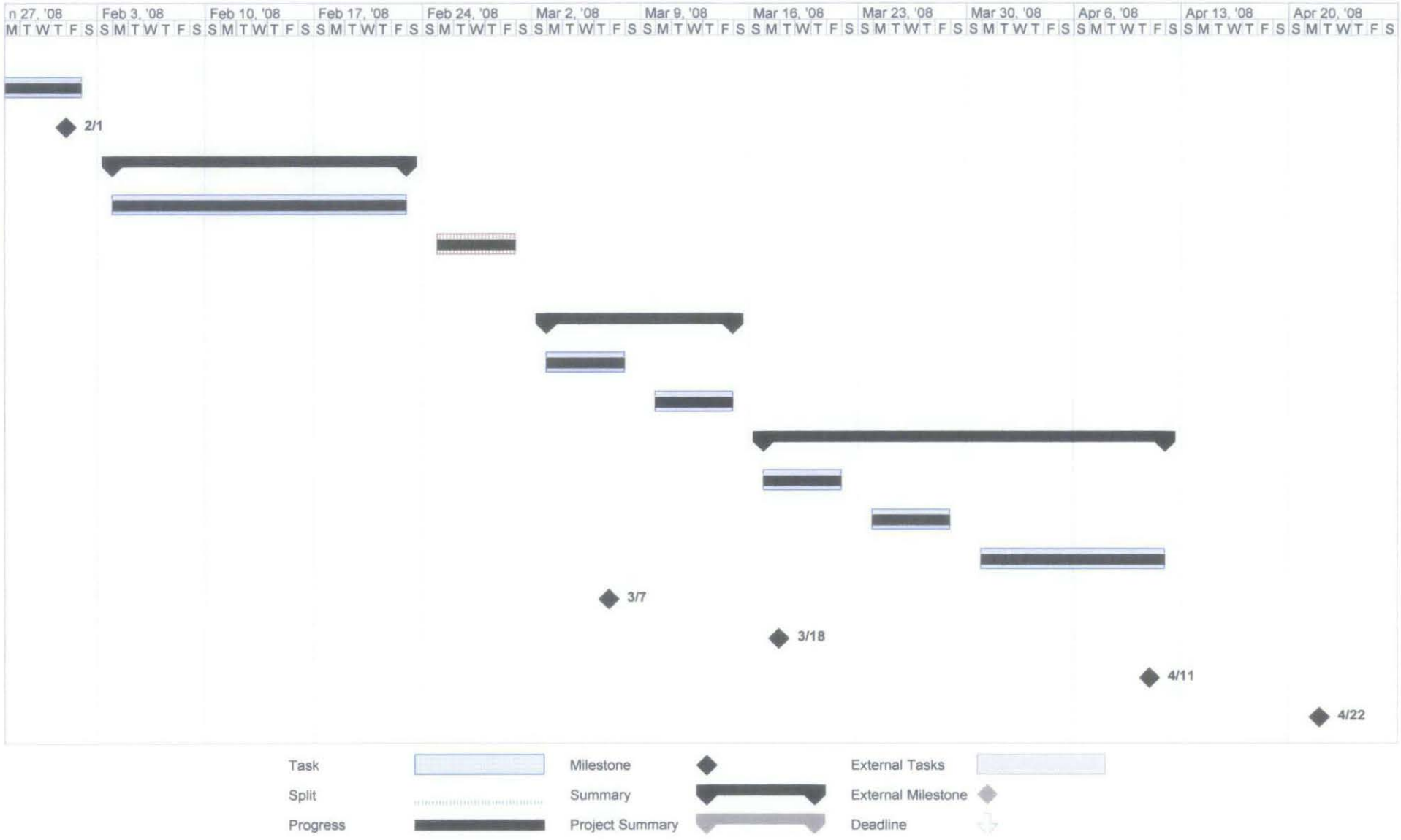
1. J. Eliasson and R. Sandstrom, 1995, Application of Aluminium Matrix Composites. *Metal Matrix Composites Part 1: Application and Processing*: 3-31.
2. Serope Kalpakjian and Steven R. Schmid, 2000, Manufacturing Engineering and Technology, 4th Edition, New Jersey, Prentice Hall International.
3. T.W Clyne, PJ Withers, 1995, *An Introduction to Metal Matrix Composite*, UK, Cambridge.
4. B. Terry, G. Jones, 1990, *Metal Matrix Composites*, Oxford England, Elsevier Science Publishers Ltd.
5. M. D. Huda, M. S. J. Hashmi and M.A El-Baradie, 1995, MMCs: Materials, Manufacturing and Mechanical Properties. *Metal Matrix Composites Part 1: Application and Processing*: 37-61
6. I. Jenkins, and J. V.Wood, 1991, *Powder Metallurgy – An Overview*. Charlton House Terrace, London SW 1Y 5DB: The Institute of Metals
7. Randall M. German, 1989, Particle Packing Characteristics. Metal Powder Industries Federation, New Jersey, Princeton.
8. Abolfazl Babakhani, Ali Haerian b, Mohammad Ghambari, 2006, On the combined effect of lubrication and compaction temperature on properties of iron-based P/M parts. *Materials Science and Engineering A 437 (2006) 360–365*
9. Bhagwan D. Agarwal, Lawrence J. Broutman, 1990, Analysis and performance of fiber composites, 2nd Edition, Canada, John Wiley & Sons, Inc
10. G. O'Donnell, L. Looney, 1998, Production of aluminum matrix composite components using conventional PM technology, *Materials Science and Engineering A303 (2001) 292–301*
11. Sintering process. Retrieved 6 Feb 2008 from the Wikipedia article: http://en.wikipedia.org/wiki/Sintering.
12. J. L. Estrada, V. M. Carreno, H. Balmori and J. Duszcyk, 1996, Relationship between Sintering Atmospheres and Mechanical Properties of P/M Al-20Si-5Fe-2Ni Products. *Sintering Technology*: 277-284

APPENDIX A
FINAL YEAR PROJECT I

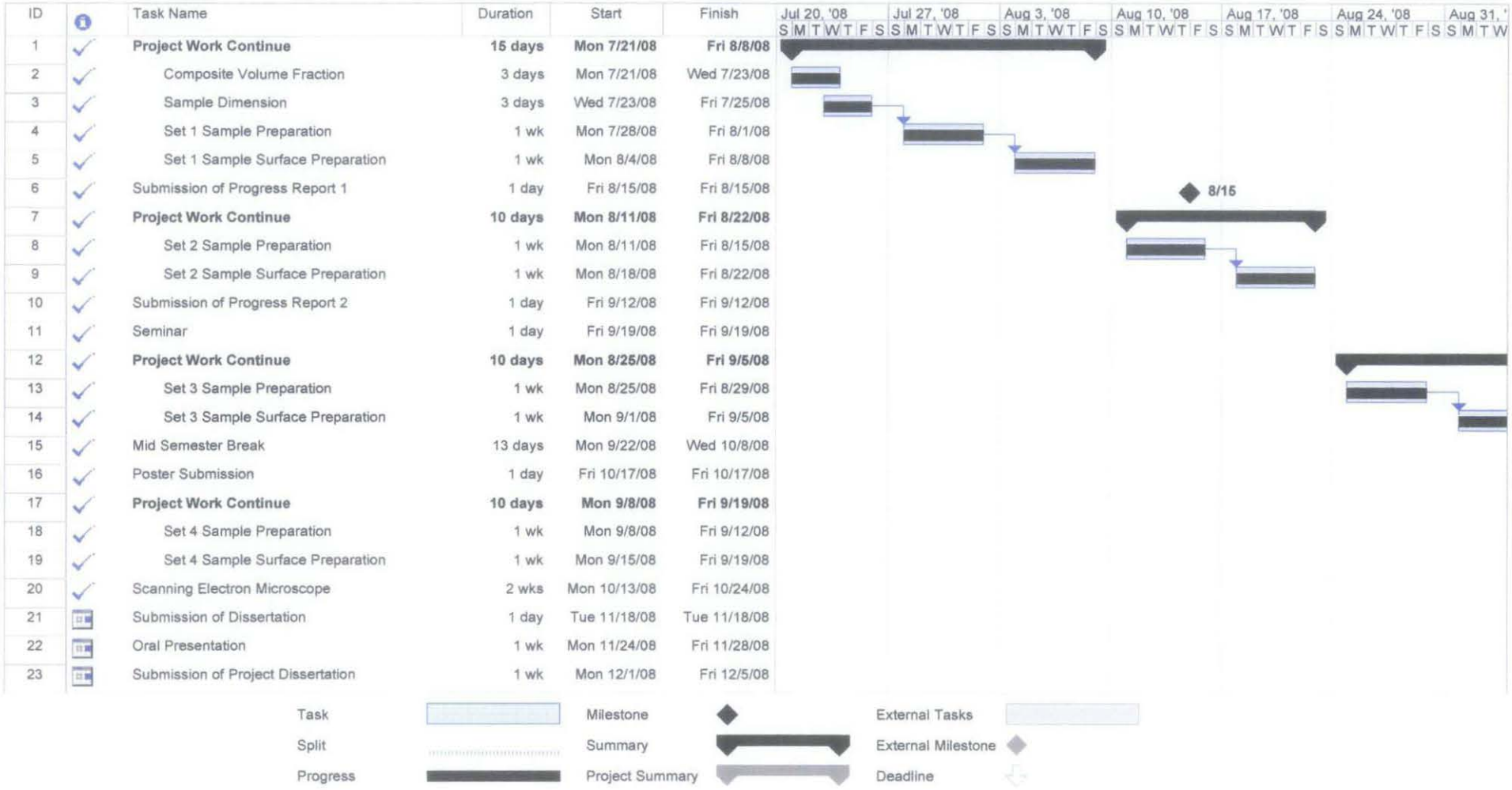
ID	Task Name	Duration	Start	Finish	Predecessors	Resource Names
1	Selection of Project Topic	2 wks	Mon 1/7/08	Fri 1/18/08		
2	Preliminary Research Work	3 wks	Mon 1/14/08	Fri 2/1/08		
3	Submission of Preliminary Report	1 day	Fri 2/1/08	Fri 2/1/08		
4	**Project Work**	**15 days**	**Mon 2/4/08**	**Fri 2/22/08**		
5	Literature Review	3 wks	Mon 2/4/08	Fri 2/22/08		
6	Mid Semester Break	1 wk	Mon 2/25/08	Fri 2/29/08		
7	**Project Work**	**30 days**	**Mon 3/3/08**	**Fri 4/11/08**		
8	**Alumina Powder Characterization**	**10 days**	**Mon 3/3/08**	**Fri 3/14/08**		
9	Particle Size Distribution	1 wk	Mon 3/3/08	Fri 3/7/08		
10	Particle Shape	1 wk	Mon 3/10/08	Fri 3/14/08		
11	**Aluminum Powder Characterization**	**20 days**	**Mon 3/17/08**	**Fri 4/11/08**		
12	Particle Size Distribution	1 wk	Mon 3/17/08	Fri 3/21/08		
13	Particle Shape	1 wk	Mon 3/24/08	Fri 3/28/08		
14	Particle Chemical Surface	2 wks	Mon 3/31/08	Fri 4/11/08		
15	Submission of Progress Report	1 day	Fri 3/7/08	Fri 3/7/08		
16	Seminar 2	1 day	Tue 3/18/08	Tue 3/18/08		
17	Submission of Interim Report Final Draft	1 day	Fri 4/11/08	Fri 4/11/08		
18	Oral Presentation	1 day	Tue 4/22/08	Tue 4/22/08		



APPENDIX A
FINAL YEAR PROJECT I



APPENDIX B
FINAL YEAR PROJECT II



ID	Task Name	Duration	Start	Finish
1	**Project Work Continue**	**15 days**	**Mon 7/21/08**	**Fri 8/8/08**
2	Composite Volume Fraction	3 days	Mon 7/21/08	Wed 7/23/08
3	Sample Dimension	3 days	Wed 7/23/08	Fri 7/25/08
4	Set 1 Sample Preparation	1 wk	Mon 7/28/08	Fri 8/1/08
5	Set 1 Sample Surface Preparation	1 wk	Mon 8/4/08	Fri 8/8/08
6	Submission of Progress Report 1	1 day	Fri 8/15/08	Fri 8/15/08
7	**Project Work Continue**	**10 days**	**Mon 8/11/08**	**Fri 8/22/08**
8	Set 2 Sample Preparation	1 wk	Mon 8/11/08	Fri 8/15/08
9	Set 2 Sample Surface Preparation	1 wk	Mon 8/18/08	Fri 8/22/08
10	Submission of Progress Report 2	1 day	Fri 9/12/08	Fri 9/12/08
11	Seminar	1 day	Fri 9/19/08	Fri 9/19/08
12	**Project Work Continue**	**10 days**	**Mon 8/25/08**	**Fri 9/5/08**
13	Set 3 Sample Preparation	1 wk	Mon 8/25/08	Fri 8/29/08
14	Set 3 Sample Surface Preparation	1 wk	Mon 9/1/08	Fri 9/5/08
15	Mid Semester Break	13 days	Mon 9/22/08	Wed 10/8/08
16	Poster Submission	1 day	Fri 10/17/08	Fri 10/17/08
17	**Project Work Continue**	**10 days**	**Mon 9/8/08**	**Fri 9/19/08**
18	Set 4 Sample Preparation	1 wk	Mon 9/8/08	Fri 9/12/08
19	Set 4 Sample Surface Preparation	1 wk	Mon 9/15/08	Fri 9/19/08
20	Scanning Electron Microscope	2 wks	Mon 10/13/08	Fri 10/24/08
21	Submission of Dissertation	1 day	Tue 11/18/08	Tue 11/18/08
22	Oral Presentation	1 wk	Mon 11/24/08	Fri 11/28/08
23	Submission of Project Dissertation	1 wk	Mon 12/1/08	Fri 12/5/08

APPENDIX B
FINAL YEAR PROJECT II