Characterization and Properties of Copper and Carbon Powders Mixture

by

Azri Izwan Bin Mazlan

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

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
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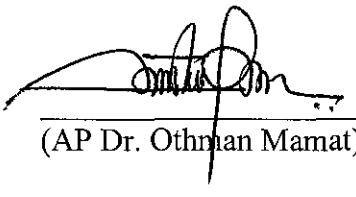
Approved by,



(AP Dr. Othman Mamat)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

December 2008

CERTIFICATION OF ORIGINALITY

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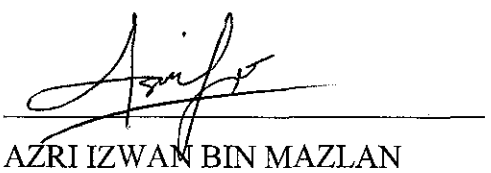

AZRI IZWAN BIN MAZLAN

ABSTRACT

Powder metallurgy (P/M) is one of a metal fabrication technique which involves three main stages, i.e. mixing, compaction and sintering. The P/M technique allows the blending or mixing of additives (carbon) to the base powder (copper) prior to compacting and sintering of parts. The additives will enhance certain property of a metal. The objective of this project is to study the homogeneity of the mixture, the compressibility of the mixture, and the properties of the sintered mixture (copper and carbon). Experiment will be conducted to analyze the characterization and properties of the new metal produce. The copper and graphite powder are used to produce this mixture. The graphite powders have been grind using mortar grinder to become smaller particles. The copper and the graphite powder have been mixed based on weight fraction (98.5wt% Copper and 1.5wt% Carbon). It is compacted using autoPallet press machine and sintered by using furnace. Microscopic analyses using SEM have been done on the powder, mixing mixture, compacted pallet and also sintered pallet. The powder mixture was found homogeneous. The highest densities of green and sintered were 6.66 g/cm^3 and 7.28 g/cm^3 respectively. The highest hardness of the Cu – C mixture was 27.5 HV.

ACKNOWLEDGEMENT

Firstly, I would like to express my highly gratitude to my supervisor, AP. Dr. Othman Mamat for his guidance, inspiration and support through the course of this project. Also for his valuable advice, understanding, patience, and encouragement from the beginning of my involvement in this project under the title "Characterization and Properties of Copper and Carbon Powders Mixture".

To all the technicians; Mr Faizal, Mr Shairul, M Ikhwan and especially Mr. Anuar, I would like to extend a heartfelt thank you for the guidance and effort in helping me with all the technical expertise and facilities. To Mrs. Hafizah who always helps in coordinating and arranging all the meeting and presentation, I would also like to extend a heartfelt thank you.

Special thanks to my family, housemates and fellow friends for keep me calm, help me when pressure comes and also in giving morale support.

Last but not least, I would like to thank all those individuals who have contributed in one way or another in initiating this project.

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1
INTRODUCTION

1.1 Background of Study

Powder metallurgy (P/M), the technology of utilizing metal powders, offers the engineer a means conserving materials, reducing machining and securing a uniform product at a reasonable cost. This unique metal-forming method permits the production of parts with close tolerances and a minimum of scrap. It also enables the development of products that cannot be produced by any other method. By proper selection of powders, the powder metallurgy (P/M) specialist can control the density of products over a wide range and secure a wide range of mechanical and physical properties. He can produce mixtures of metals that are insoluble in each other or mixtures of metals and nonmetals that combine the properties of both. [1]

Density can be controlled to produce parts with porosities as high as 60% or, conversely, those that are practically pore-free and have densities approaching the theoretical density of the metal. It is also possible to vary the density in a single part. And by producing parts with interconnected pores, the metallurgist can obtain a skeleton that can be impregnated with oils, plastics or even a metal having a lower melting point. [1]

In its simplest form, this fabrication technology can be considered to have three basic steps:

1) Mixing
2) Compaction
3) Sintering

1.2 Problem Statement

1.2.1 Problem Identification

Copper are largely used in fabrication industry. Copper as pure component is weak. Copper can be improved by introduce carbon to the mixture. Mixture of these powders is expected to have improved characteristic and properties compared to pure copper. By producing new material, study need to be done to evaluate the characteristics and properties of the new mixture.

1.2.2 Significant of the Project

The experimental project is significant in producing a new materials with better properties compared to the pure copper materials. This will widen the usage of copper in the fabrication industry. The study gives the understanding on P/M production and properties of the copper and carbon mixture.

1.3 Objective and Scope of Study

The objectives of the project are:

- To study the homogeneity of the copper and carbon powders mixture.
- To study the compressibility of the copper and carbon powders mixture.
- To study the properties of the sintered copper and carbon powders mixture.

The scope of study for this project is to analyze the characterization and properties of the new materials formed. Moreover, the method and procedure on producing the mixture using P/M will be determined.

1.4 The Relevancy of the Project

The project is a platform to produce a new material using copper and graphite powders provided in the laboratory. The new material is produced by using P/M technique. Therefore, the characterization and properties of the new material will be analyzed.

1.5 Feasibility of the Project

Mechanical engineering students need to complete the project within 2 semesters. It is presume that the project is feasible within the scope and time frame if there are no issues with regard to equipment function and material availability.

CHAPTER 2
LITERATURE REVIEW

2.1 Copper

Copper is widely used in industry when high electrical and thermal conductivity is needed. A wide variety of copper alloys are commercially available that offer higher strength levels, but usually at the cost of reduced conductivity values. Precipitation hardened alloys can be a good compromise if both high levels of conductivity and strength is required. [2]

However, if the material is subjected to prolonged heating at temperature above the initial precipitation heat treatment either or both properties may suffer. The only way to overcome this is to use dispersion-strengthened copper alloys, where ultra fine ceramic particles such as oxides or carbides in homogeneous distribution generate the high mechanical strength needed. [2]

And that's where P/M comes into its own. The particle distribution required to manufacture dispersion strengthened alloys cannot be achieved by melting technologies of the tendency of the ceramic particles to segregate. They normally form the slag of a melt. The starting powders are mechanically alloyed by ball-milling. During milling, two processes are being carried out at the same time; breaking the powder down through cold working during the milling, and particle growth due to cold welding of the powder particles. In the first stage of milling both processes are roughly in balance leading to an intimate and homogeneous mixture of the different component. [2]

2.2 Graphite

Natural graphite is a modified form of pure carbon. Its basis structure consists of hexagonal groups of carbon atoms, which form stable planar grids with only weak inter-layer bonding. The unique properties of graphite resulting from its distinctive layered structure and chemical inertness make it the material of choice in many applications. It demonstrates very high levels of electrical and thermal conductivity combined with excellent lubricating properties particularly at elevated temperatures and pressures. Its high oxidation resistance and durability against aggressive chemicals mean that graphite does not represent explosion risk. Following along that track, it is environmentally friendly and poses minimal health risks. [3]

In addition, its layered structure enables chemical molecules to be intercalated between the graphite layers. [3]

In powder metallurgy metal powders are compacted by die-pressing and afterwards sintered at temperatures below the melting point of powders. During the sintering process the individual powder particles are "welded" together. Although graphite is added only in very low percentage quantities to P/M premixes, it has two general uses. [3]

First, graphite can be used as an alloying element to increase the strength of the sintered part. During the sintering process the graphite should react with and diffuse into the metallic particles. [3]

Second, graphite can be used for lubrication and/or friction moderation. In this case graphite should only partially dissolve. Some graphite particles then remain for the lubrication and friction moderation of the part. [3]

Varying customer requirement have resulted in the development of different natural graphite grades that can be made application specific by different production method. [3]

2.3 Mixture

A mixture is a material composed of two or more elements or compounds mixed together, but not chemically joined. A mixture is not just one material. In its simplest form, it is still made up of at least two elements or compounds. [4]

The difference between a mixture and a compound is the ease with which the elements can be separated. The elements in a mixture are not chemically joined, while the elements in a compound are chemically joined. Normally, filtering can be used to separate the components of a mixture. [4]

In a mixture, no component completely loses its own identity. Therefore, the characteristics of a mixture are similar to the characteristics of the item that make it up. This is another way in which a compound and a mixture are different. [4]

Iron-rich vitamin tablets contain a mixture of iron and other vitamins. The iron can be removed by grinding up the tablet, then using a magnet to collect the iron particles. [4]

Muddy water is a mixture. In this case, a filter is not even necessary to separate the dirt from water. Just leaving the jar of muddy water stand for a period of time will permit the item in the mixture to separate. [4]

The copper and carbon atoms in steel do not chemically combine with each other. Compounds and molecules are not formed in steel. The atoms of copper and carbon are merely "mixed" together and become an alloy, or solid solution. [4]

2.4 Production Copper and Copper Alloy Powders

Granular copper powder can be produced by a number of methods, the most important being atomization, electrolysis, hydrometallurgy and solid state reduction. Each method yields a powder having certain inherent characteristics. Particle shapes are shown in **Figure 1** [1]

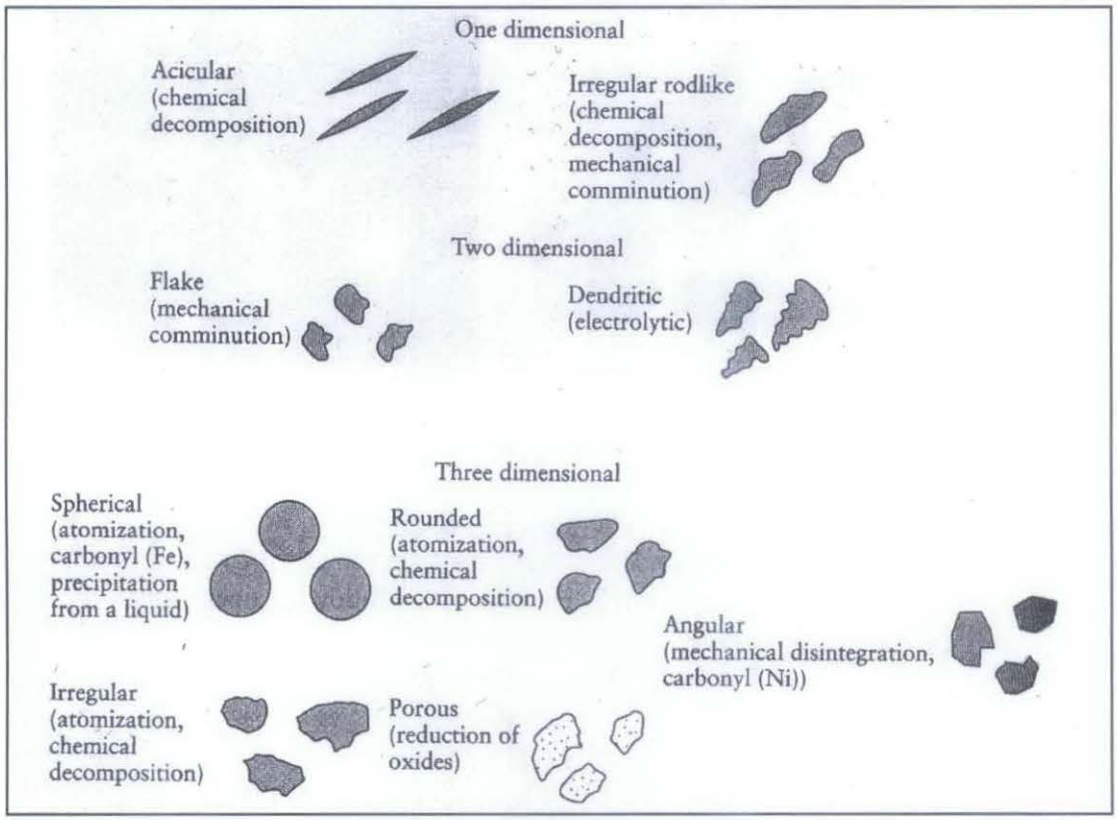


Figure1: Particle Shapes [5]

2.4.1 Atomization

Typically, copper is melted and the liquid metal flows through an orifice where it is struck by a high velocity stream of gas or liquid, usually water, thus breaking the molten metal into particles which solidify rapidly. Particle size and shape are influenced particularly by the atomizing medium, the pressure and the flow rate. Controlled small additions of deoxidizing elements, such as phosphorus, also influence the particle size and shape. After atomization and annealing in a reducing atmosphere to decrease any surface oxide formed during atomization, the product is milled, classified and blended to achieve the particle size distribution required. [1]

The purity of the product depends on that of the raw material since refining of the melt prior to atomization is generally not practiced. Purity is generally over 99%. The powder can be made either spherical or irregular in shape (**Figure 1**). Particle size and shape, apparent density, flow and green strength are influenced not only by atomization variables but also by controlling oxidation during atomization, subsequent reduction during annealing, and by final processing. [1]

2.4.2 Electrolysis

Electrolytic copper powder is produced by following principles used in electroplating with the conditions changed to produce a loose powdery deposit rather than a smooth adherently solid layer. The formation of powder deposits that adhere loosely to the cathode is favored by low copper ion concentration in the electrolyte, high acid concentration and high cathode current density. The addition of colloids, such as glucose, results in the formation of a uniform copper deposit. The starting material is pure cathode copper. Properties of the powder depend on a number of variables including the concentration of sulfuric acid and copper sulfate, type and quantity of the addition agent, temperature of the electrolyte, the current density and the frequency of brush-down. After deposition, the powder is washed to remove all traces of the electrolyte, annealed in a reducing atmosphere, fed to high velocity impact mills to break up clusters, screened, classified and blended to the desired particle size distribution. The properties are influenced also by the temperature used in reducing the powder. [1]

The copper powder obtained by electrolysis is high purity material, averaging more than 99% copper. The powder is dendritic in shape as indicated in **Figure 1**. A wide range of powders having different apparent densities and high green strengths can be obtained by this method. [1]

2.4.3 Hydrometallurgy

The hydrometallurgy process can be used to produce copper powder from cement copper, concentrates or scrap copper. The copper is leached from these materials with sulfuric acid or ammoniacal solutions and the pregnant solution is separated from the residue by filtration. The copper is precipitated from solution by reduction with hydrogen under pressure. In one process, for example, reduction is accomplished in an autoclave at 225-280F (107-138C) in one hour under a partial pressure of hydrogen of 400 psig (total pressure 425 psig) with a thickening agent added to minimize plating and control the particle size. During reduction, 90-95% of the copper is precipitated as powder. The powder is pumped as a slurry to a centrifuge where the powder is separated from the liquid and washed. The wet

copper powder is dried in a reducing atmosphere, milled, classified and blended to achieve the particle size distribution desired. The physical characteristics of the powder can be varied over a considerable range. Temperature and time of reduction and the quantity of acrysol addition have a marked influence on the powder properties. [1]

The process yields a high purity powder, averaging more than 99% copper. Generally, the powder obtained has fine particle sizes with relatively low apparent densities and high green strength. The particle shape is indicated in **Figure 1**. [1]

2.4.4 Solid State Reduction

In this method, oxides including mill scale are first ground to control particle size and then reduced by a gas, usually carbon monoxide, hydrogen or cracked natural gas at temperatures below the melting point of copper. Particle size and shape can be controlled within rather wide limits by varying the particle size and shape of the oxides, the reducing temperature, pressure and flow of the gas. The resulting powder is milled, classified and blended to the desired specifications.
The purity of the product depends on the purity of the oxide since there is no refining during the reduction process. Generally, the powders produced by this method tend to be porous and have high apparent densities and green strength. An irregular particle shape is obtained as is indicated in **Figure 1**. [1]

2.4.5 Production of Alloy Powders

Most alloy powders are produced by atomization. Pre-blended powders are mixtures of the desired composition, with or without lubricant, which will form the alloy during sintering. Pre-alloyed powders are produced by atomization of the alloy composition by the methods mentioned for the production of copper powder. Pre-alloyed powder can also be produced by sintering a blend and grinding to obtain powder with desired characteristics. [1]

Alloy powders are available commercially in various materials. They include brasses ranging from 95Cu-5Zn to 60Cu-40Zn (and leaded versions of these alloys), nickel silvers, tin bronzes, aluminum bronzes and beryllium bronzes. As mentioned previously, any copper alloy can be produced in powder form. [1]

2.4.6 Production of Flake Powders

The powders discussed previously have been granular in form and are used primarily for the production of P/M parts. Flake powders are used for other purposes. Although pure copper powder is produced in flake form, most flake powder, the so-called "gold bronze" powders, is produced from alloys of copper with zinc and aluminum. Special colors are produced by modifying the base alloys with tin or nickel. [1]

The alloy is powdered by atomization or is melted to produce spatter and the particles are charged into ball mills with a lubricant such as stearic acid and reduced to the desired fineness. Alternately, the Hall paste process involving ball milling in mineral spirits or the Hametag modification of ball milling can be employed. After milling, additional lubricant is added and the powder is polished in drums and stored to develop suitable leafing properties. [1]

2.4.7 Production of Copper Compounds

Cuprous oxide (Cu_2O), cupric oxide (CuO) and cuprous sulfide (Cu_2S) are produced as powders by the controlled reaction of oxygen with copper powder. The products are used in antifouling paints (Cu_2O), reagents in chemical reactions, catalysts in the production of silicone compounds and in foundries for hydrogen degassing of non-ferrous melts. [1]

2.5 Mixing and Blending Metal Powders

Blending is defined as the intermixing of powders of the same nominal composition. It is used to achieve a desired particle size distribution. Mixing implies intermingling powders of different chemical composition. Both can be mathematically described as an instantionary change of concentration of the components along a local coordinate.

The concentration change occurs by a convective or dispersive transport of the particle. The latter mode can be subdivided into diffusional transport and a stochastic particle motion caused by an energy input via stirring or other measures. The optimum dispersion which can be expected from a mixing process is a random distribution. [6]

Very varied equipment is available for blending, mixing and dispersing of powders. Tumble mixers utilize gravitational forces for producing particle motion. Shear-agitated mixers use paddles or other moving component in stationary container for shearing plane planes within the bulk powder. Centrifugal mixers have a similar design but operate in range of rotational speeds, in which particle motion is controlled mainly by centrifugal forces. In fluidized bed blenders, the powder takes character of liquid and the contribution of convection and diffusion to particle motion and mixing increased compared with the former methods. For dry mixing and blending the of metal powders, tumble and low-shear mixers are mainly used in practice, due to possible particle degradation in high shear equipment. [6]

Mixing is important and need to be carried out because:

- Uniformity can be achieved by mixing the powders that have different sizes and shapes.
- Powders of different metals and other materials may be mixed in order to impart special physical and mechanical properties and characteristics to the P/M product.
- Lubricants may be mixed with the powders to improve the powders' flow characteristics. Such blends result in reduced friction between the metal particles, improved flow of the powder metals into the dies, and longer die life. [5]

2.6 Compaction and Shaping

Compaction is the second step in P/M after the mixing process. In compaction, blended powders are pressed into shapes in dies using presses that are either hydraulically or mechanically actuated. The purposes are to obtain the required shape, density, and particle-to-particle contact and to make the part strong enough to be processed further. The as-pressed powder is known as a green compact.

Bulk powders are transformed into performs of a desired shape and density by compaction or shaping. Some process variants combine the shaping operation with the sintering step and end up with the sintered part instead of a preform. [6]

2.6.1 Pressure-assisted Shaping

The pressure-assisted forming operation can be subdivided into cold and hot compaction methods. From the material's point of view, cold compaction takes place in a temperature range within which high temperature deformation mechanisms like dislocation or diffusional creep can be neglected. In most particle cases, cold compaction occurs at ambient temperature, while hot compaction is carried out at enhanced temperatures. Low melting materials like lead, however, undergo hot compaction even at room temperature. [6]

2.6.2 Cold Compaction

Cold pressing is the most important compaction method in P/M. It starts from bulk powders containing very small amounts and sometimes even no lubricant or binder addition. One usually distinguished between axial (die) and isostatic pressing. In axial pressing, the powder is compacted in rigid dies by axially loaded punches. The axial compaction pressure, which is defined as the punch load divided by the punch face area is the main process variable. In isostatic pressing, the powder is sealed in an elastic mould and exerted to the hydrostatic pressure of a liquid pressure medium. [6]

Axial pressing is by far the most practical forming method. The powder is compacted between the punch faces and the die walls, which undergo only very limited elastic deformation. The compacts can, therefore, be fabricated to very close geometrical tolerances. The compaction sequence consisting of die filling, compaction, and ejection of the compact can be carried out in both mechanical and hydraulic presses or as mixed mode presses at high production rates. Axial powder pressing is therefore a very economic method for mass production of precision parts. [6]

2.7 Sintering

Sintering is the process where compressed metal powder is heated in a controlled atmosphere furnace to a temperature below its melting point, but sufficiently high to allow bonding (fusion) of the individual particles.

During sintering the particles contacts increase in quality due to the formation of bondings between the atoms or iron comparable with the bonding strength of a regular lattice. In pure, single component, sintering takes place completely in the solid state. In multicomponent systems a liquid phase may be involved, but only to the extent that the solid skeleton guarantees the geometrical stability of the part (**Appendix B**). Sintering may be accompanied by shrinkage, leading to densification, especially in fine powders; coarser powders may sinter with almost perfect dimensional stability. Sintering and shrinkage are by no means identical. Like other thermally activated processes, sintering depends strongly on temperature. [6]

Sintering temperatures are generally within 70% to 90% of the melting point of the metal or alloy. [5]

Proper control of the furnace atmosphere is essential for successful sintering and to obtain optimum properties. A vacuum is generally used for sintering refractory metal alloys and stainless steels. The gases most commonly used for sintering a variety of other metals are hydrogen, dissociated or burned ammonia, partially combusted hydrocarbon gases, and nitrogen. [5]

Depending on temperature, time, and processing history, different structures and porosities can be obtained in a sintered compact. However, porosity cannot be completely eliminated, because voids remain after compaction, and gases evolve during sintering. Porosities can consist of either a network of interconnected pores or closed holes. [5]

Typical sintering temperature and time of copper alloys and steels are shown (**Figure 2**).

Material	Temperature		Time, min
	°C	°F	
Bronze	760-870	1400-1600	10-20
Copper	840-900	1550-1650	12-45
Brass	840-900	1550-1650	10-45
Iron, Iron-graphite, etc.	1010-1150	1850-2100	30-45
Nickel	1010-1150	1850-2100	30-45
Stainless steel	1095-1285	2000-2350	30-60

Table1: Typical sintering temperature and time copper alloys and steels [7]

CHAPTER 3
METHODOLOGY

3.1 Procedure Identification

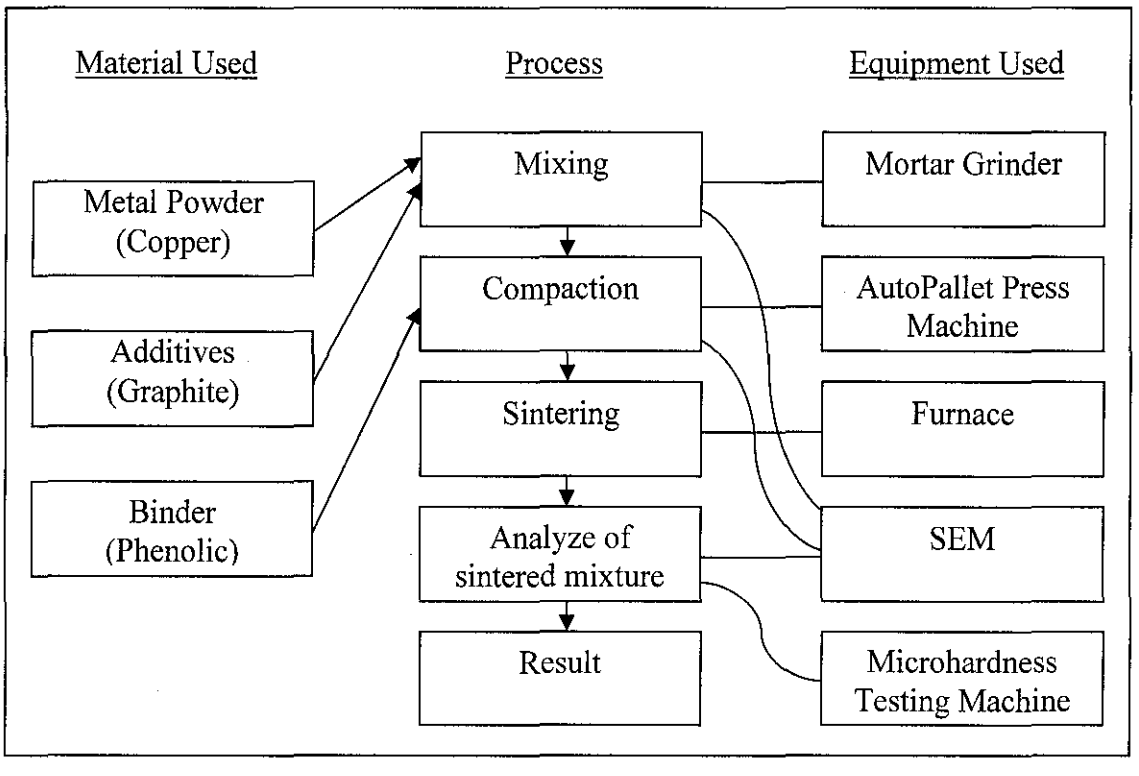


Figure 2: Flow diagram of the procedure used for this project

In producing the powder metallurgy, firstly, both powders which are copper powder and carbon powder (graphite) are mixed together. Prior to the mixing, graphite powder are grind into smaller particle. Second, compaction process; where the blended powders are pressed into shapes in dies using presses that are either hydraulically or mechanically actuated. The purposes of compaction are to obtain the required shape, density, and particle-to-particle contact and to make the part strong enough to be processed further. Binder is added to improve the powders contact. Third, sintering process is applied to the compacted powder. Sintering is the process whereby compressed metal powder is heated in a controlled atmosphere furnace to a temperature below its melting point, but sufficiently high to allow bonding (fusion)

of the individual particles [5]. Lastly, the characteristics of the powder will be analyzed and the properties of the mixture will be tested.

3.2 Equipment used

In this project, equipments used by the student are mortar grinder, autoPallet press machine, furnace, scanning electron microscope (SEM), microhardness test machine and weight balance machine.

Mortar grinder used to grind the powders into smaller particles. Powders need to be about the same size so it can mix well to form the mixture.

Figure 3: Mortar Grinder

Autopallet press machine is used to compact the mixture. The machine can compact the mixture up to 25 tonne but the mixture can only be compressed up to 18000lb because of the limit of the die used.

Figure 4: AutoPallet Press Machine

Furnace is used during sintering process where the pallet was heat into certain temperature to allow fusion between the particles. There are different types of furnace that can be used. Ideally the pallet needs to be sintered in inert environment to avoid any reaction with unwanted substance that exists in the air. Due to the technical problem and unavailability of the required furnace, the student used the normal furnace for the sintering process.

Figure 5: Furnace

Scanning electron microscope (SEM) is a microscope that produces an image by using an electron beam that scans the surface of a specimen where an image is produce by reflected electron beams.

Figure 6: Scanning Electron Microscope (SEM)

Microhardness test machine is used to test the hardness of mixture formed.

Figure 7: Microhardness test machine

Weight balance machine used to accurately weigh small quantity of material and can accurately weigh sample up to 4 decimal places of gram.

Figure 8: Weight balance machine

3.3 Composition of Mixture

Each metal have its own solubility in other metal. Solubility of carbon in copper is between 1 and 2 weight percent. [8]

In this project, the student is focusing on one composition of the mixture. Student takes the middle composition which is 1.5 weight percent of carbon. Therefore the total composition of the mixture is:

Table 2: Mixture Composition of Sample

Element	**Composition (wt %)**
Copper	98.5
Carbon	1.5
Total	100.0

3.4 Gantt Chart

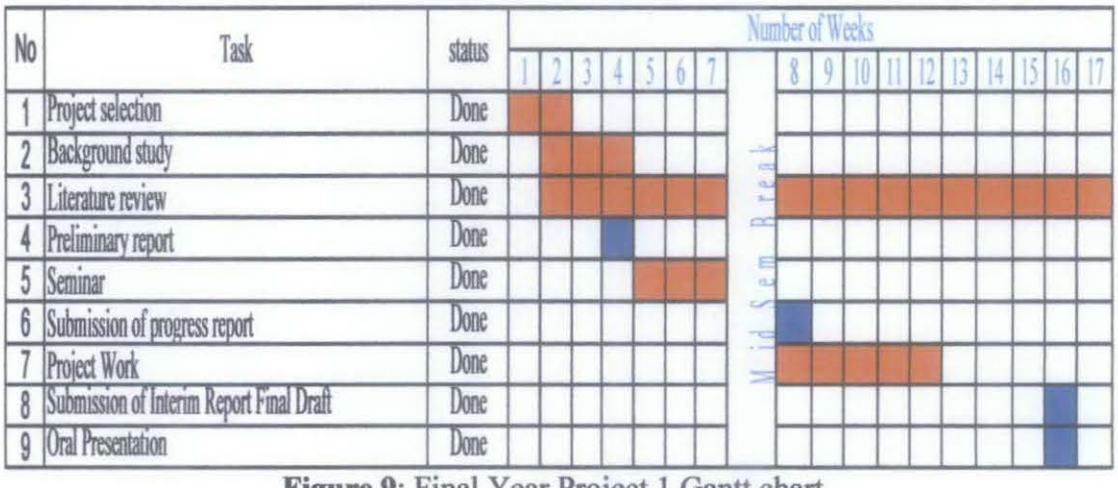


Figure 9: Final Year Project 1 Gantt chart

No	Task	status	Number of Weeks																
			1	2	3	4	5	6	7	8	9	10		11	12	13	14	15	16	17
1	Project Work Continue	Done																		
2	Submission of Progress Report 1	Done																		
3	Project Work Continue	Done																		
4	Submission of Progress Report 2	Done																		
5	Seminar (compulsory)	Done																		
6	Project work continue	Done																		
7	Poster Exhibition	Done																		
8	Submission of Dissertation (soft bound)	Done																		
9	Oral Presentation	Done																		
10	Submission of Project Dissertation (Hard Bound)	Done																		

Mid Semester Break

Figure 10: Final Year Project 2 Gantt chart

CHAPTER 4
RESULTS AND DISCUSSION

4.1 Material Selection

The student has developed the understanding on the process in powder metallurgy. Besides, the student has determined the powder that will be use to produce the mixture. The student was using copper and graphite powders that available at lab for this project. The general information about the powder can be referred to the MSDS came from the manufacturer of the powder (**APPENDIX D1 and D2**).

Figure 11: Copper powder

Figure 12: Graphite powder

The student has used Scanning Electron Microscope (SEM) to view the powders in more details.

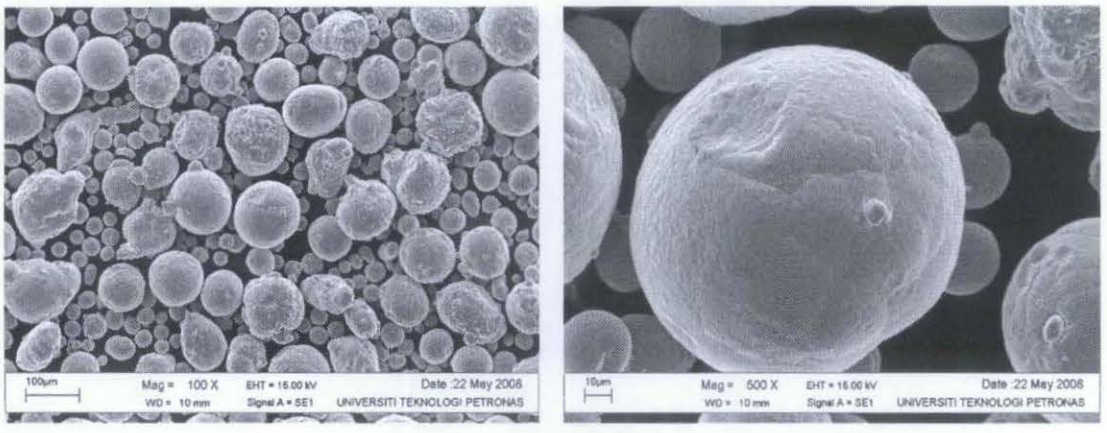


Figure 13: Copper powder zooms at 100 times (left) and 500 times (right) using SEM.

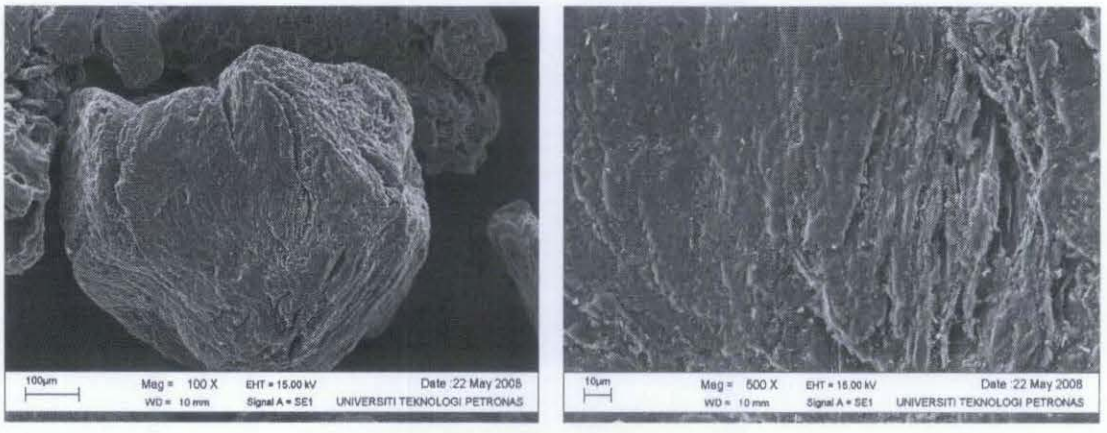


Figure 14: Graphite powder zooms at 100 times (left) and 500 times (right) using SEM.

From the SEM images, the copper powder shape is spherical. Therefore, the student concluded that the copper powders could be produce using gas atomization technique. The graphite powders are in irregular shape; therefore it could be produces by atomization or chemical decomposition.

The surface properties of the powders also can be clearly saw in the image. Copper have a smooth surface compared to graphite that have a rough surface.

4.2 Powder Preparation

The student has determined that the powders are in different sizes. Copper powders are -100 mesh, which means the copper particles pass through the 100 mesh sieve and smaller than the sieve. Graphite powders are -20+84 mesh, which means the graphite particle pass through 20 mesh sieves and retained on 84 mesh sieves.

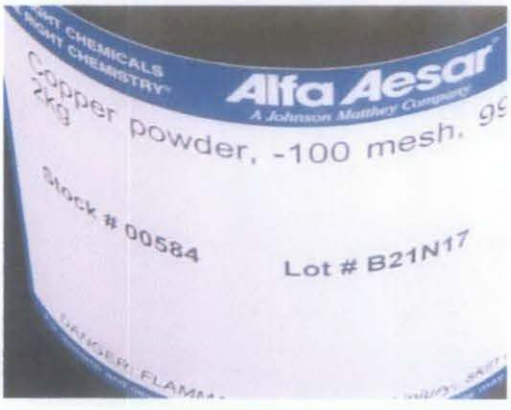


Figure 15: Label of the bottle of copper powder that been used.

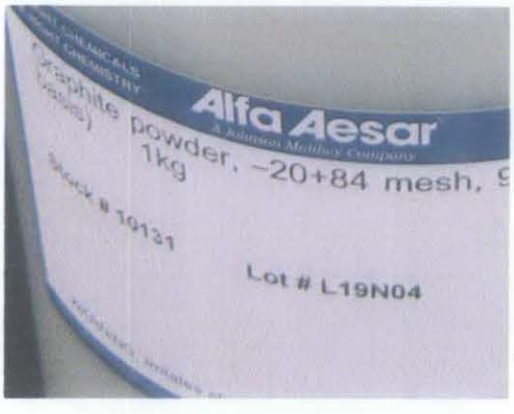


Figure 16: Label of the bottle of graphite powder that been used.

Converted this size using mesh conversion table, the student determined the size of both powders. Copper powder size is less than 149μm and the graphite powder size is between 177μm and 840μm.

Table 3: Mesh conversion table. [10]

MESH SIZE	APPROXIMATE MICRON SIZE
4	4760
6	3360
8	2380
12	1680
16	1190
20	840
30	590
40	420
50	297
60	250
70	210
80	177
100	149
140	105

Size of graphite powder:
177μm ≤ Graphite Powder < 840μm

Size of Copper powder:
Copper Powder < 149μm

Due to the difference in size, the powders need to be grind to the level that the size of both powder are about the same. Mortar grinder can be used for this purpose.

At first student put both powders in the mortar grinder so it grinds both powders and subsequently mixed the mixture. Then, the student has used Scanning Electron Microscope (SEM) to view the mixture in more details.

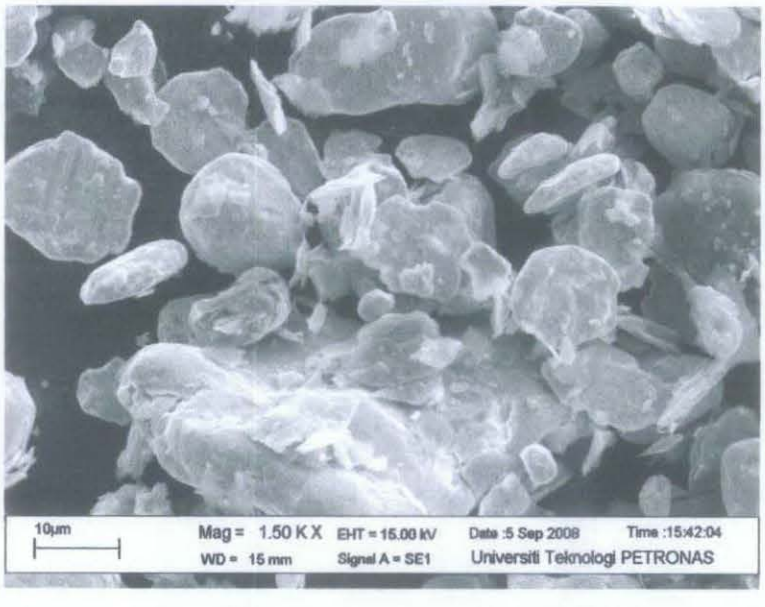


Figure 17: Copper and graphite powder after mix and grind zoom at 1500 times.

However, the mortar grinder is a very hard grinder. From the SEM image of the mixture, it shows that mortar grinder has crushes the mixture particle and destroy it shape. This will affect the homogeneity of the mixture and made the mixture harder to fuse with each other.

The student then grinds only the graphite powder using the mortar grinder. The copper powder as the base powder remains in the same size to protect it particle shape. SEM image of the mixture shows that the graphite powder has been reduced to the size that about the same as the copper powder.

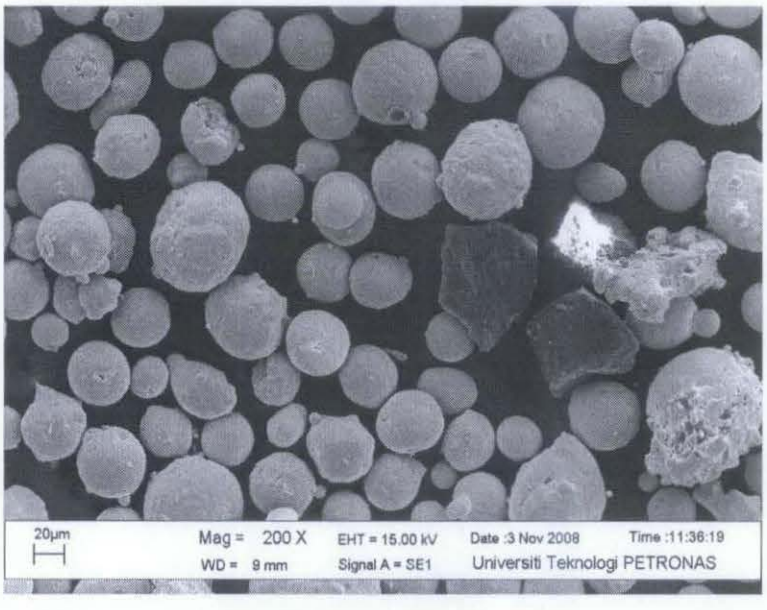


Figure 18: Copper and graphite powder after mix zoom at 200 times.

4.3 Mixing

The student use conventional way in mixing the powders. Prior to the mixing, each powder weighs accurately using the weight balance machine to get the weight fraction needed.

Table 4: Weight of copper and graphite powder in the sample mixtures

Sample	Weight ratio	Copper powder (g)	Graphite powder (g)	Total weight (g)
1	9.85:1.5	29.5508	0.4523	30.0031

After accurately weigh both powders, the mixture then put in closed bottle and shake manually for 2 minutes to uniform the mixture.

Figure 19: Bottle been shakes manually for 2 minutes to mix the mixture.

4.4 Compaction

After that, the mixture was compacted using autopallet press machine. Prior to the compaction process, the mixture was weighed and divided to 5g sample so that each pallet compacted are weigh about 5g. 5 samples were made to be compacted.

Each sample compacted using the same die but with different pressure. The dwell time of the compaction process were set to 2 minutes.

During the compaction process, the pallet of the mixture broke and become the loose powder again. This happens because of the characteristic of the graphite that acts as the lubricant and looses the compacted pallet.

Binder been used to help bind the mixture temporarily before it been sent for sintering. Phenolic thermosetting powder is selected because of the availability in the lab. Experimentally, the less amount of binder that can be used is found out to be 2wt% of the mixture.

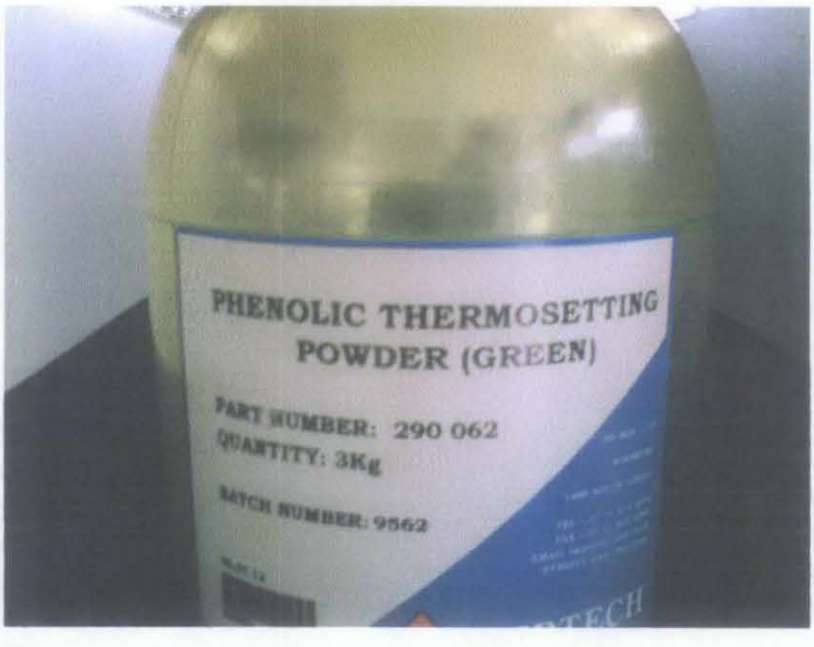
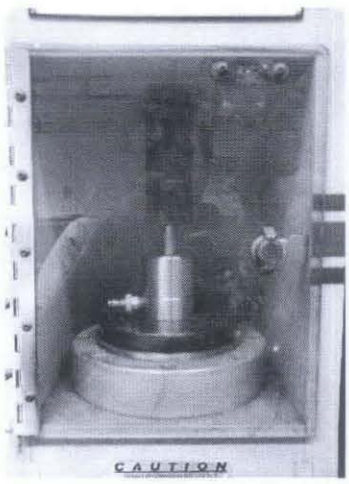


Figure 20: Label of the phenolic thermosetting powder that used as the binder.



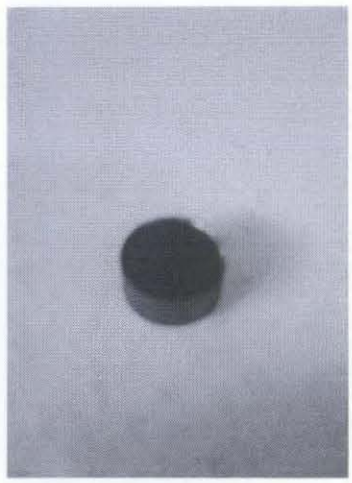


Figure 21: AutoPallet press machine during compaction process (left) and compacted pallet (right).

The process repeated three times to produces more accurate and reliable data. Below are the tables of green density of different compaction pressure. Sample calculation of green density is attached in **Appendix E.**

Table 5: Green density of the compacted pallet for 44482N (10000lb)

Sample	Sample weight, m (g)	Sample volume, v (cm^3)	Green density, ρ (g/cm^3)
1	4.9838	0.8109	6.146
2	4.9586	0.8122	6.105
3	4.9785	0.8090	6.154
Average Green Density (g/cm^3)			6.135

Table 6: Green density of the compacted pallet for 53379N (12000lb)

Sample	Sample weight, m (g)	Sample volume, v (cm^3)	Green density, ρ (g/cm^3)
1	4.9751	0.7770	6.403
2	4.9648	0.7731	6.422
3	4.9565	0.7731	6.411
Average Green Density (g/cm^3)			6.412

Table 7: Green density of the compacted pallet for 62275N (14000lb)

Sample	Sample weight, m (g)	Sample volume, v (cm^3)	Green density, ρ (g/cm^3)
1	4.9785	0.7590	6.559
2	4.9854	0.7616	6.546
3	4.9940	0.7540	6.623
Average Green Density (g/cm^3)			6.576

Table 8: Green density of the compacted pallet for 71172N (16000lb)

Sample	Sample weight, m (g)	Sample volume, v (cm^3)	Green density, ρ (g/cm^3)
1	4.9883	0.7509	6.643
2	4.9856	0.7501	6.647
3	4.9910	0.7514	6.642
Average Green Density (g/cm^3)			6.644

Table 9: Green density of the compacted pallet for 80068N (18000lb)

Sample	Sample weight, m (g)	Sample volume, v (cm^3)	Green density, ρ (g/cm^3)
1	4.9764	0.7470	6.662
2	4.9852	0.7482	6.663
3	5.0311	0.7556	6.658
Average Green Density (g/cm^3)			6.661

The compressibility of the mixture determined by plot the graph of density versus pressure.

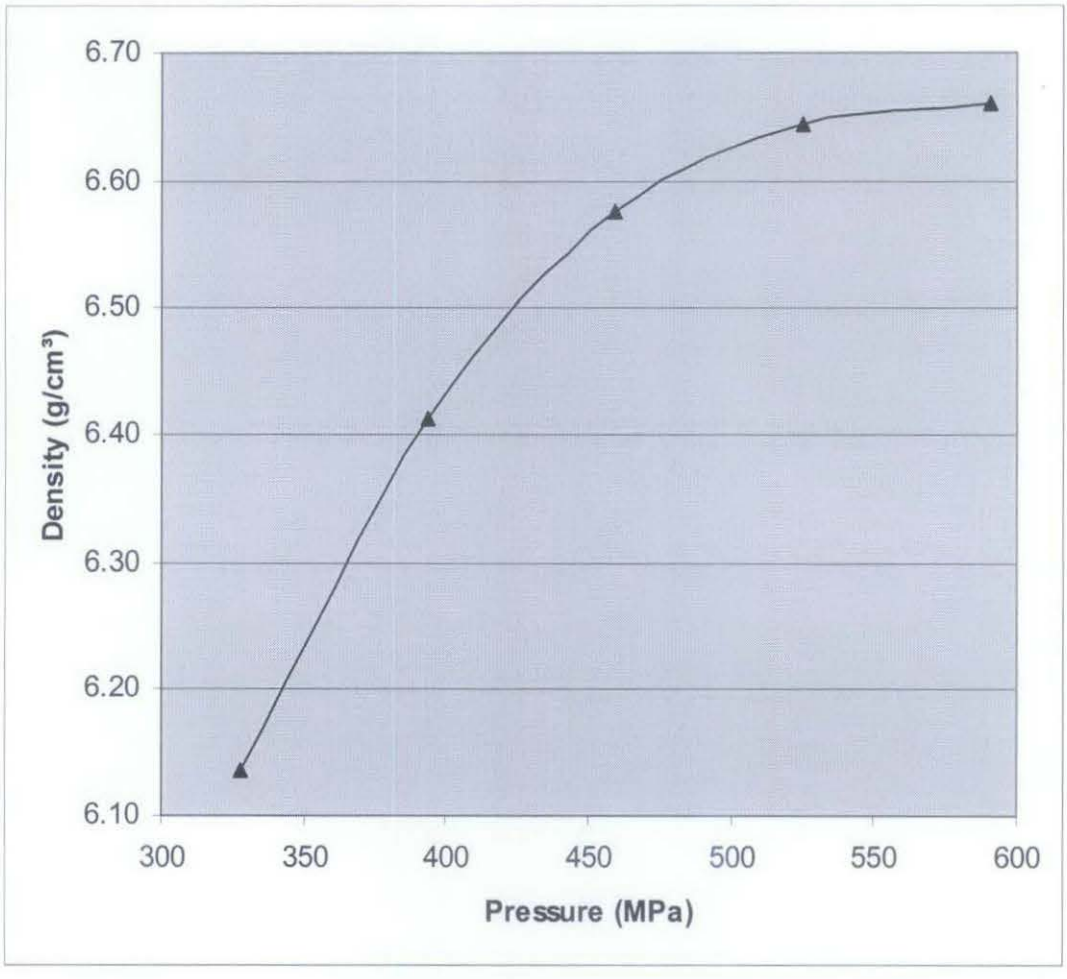


Figure 22: Compressibility curve of the compacted powders mixture.

From the graph, it shows that the compressibility of the mixture increase with pressure and become steady at certain level. The compressibility is high at the lower pressure and slowly decreases at the pressure of 400MPa and beginning to become steady at 550MPa. It is assumed by the student that at the pressure of 600MPa the compressibility of the mixture become steady and it cannot be compress further. That is the highest pressure that the mixture can be compressed and increasing the pressure above that stage will not increase the density of the mixture and may broke the mixture. The highest green density obtained was 6.66 g/cm^3.

The images of the compacted mixture that used the first mixing method are as below:

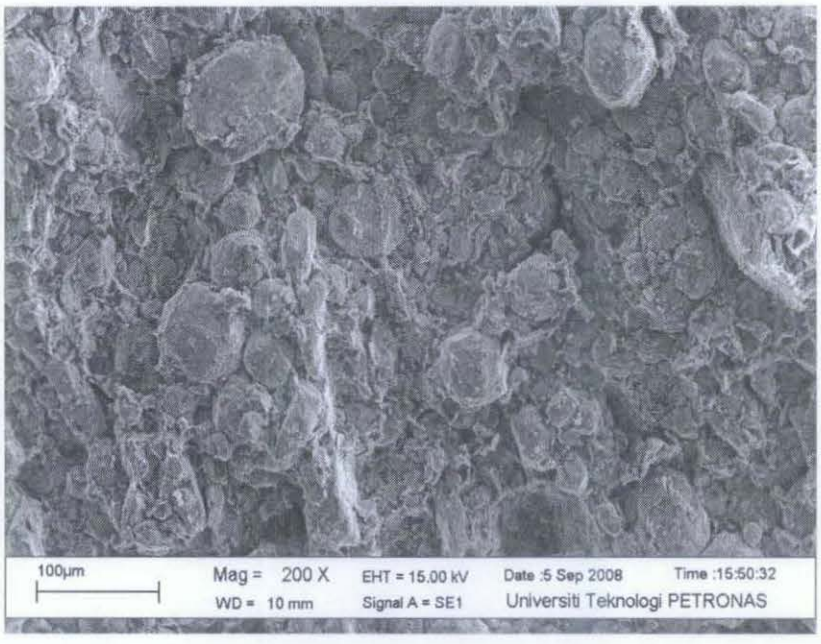


Figure 23: Compacted mixture zooms at 200 times using SEM.

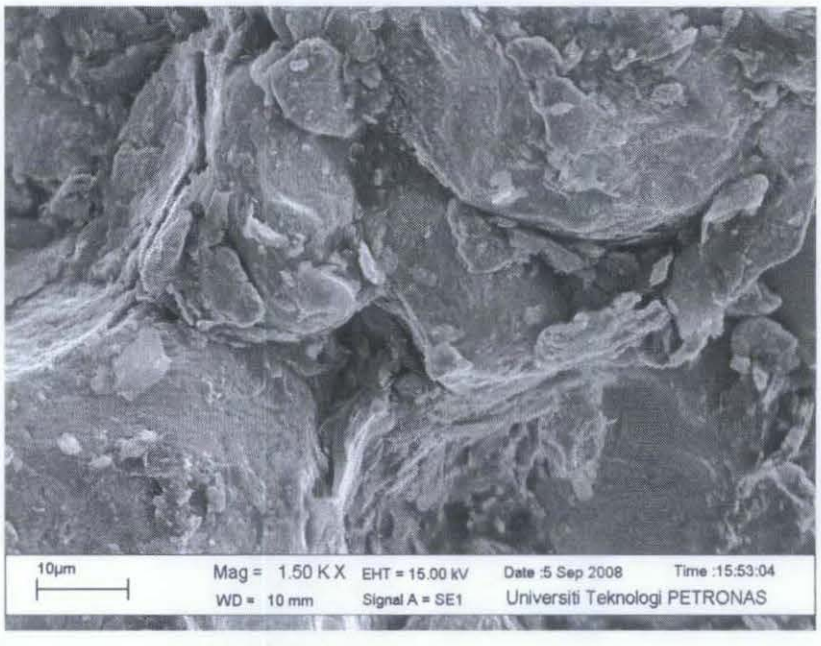


Figure 24: Compacted mixture zooms at 1500 times using SEM.

From the images, it showed that mixture are not so homogeneous and the particle shape of the powder particle are completely destroy due to the usage of the mortar grinder.

The images of the compacted mixture that used the second mixing method are as below:

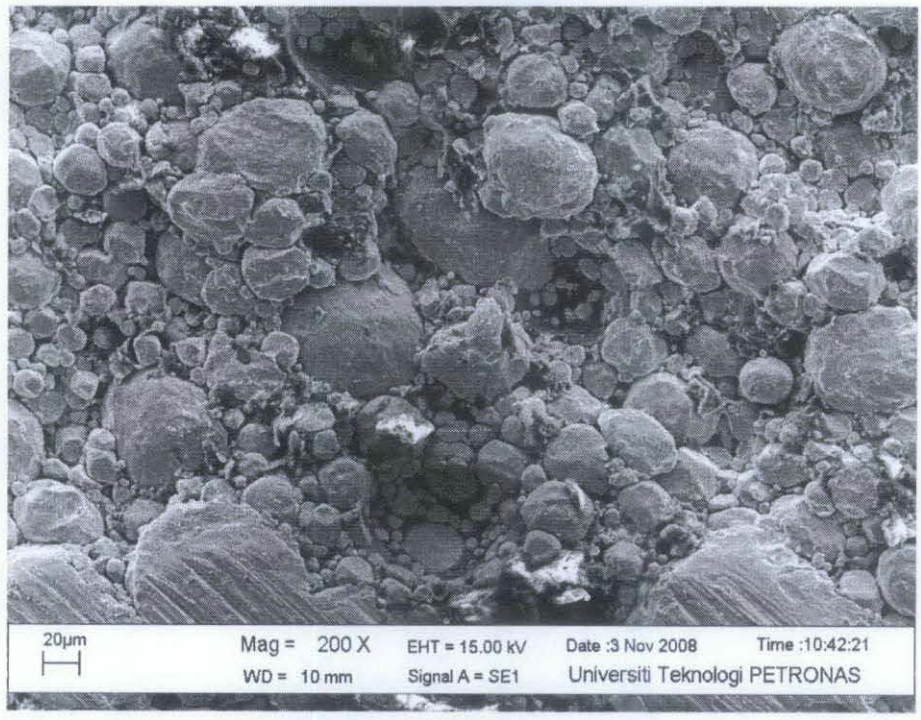


Figure 25: Compacted mixture zooms at 200 times using SEM.

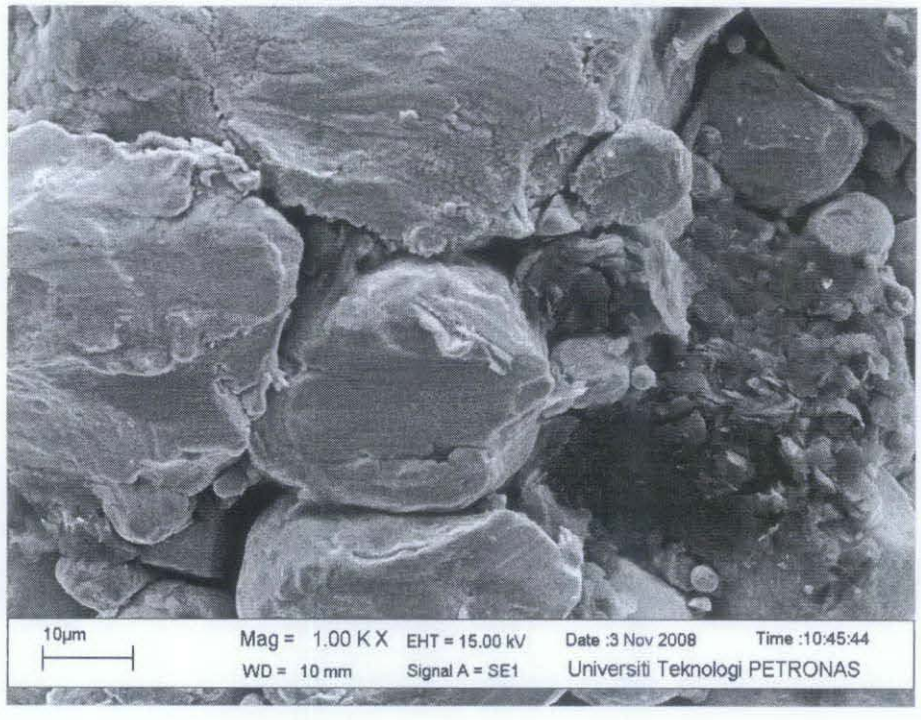


Figure 26: Compacted mixture zooms at 1000 times using SEM.

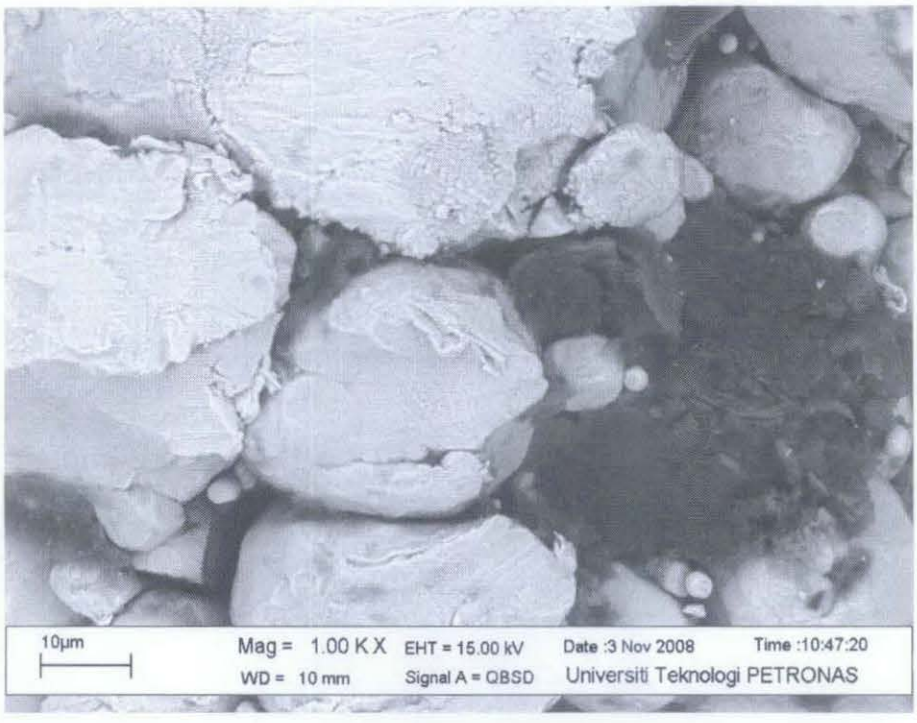


Figure 27: Compacted mixture zooms at 1000 times using QBSD.

The compacted mixture using the second mixing method is more homogenous and the particle shapes of copper powder are still in the round shape.

Using just SEM, student having a difficulty in differentiate between the copper and carbon in that mixture. Then, Quadrant Backscattered Electron Detector (QBSD) has been used to easily differentiate between these two components. QBSD differentiate particle with different density, heavier component resulting in brighter image and lighter component resulting in darker image.

From QBSD images of the compacted mixture, it can be seen clearly that the graphite are filling the pores between copper particles and connected well in the mixture.

4.5 Sintering

Sintering has been done for the compacted pallet using furnace. Sintering time is set to 30 minutes at temperature of 870°C. The density of the sintered mixture measured using Archimedes Method. Weigh balance machine attached with density measurement equipment was used to directly measure the density of the sintered mixture.

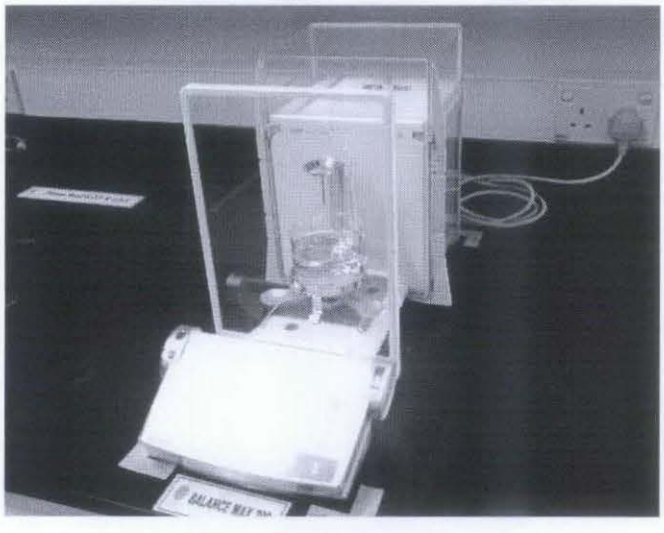


Figure 28: Weigh balance machine attached with density measurement equipment.

Below are the tables of sintered density of different compaction pressure. Sample calculation of sintered density is attached in **Appendix E.**

Table 10: Sintered density of the compacted pallet for different pressure

Compaction Force (N)	Sample Density (g/cm³)			Average sintered density (g/cm³)
	1	2	3	
44482	6.492	6.476	6.493	6.487
53379	6.921	6.914	6.898	6.911
62275	7.140	7.148	7.153	7.147
71172	7.249	7.257	7.265	7.257
80068	7.275	7.268	7.294	7.279

Graph of sintered density versus pressure were plotted to study sinterability of the sintered compacts.

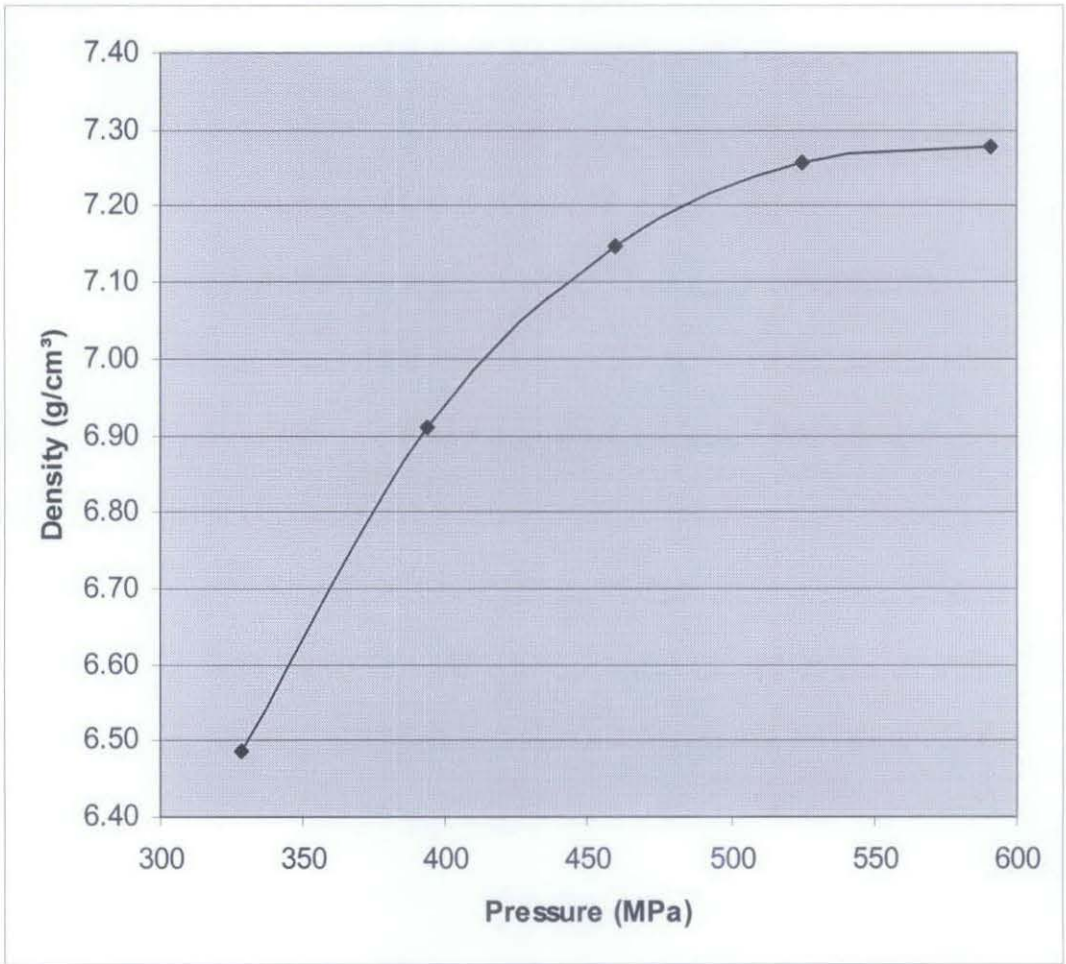


Figure 29: Sinterability (Sintering Curve) of the sintered compacts.

The graph is almost the same as the green density graph, it shows that the compressibility of the mixture increase with pressure and become steady at certain level. It shows the consistency of the compressibility of the mixture before and after the sintering process. Comparing between sintered and compacted mixture, the density of the mixture is increased after sintering process due to the fusion and bonding between the individual particles in the mixture. The highest sintered density obtained was 7.28 g/cm³.

To further study about the sintered mixture, SEM images are gathered. Below are the images of sintered mixture using the first mixing method and sintered in the furnace for 30 minutes at temperature of 850°C.

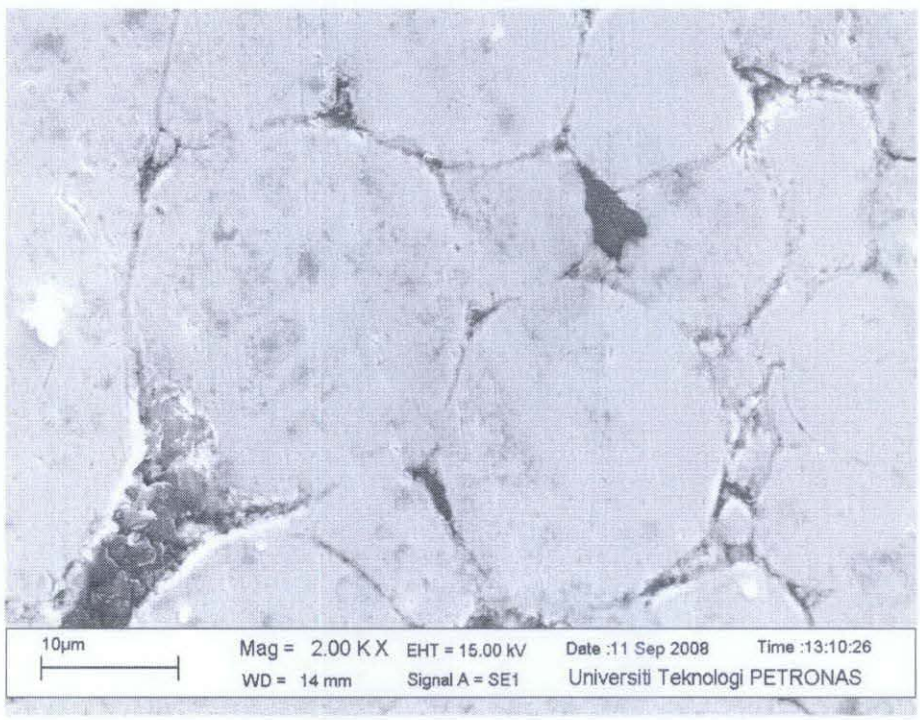


Figure 30: Sintered mixture zooms at 2000 times using SEM.

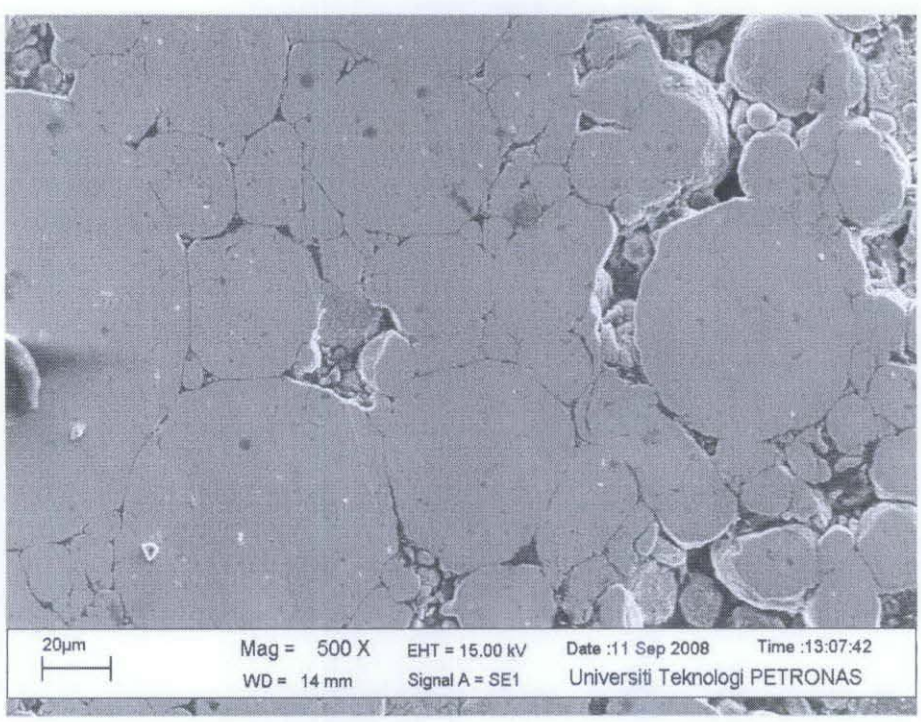


Figure 31: Sintered mixture zooms at 500 times using SEM.

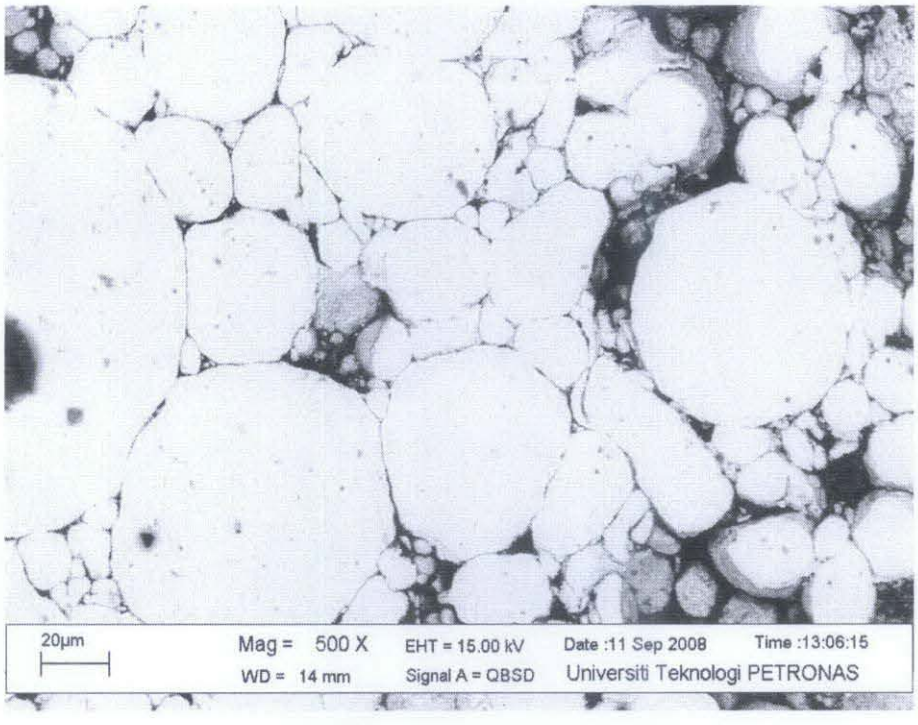


Figure 32: Sintered mixture zooms at 500 times using QBSD.

From the images, it shows that the fusions between particles are not well achieved. The particles can still be seen as single particle and the boundary between particles are shown clearly.

To differentiate between copper and graphite particles, QBSD have been used. From the QBSD image, it shows clearly the individual particle in the mixture. Fusions between particle hardly to see and carbon are scattered randomly in the mixture.

The student concluded that this mixture is not sintered very well and the objective of sintering is not met.

Below are the images of sintered mixture using the second mixing method and sintered in the furnace for 30 minutes at temperature of 870°C.

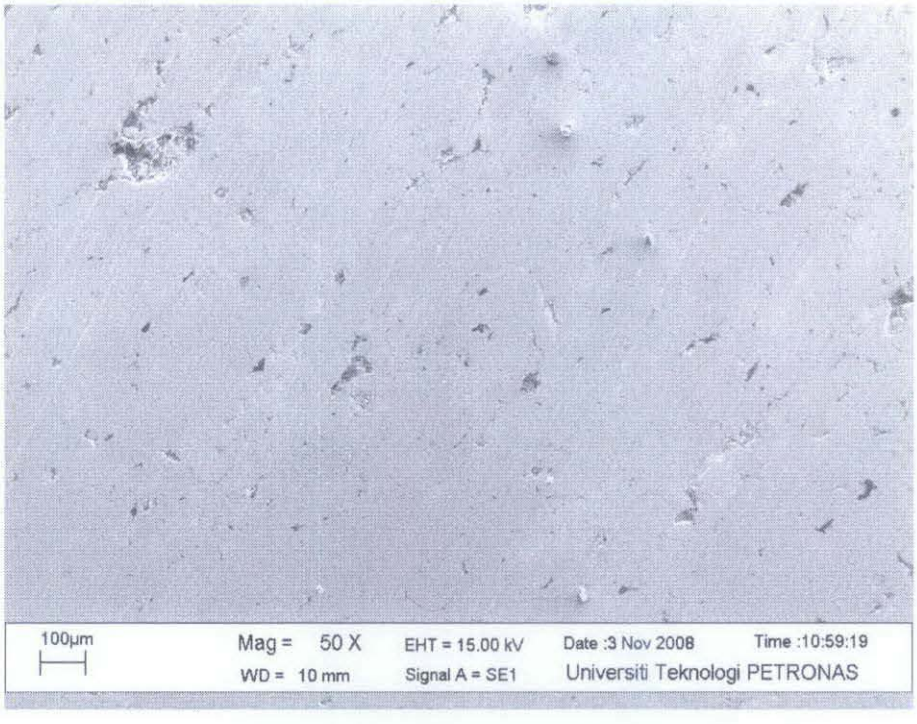


Figure 33: Sintered mixture zooms at 50 times using SEM.

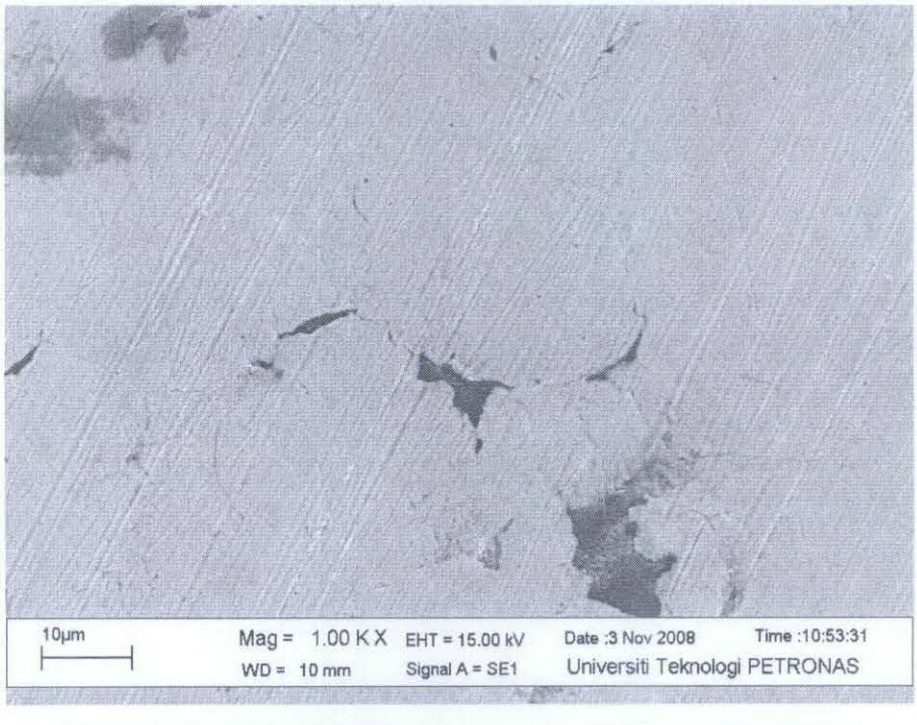


Figure 34: Sintered mixture zooms at 1000 times using SEM.

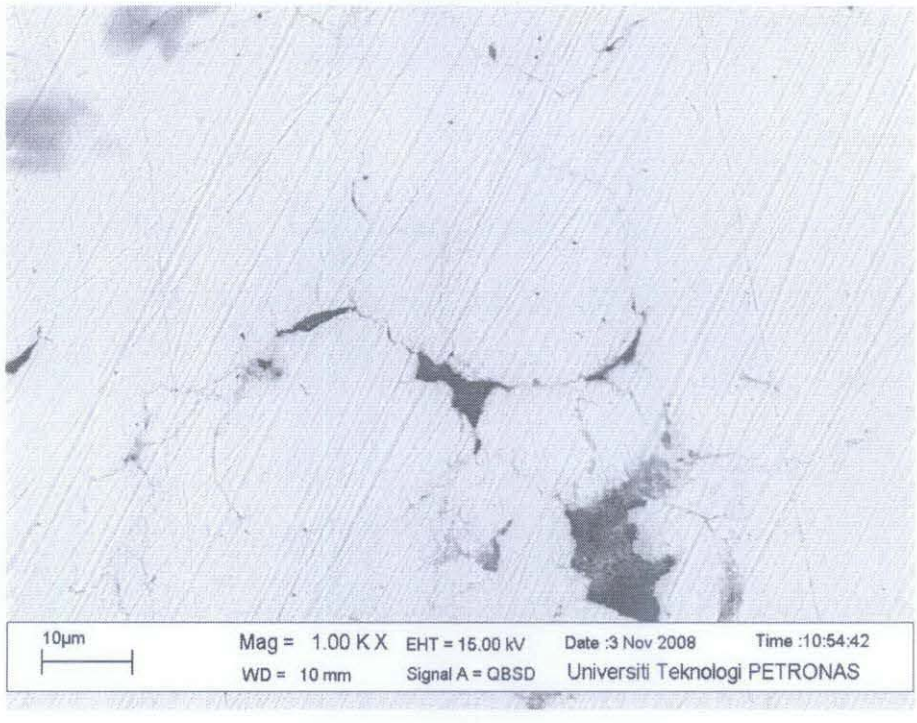


Figure 35: Sintered mixture zooms at 1000 times using QBSD.

From the images, it shows that the mixture completely bond with each other. The region between individual particles is hardly to see. The mixture is homogenous where carbon can be seen fully scattered in the mixture.

The QBSD image also shows the bonding happen between the particles where the individual particle is hardly to differentiate.

To further see contact between copper and carbon in the mixture. The mixture is zooming further.

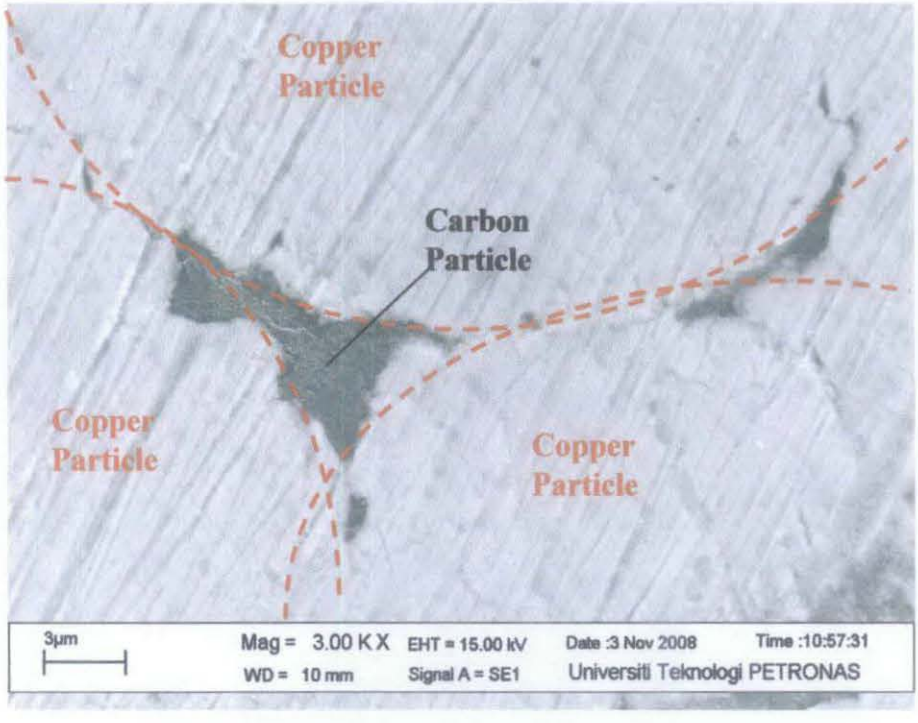


Figure 36: Sintered mixture zooms at 3000 times using SEM.

From this image, copper particle can be clearly seen. The dotted line shows the individual particle of copper. Carbon can be seen clearly at the intersection of the copper particle.

Carbons are not fused with copper directly, it just filling the pores between the particles.

The student concluded that the homogeneity of the mixture is achieved by carbon scattered fully in the mixture. Carbon act as the filler in the copper mixture that fills the gap between particles and does not directly fused with the copper mixture.

4.6 Hardness Measurement

Hardness of every pallet has been measured by using microhardness testing machine. Hardness Vicker scale (HV) that using a diamond pyramid indenter has been chose. The load applied is about 0.2 kg. Below is the figure of the microhardness testing machine. In doing hardness measurement, a small indentation is formed and a microscope is required to obtain the measurement.

The results of the microhardness test of the pallet are as below:

Table 11: Hardness Measurement Reading

Compaction Force (N)	**Reading (HV)**					**Average (HV)**
	1	**2**	**3**	**4**	**5**	
44482	10.9	10.3	12.8	11.5	11.1	11.3
53379	16.7	14.4	16.6	15.7	16.7	16.0
62275	22.2	22.7	21.5	30.3	31.8	25.7
71172	28.8	31.9	22.7	25.6	27.2	27.2
80068	30.2	27.5	25.1	26.8	27.9	27.5

From the table, it shows that the hardness value of the mixture increase with increase in the compaction force until the force of 71172N (16000lb). The hardness of sample 71172N (16000lb) and 80068N (18000lb) has quite the same value.

The microhardness testing machine gives a different value at a different point. The point with high hardness value is due to the present of the carbon in the mixture. The highest average hardness obtained was 27.5 HV

CHAPTER 5
CONCLUSION

From the study, the powder mixture was found more homogenous when the graphite powder was grinded.

Compressibility of the mixture increased with pressure and become constant at 550MPa. The densities of the compacts were found increases after sintering process. The highest densities of green and sintered were 6.66 g/cm^3 and 7.28 g/cm^3 respectively. The sintered compacts were found nearly to 91% of the theoretical density. Results of particles diffusion were clearly observed.

The hardness of the sintered compacts was increased with temperature and pressure until the sintering plateau is reached. The highest average hardness of the mixture was 27.5 HV.

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10. www.alfa.com

APPENDICES

APPENDIX A

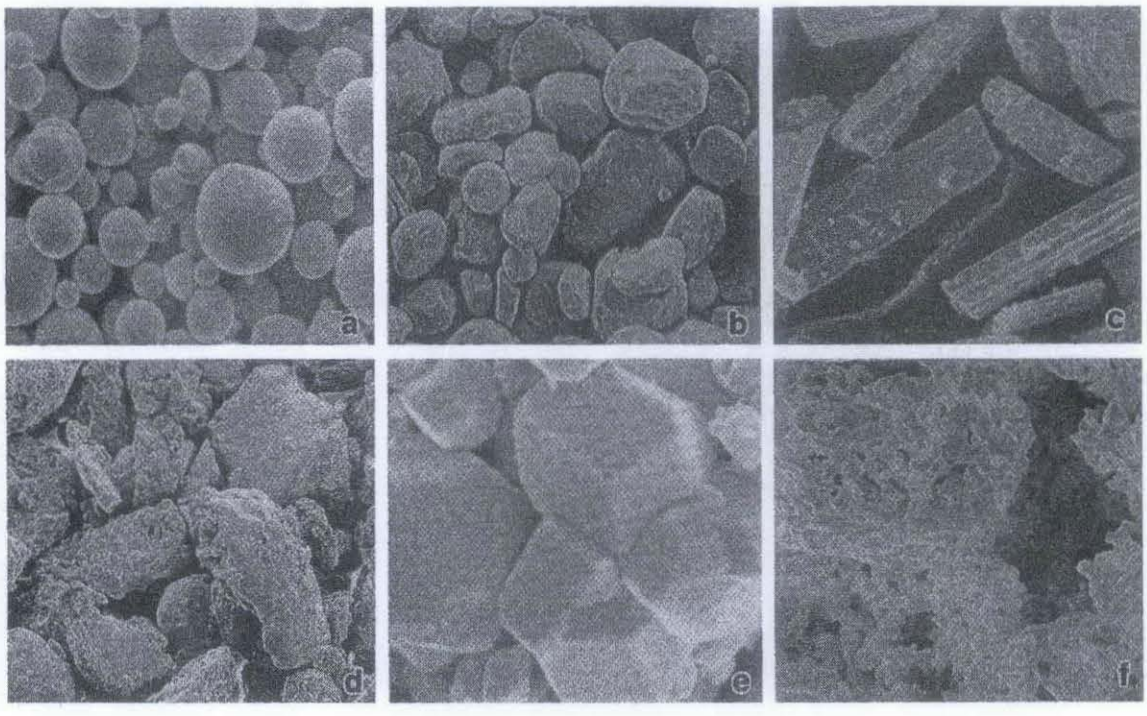

Figure A1: Diversity shapes in powders using SEM; (a)spherical; (b)rounded; (c)angular; (d)irregular; (e)polygonal or cubic; (f)sponge. [9]

APPENDIX B

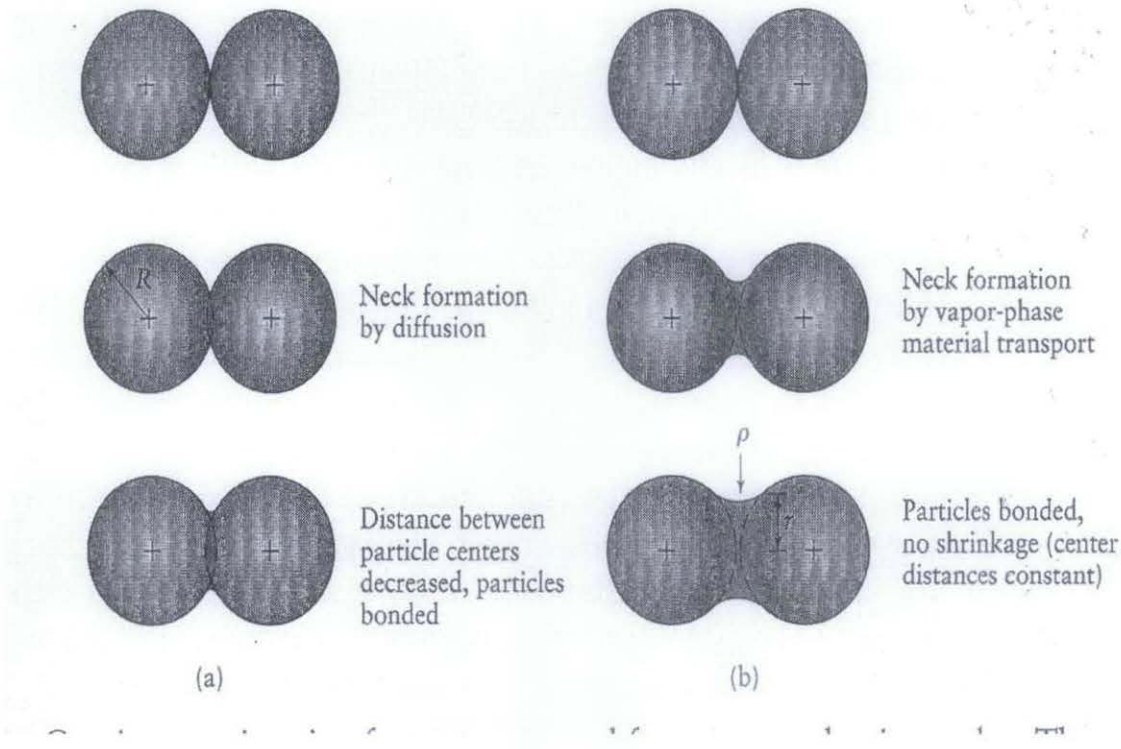

Figure B1: Shematic Illustration of two mechanisms for sintering metal powders: (a) solid-state material transport; and (b) liquid phase material transport. R = particle radius, r = neck radius, and ρ = neck profile radius. [5]

APPENDIX C

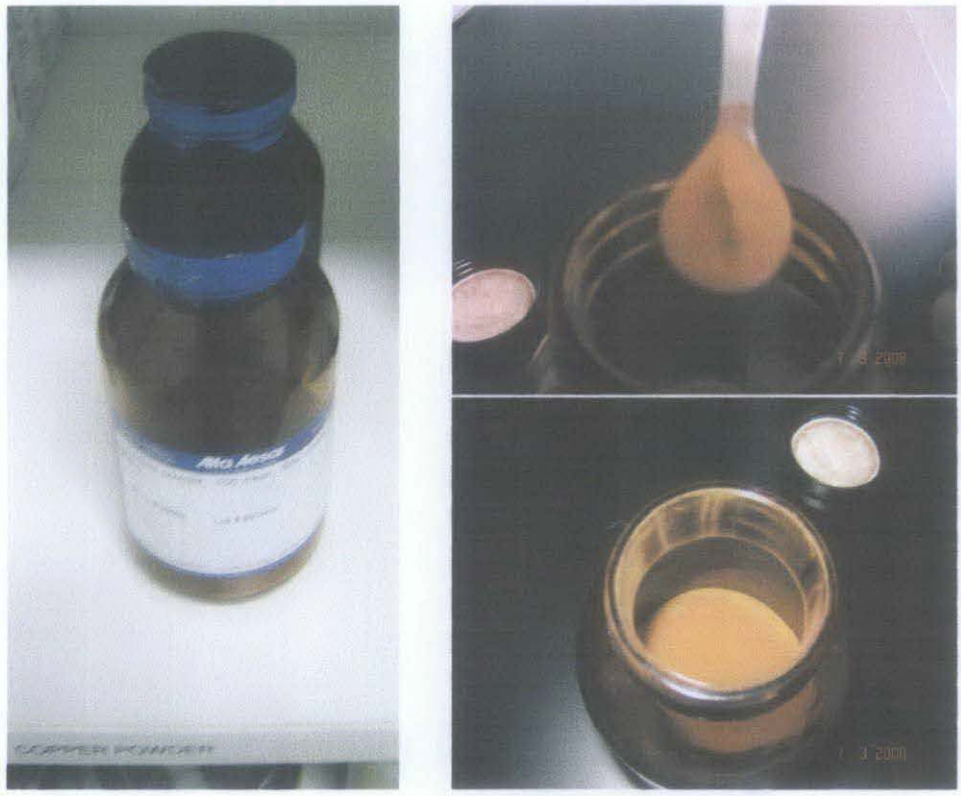

Figure C1: Copper powder (#00584 Alfa Aesar) available in the UTP laboratory

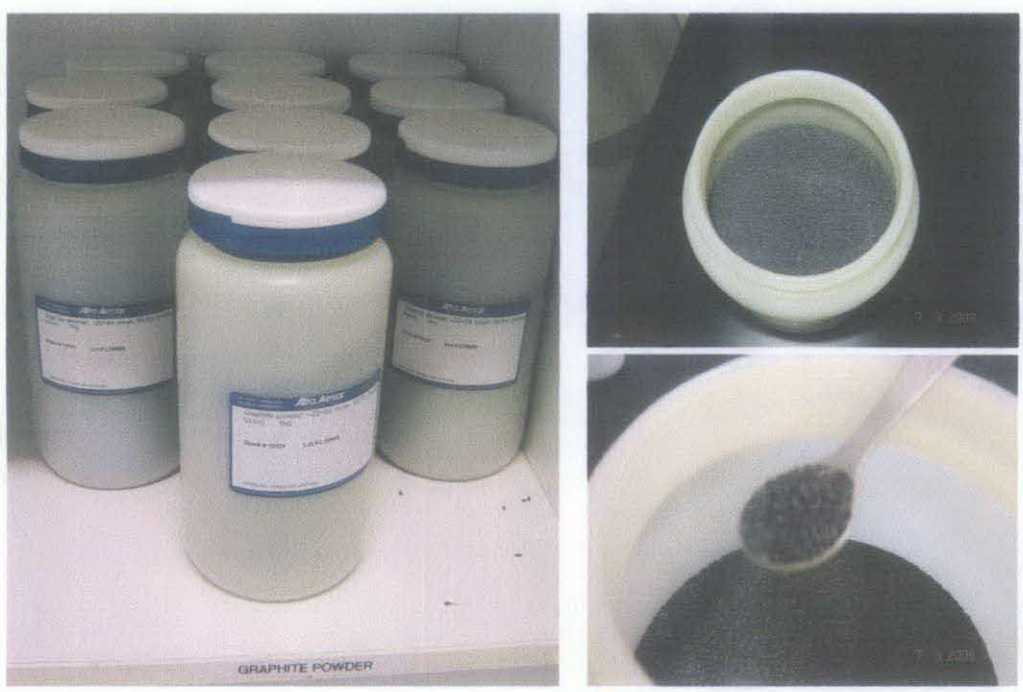

Figure C2: Graphite powder (#10131 Alfa Aesar) available in the UTP laboratory

APPENDIX D

General Information	
Form:	Powder
Color:	Copper colored
Odor:	Odorless
Change in condition	
Melting point / Melting range:	1083°C (1981°F)
Boiling point / Boiling range:	2595°C (4703°F)
Sublimation temperature / start:	Not determined
Flash point:	Not applicable
Flammability (solid, gaseous)	Highly flammable
Ignition temperature:	Not determined
Decomposition temperature:	Not determined
Explosion limits	
Lower:	Not determined
Upper:	Not determined
Vapor pressure:	Not determined
Density at 20°C (68°F):	8.94 g/cm^3

Table D1: Physical and chemical properties of copper powder produce by Alfa Aesar, stock number 00584 based on Material Safety Data Sheet (MSDS).

General Information	
Form: Color: Odor:	Solid Black Odorless
Change in condition Melting point / Melting range: Boiling point / Boiling range: Sublimation temperature / start:	 3652 - 3697°C (6606 - 6687°F) Not determined Not determined
Flash point:	Not applicable
Ignition temperature:	Not determined
Decomposition temperature:	Not determined
Danger of explosion:	Product does not present an explosion hazard
Explosion limits Lower: Upper:	 Not determined Not determined
Vapor pressure:	Not determined
Density at 20°C (68°F):	2.25 g/cm^3
Solubility in / Miscibility with water:	Insoluble

Table D2: Physical and chemical properties of graphite powder produce by Alfa Aesar, stock number 10131 based on Material Safety Data Sheet (MSDS).

APPENDIX E

Sample calculation the weight fraction of mixture

Total weight of mixture = 5 g

Ratio of sample = 9.85:0.15 (98.5wt% copper powder, 1.5wt% graphite powder)

Weight of copper powder = $\frac{98.5}{100} \times 5g = 4.925$ g

Weight of graphite powder = (5 – 4.925) g = 0.075 g

Weight of binder added = 2wt% of mixture

Weight of binder = $\frac{2}{100} \times 5g = 0.1$ g

Sample calculation the density of green compact

Pallet diameter, D = 1.314 cm

Pallet thickness, t = 0.598 cm

Pallet volume, V = π x r^2 x t = π x $(1.314/2)^2$ x 0.598 = 0.8109 cm^3

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

Pallet weight = 4.9838 g

Pallet density, $\rho = \frac{4.9838}{0.8109} = 6.146 g/cm^3$

Sample calculation the density of sintered pallet

Sintered density,

$$\rho = \frac{A}{A-B}(\rho_O - \rho_L) + \rho_L$$

ρ = density of sample (g/cm³)

A = weight of sample in air (g)

B = weight of sample in auxilary liquid.

ρ_O = density of auxilary liquid (0.99777 g/cm³ for distilled water @ 22.1°C)

ρ_L = air density (0.0012 g/cm³)

Example sample compact for 16000lb,

A = 4.9847g,
B = 4.3619g,

$$\rho = \frac{4.9674}{4.9674-4.2851}(0.99777-0.0012)+0.0012$$

Sintered density, ρ = 7.257 g/cm³

APPENDIX F (SEM Images of the Powders)

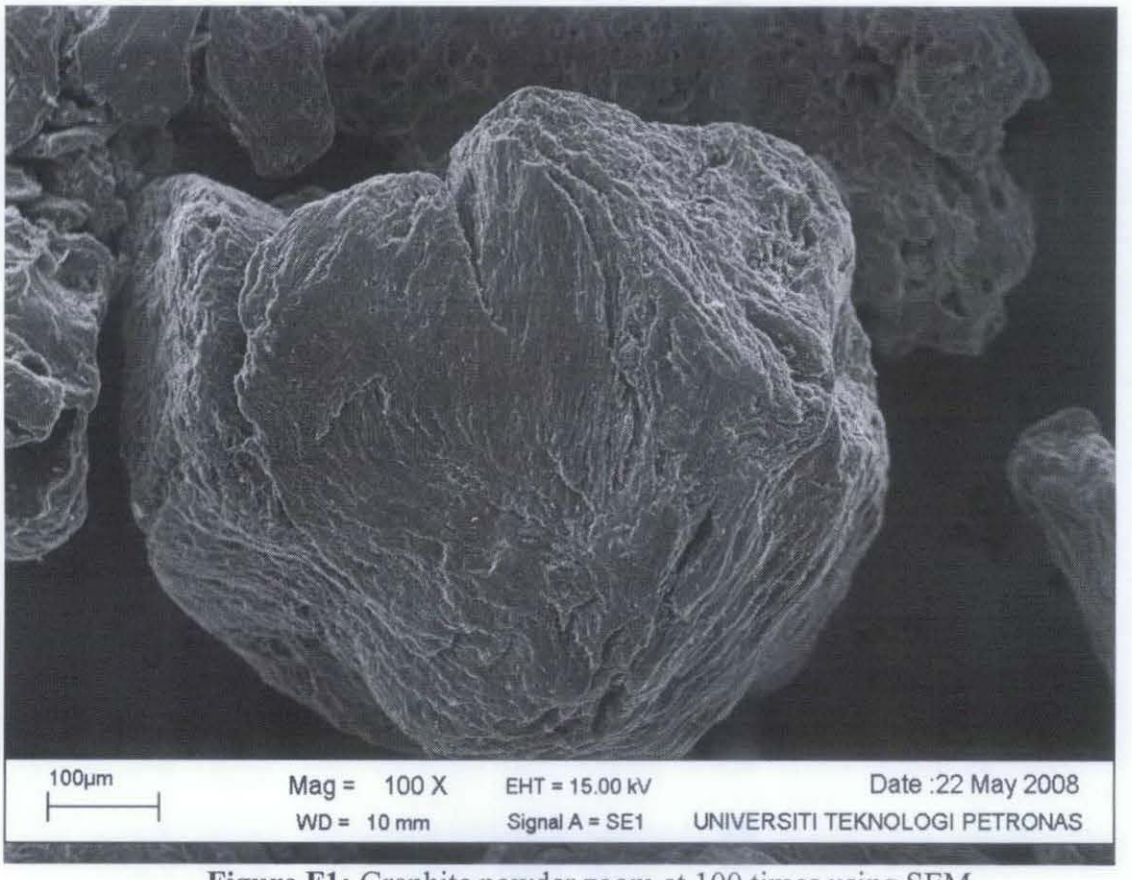

Figure F1: Graphite powder zoom at 100 times using SEM.

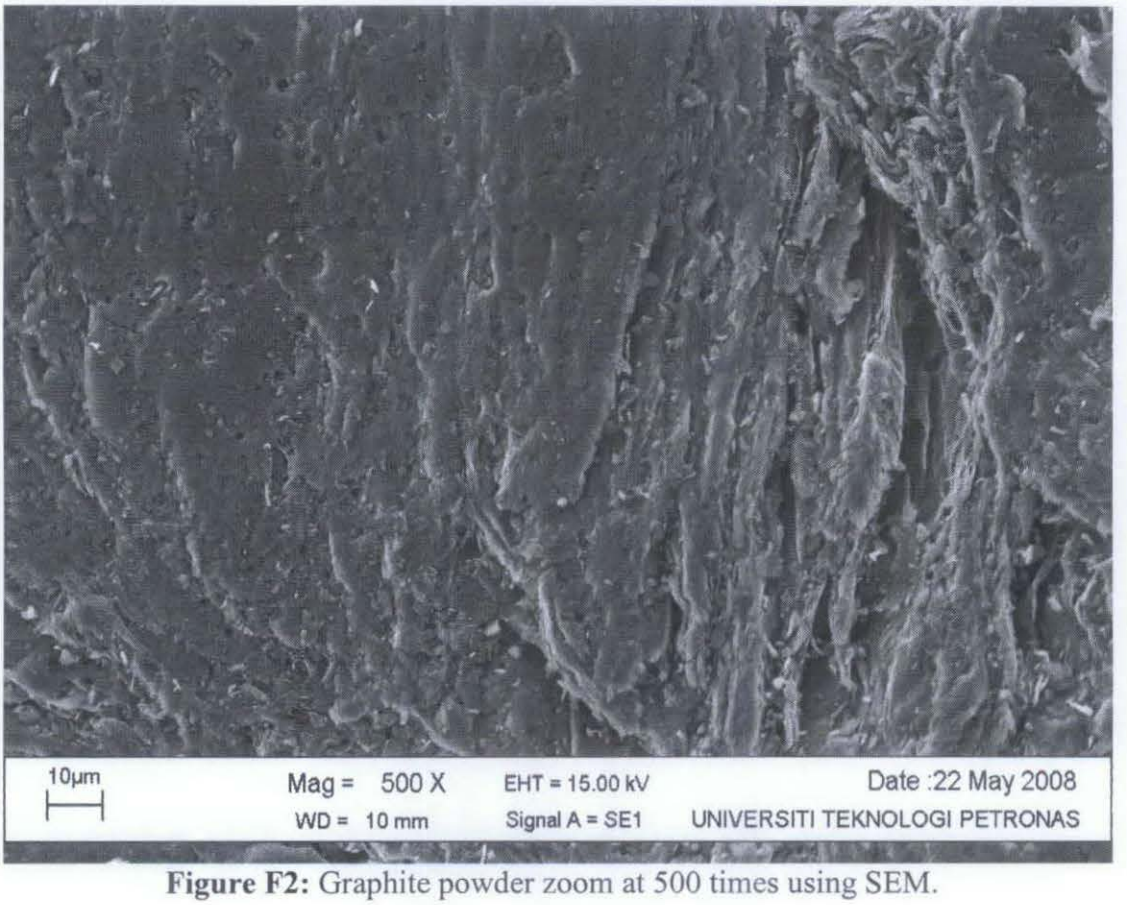

Figure F2: Graphite powder zoom at 500 times using SEM.

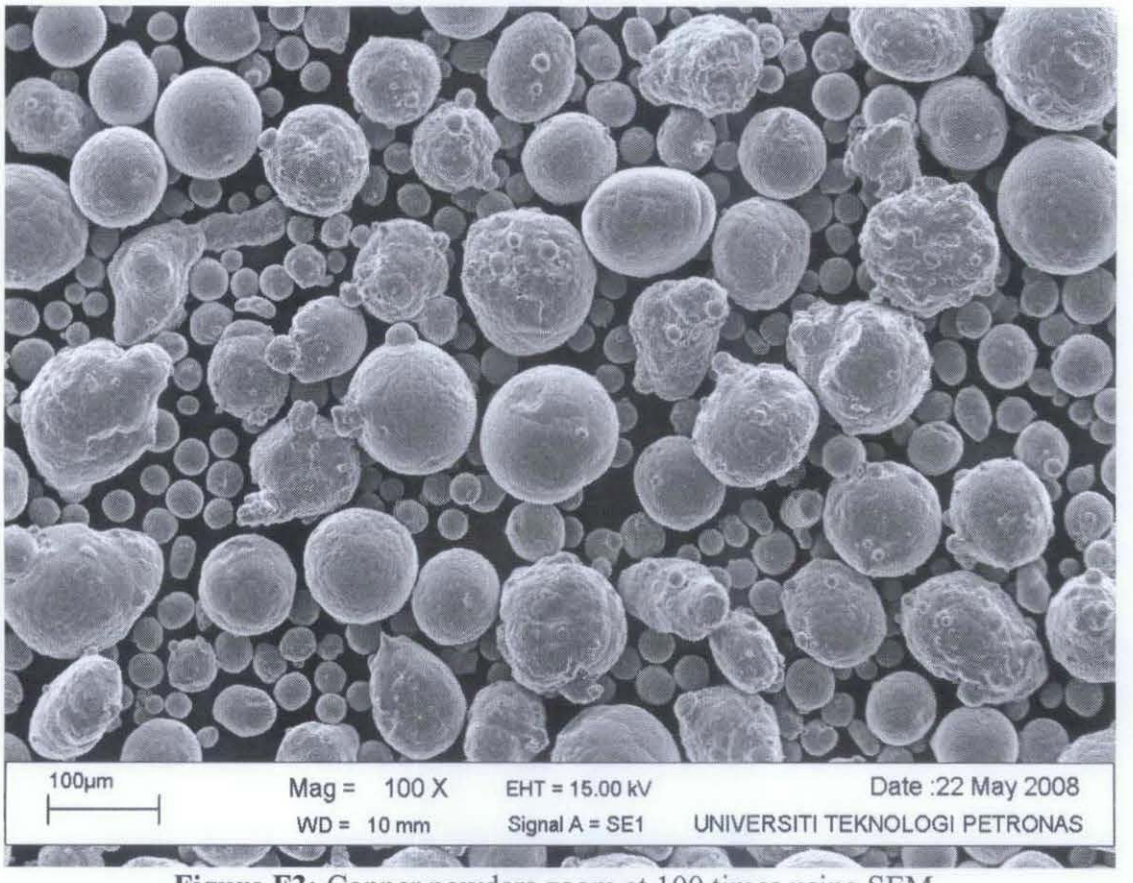

Figure F3: Copper powders zoom at 100 times using SEM.

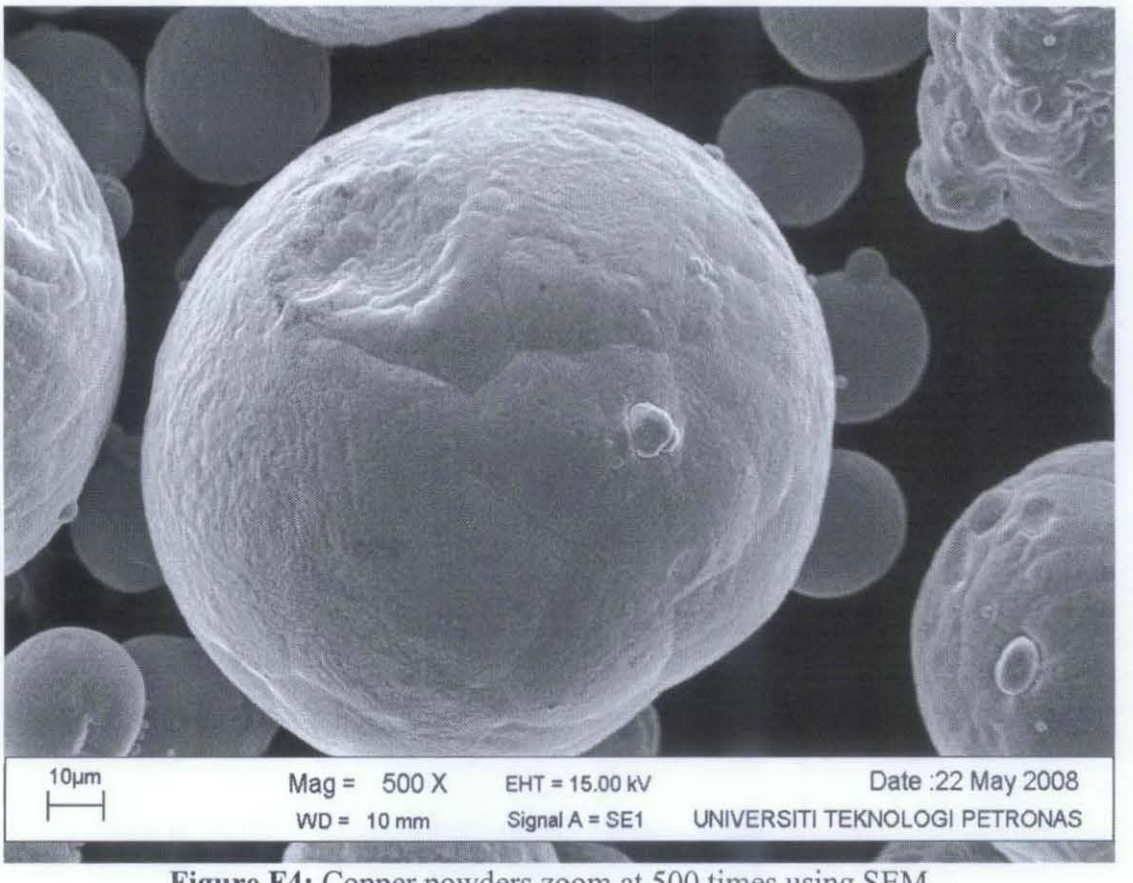

Figure F4: Copper powders zoom at 500 times using SEM.

APPENDIX G (SEM Images of the Mixture using 1st Mixing Method)

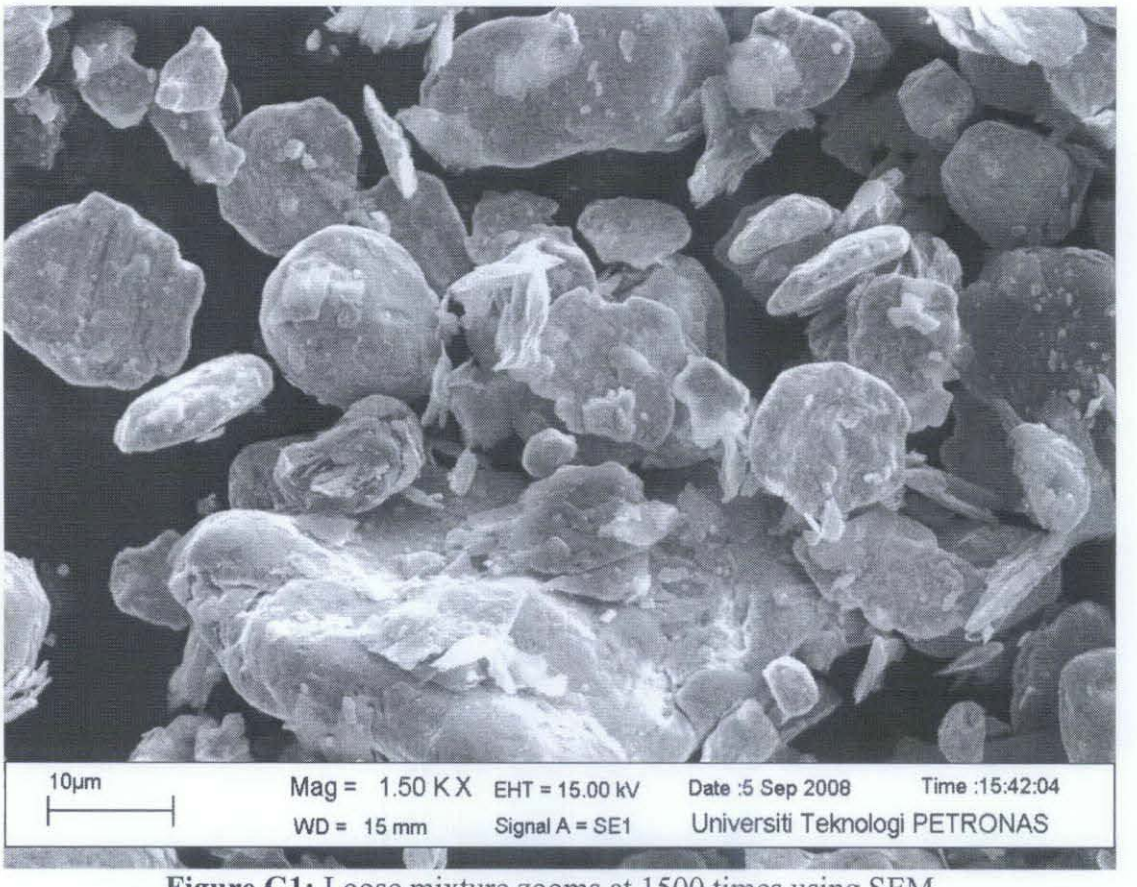

Figure G1: Loose mixture zooms at 1500 times using SEM.

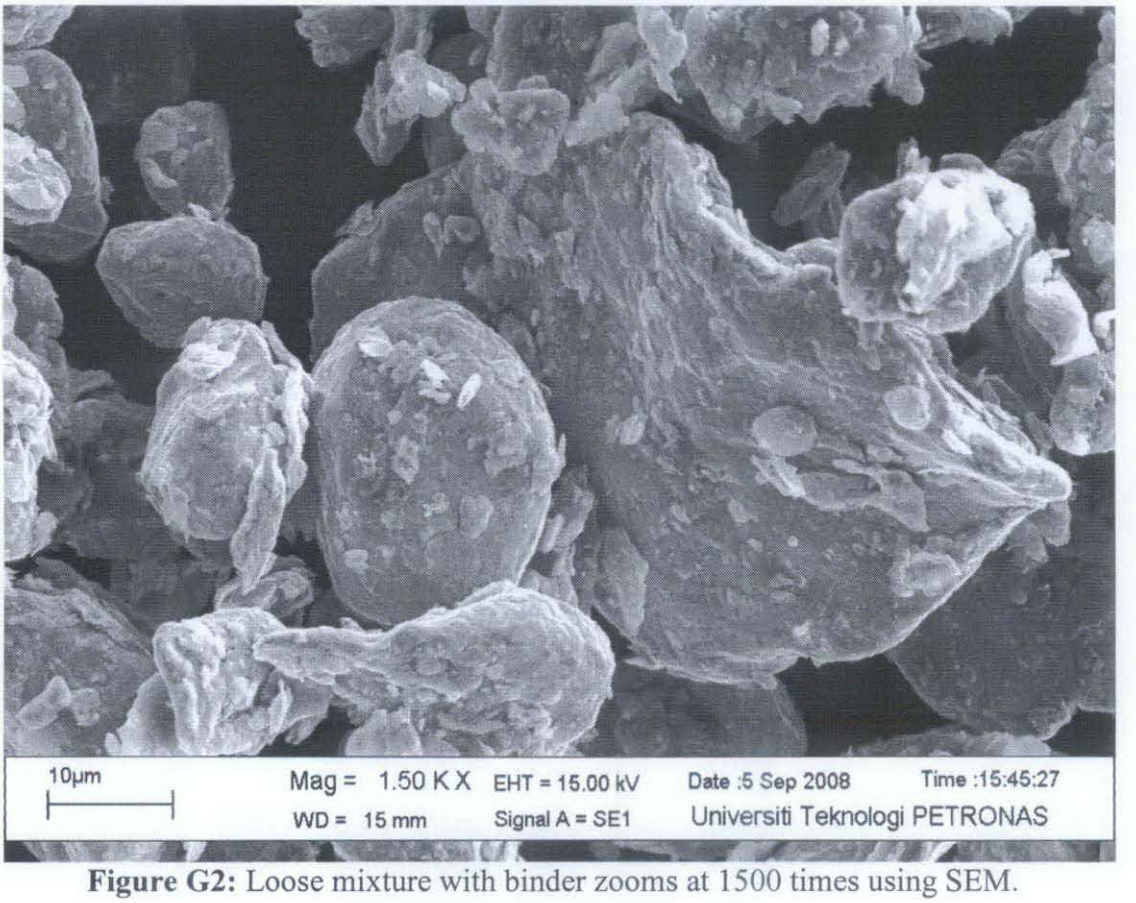

Figure G2: Loose mixture with binder zooms at 1500 times using SEM.

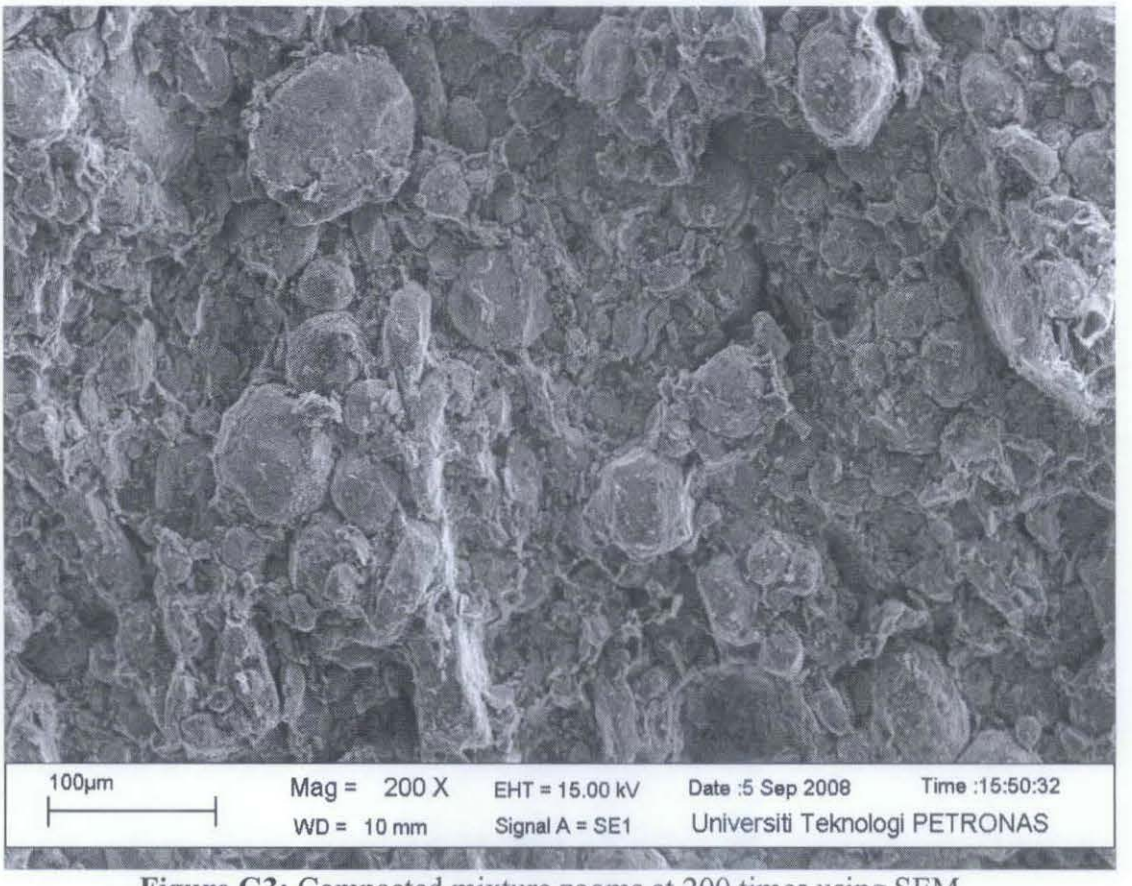

Figure G3: Compacted mixture zooms at 200 times using SEM.

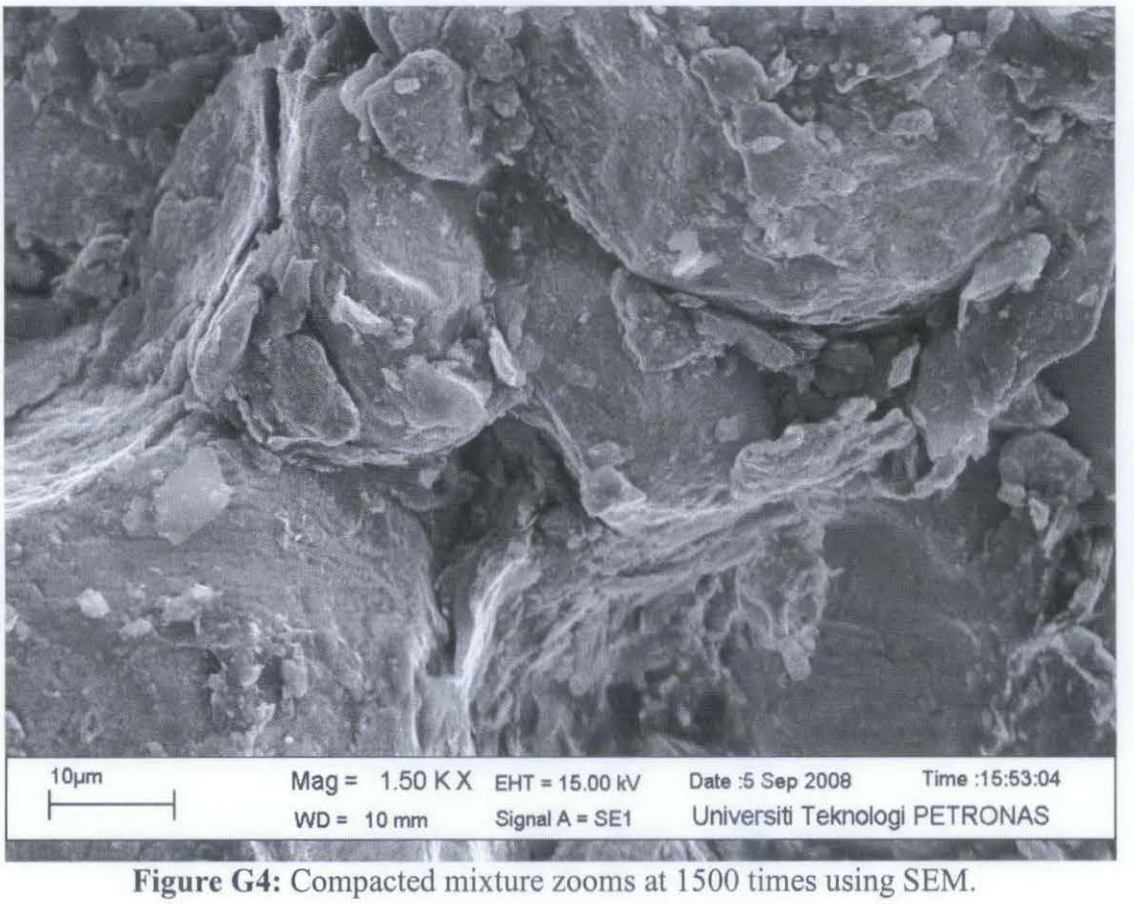

Figure G4: Compacted mixture zooms at 1500 times using SEM.

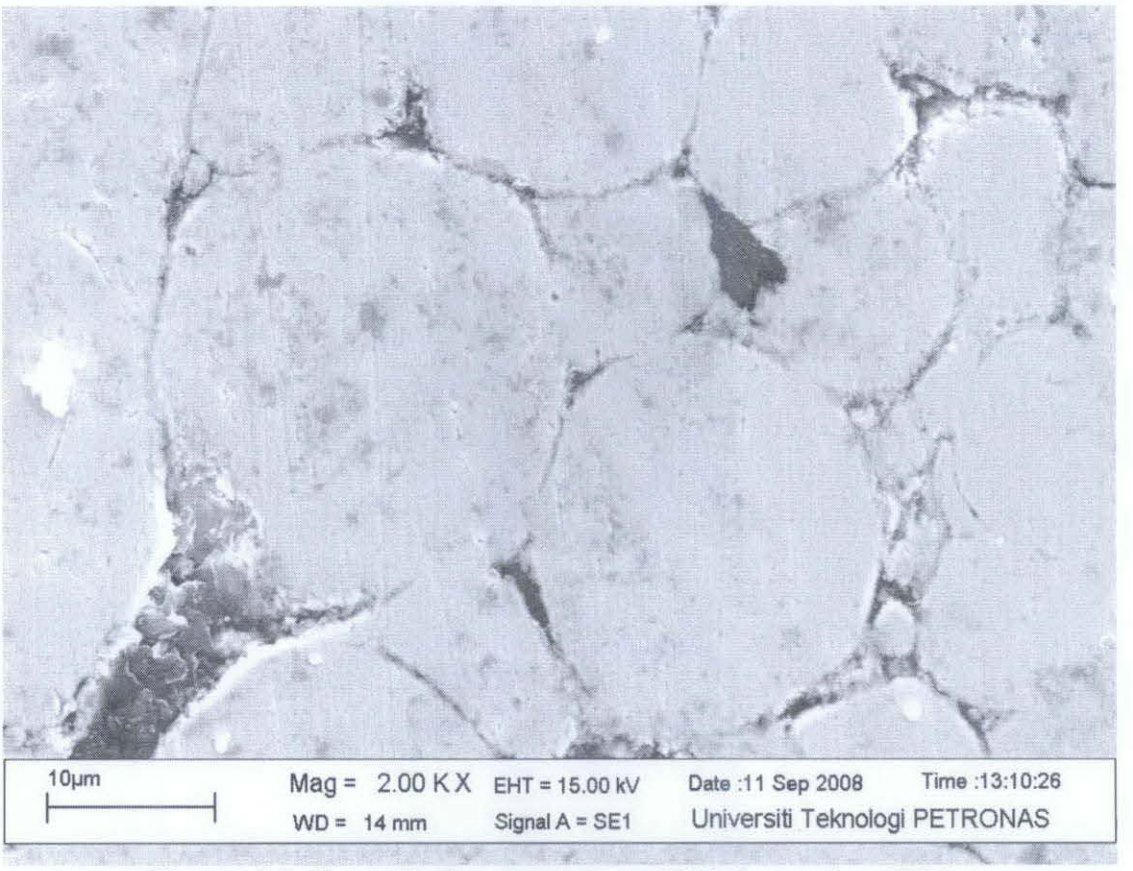

Figure G5: Sintered mixture zooms at 2000 times using SEM.

Figure G6: Sintered mixture zooms at 10000 times using SEM.

APPENDIX H (SEM Images of the Mixture using 2nd Mixing Method)

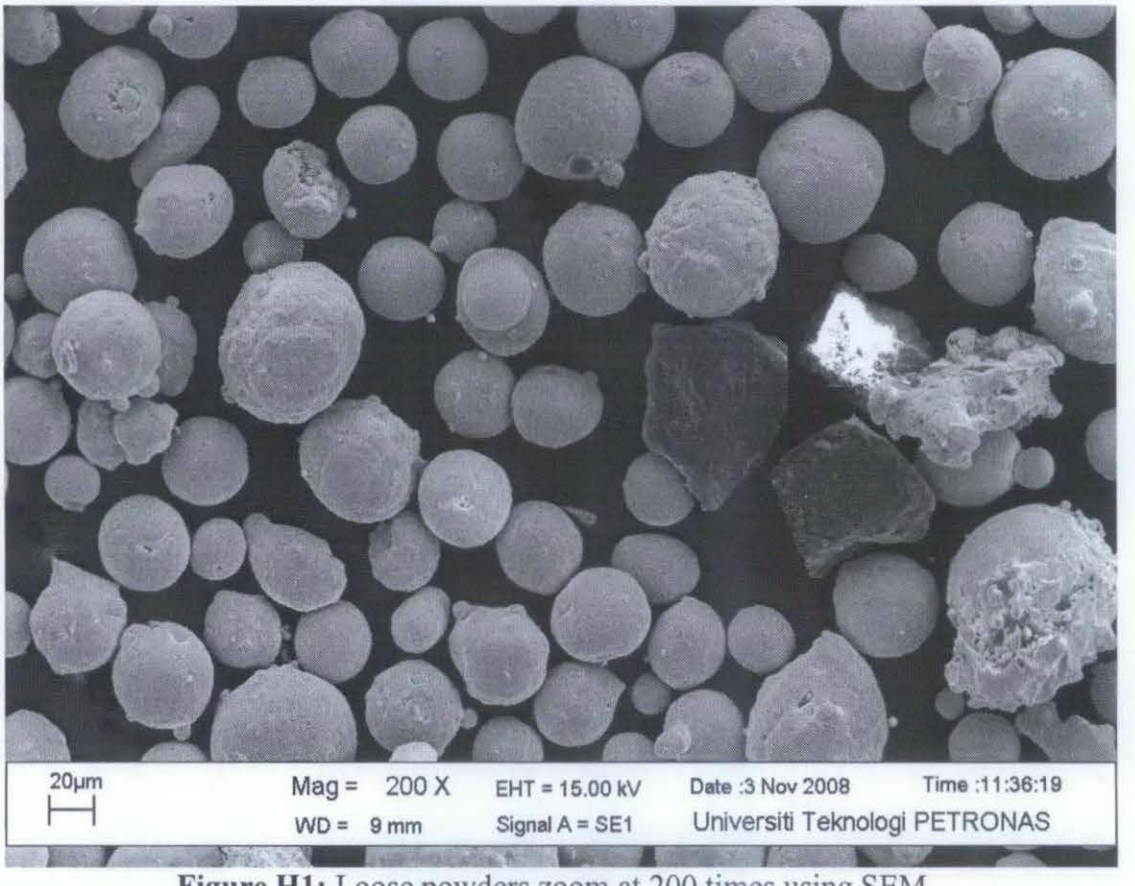

Figure H1: Loose powders zoom at 200 times using SEM.

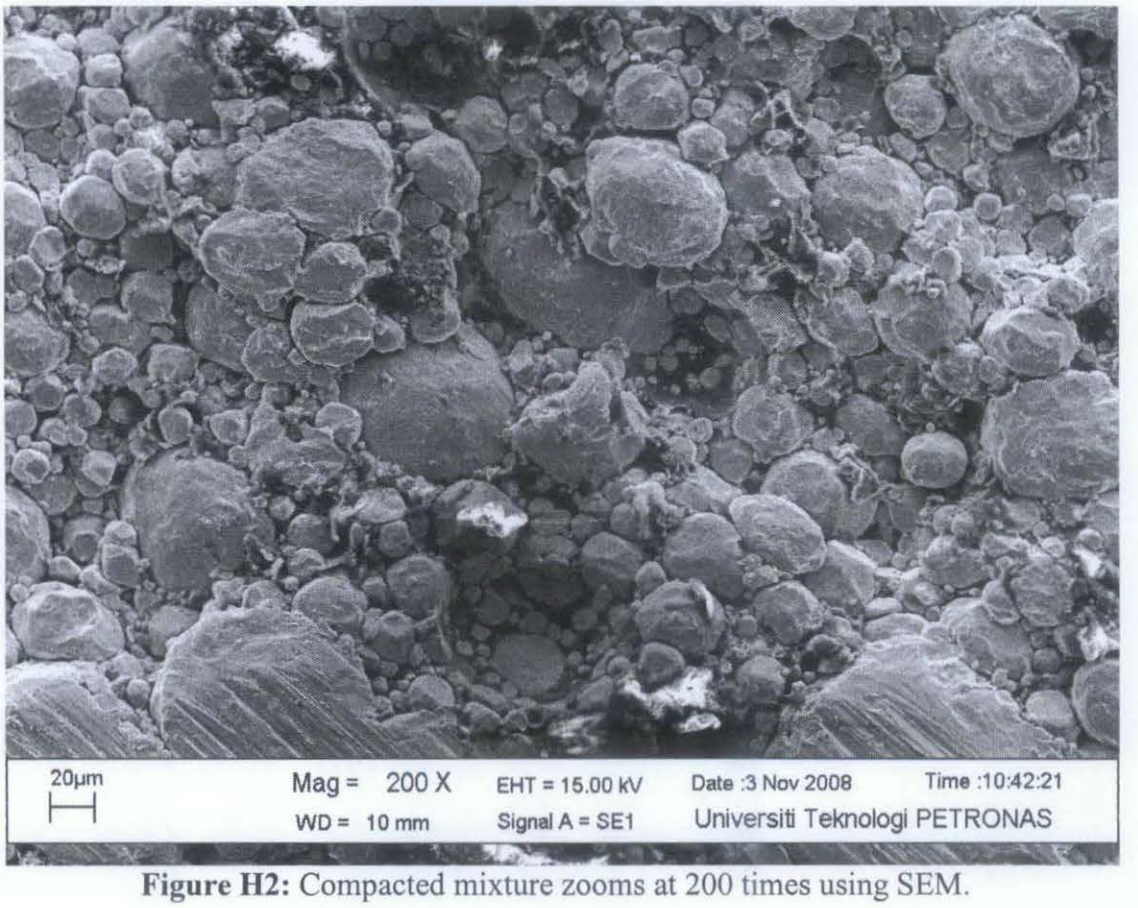

Figure H2: Compacted mixture zooms at 200 times using SEM.

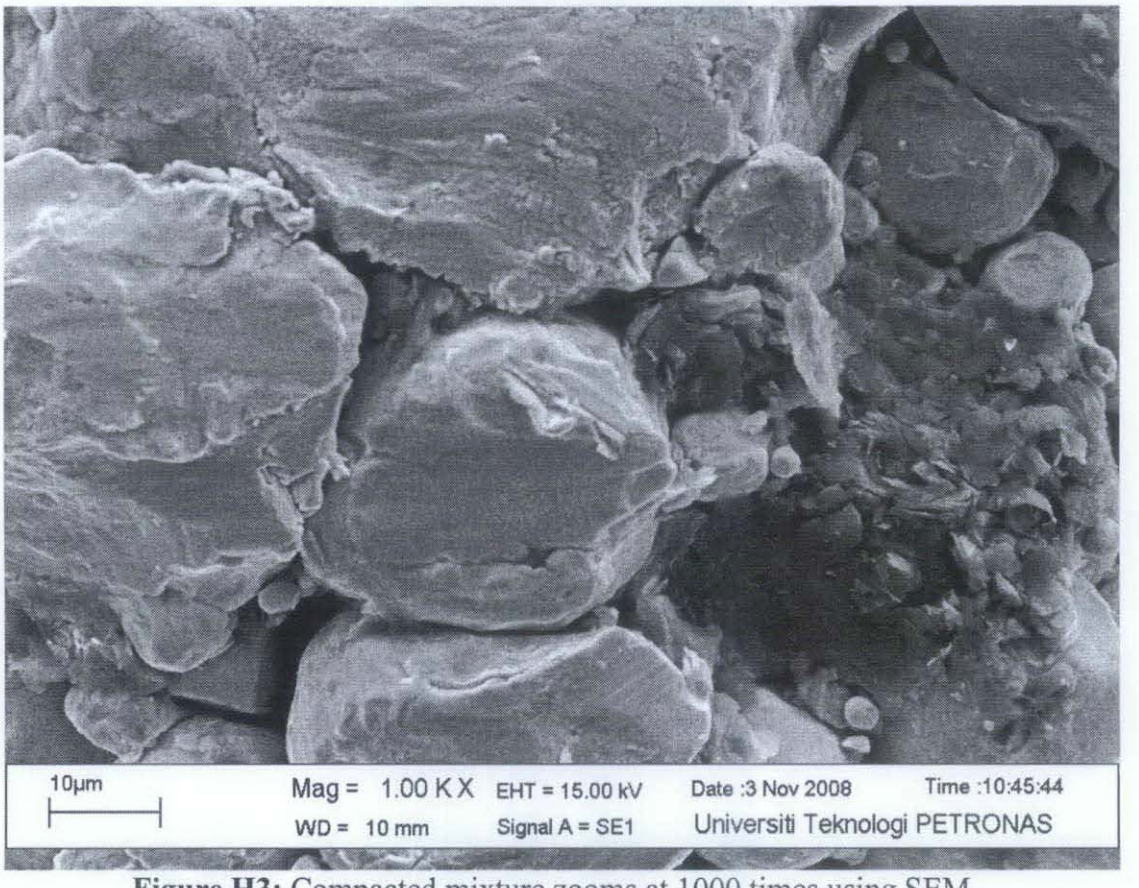

Figure H3: Compacted mixture zooms at 1000 times using SEM.

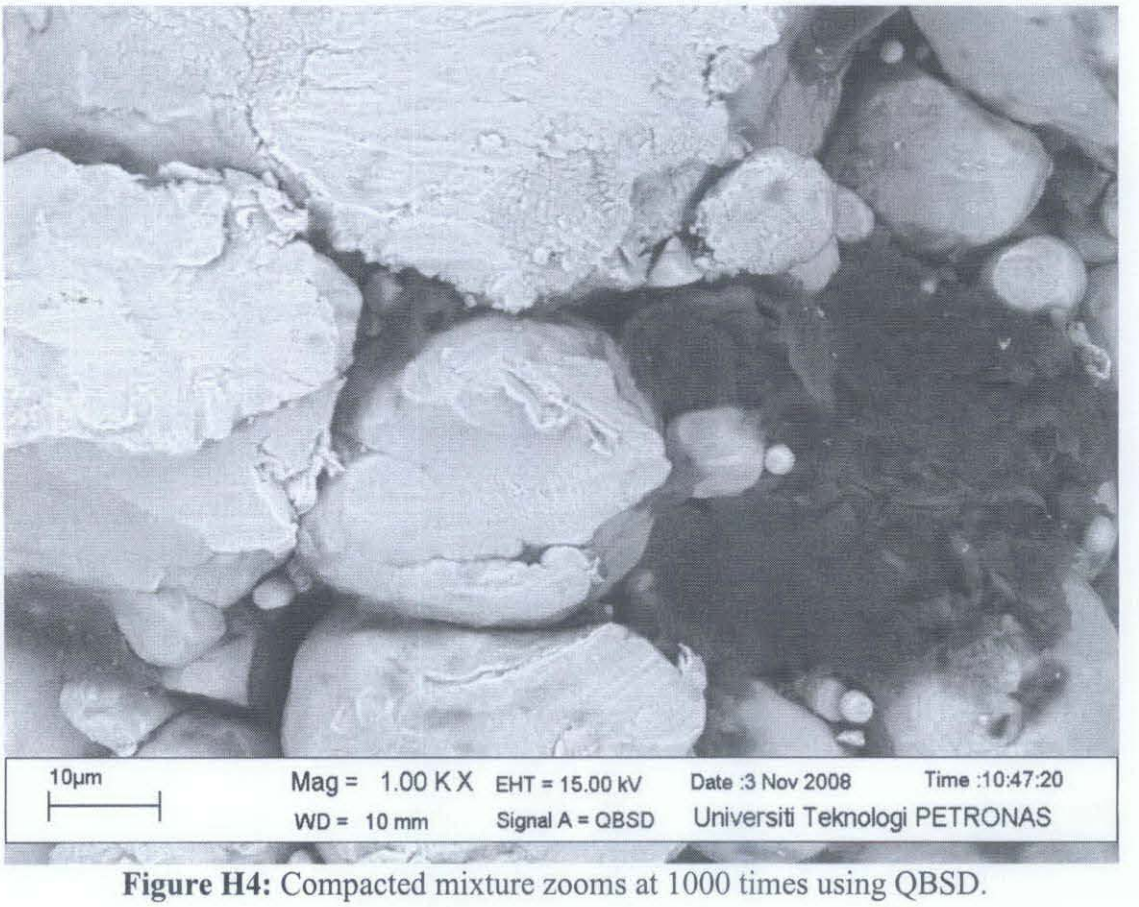

Figure H4: Compacted mixture zooms at 1000 times using QBSD.

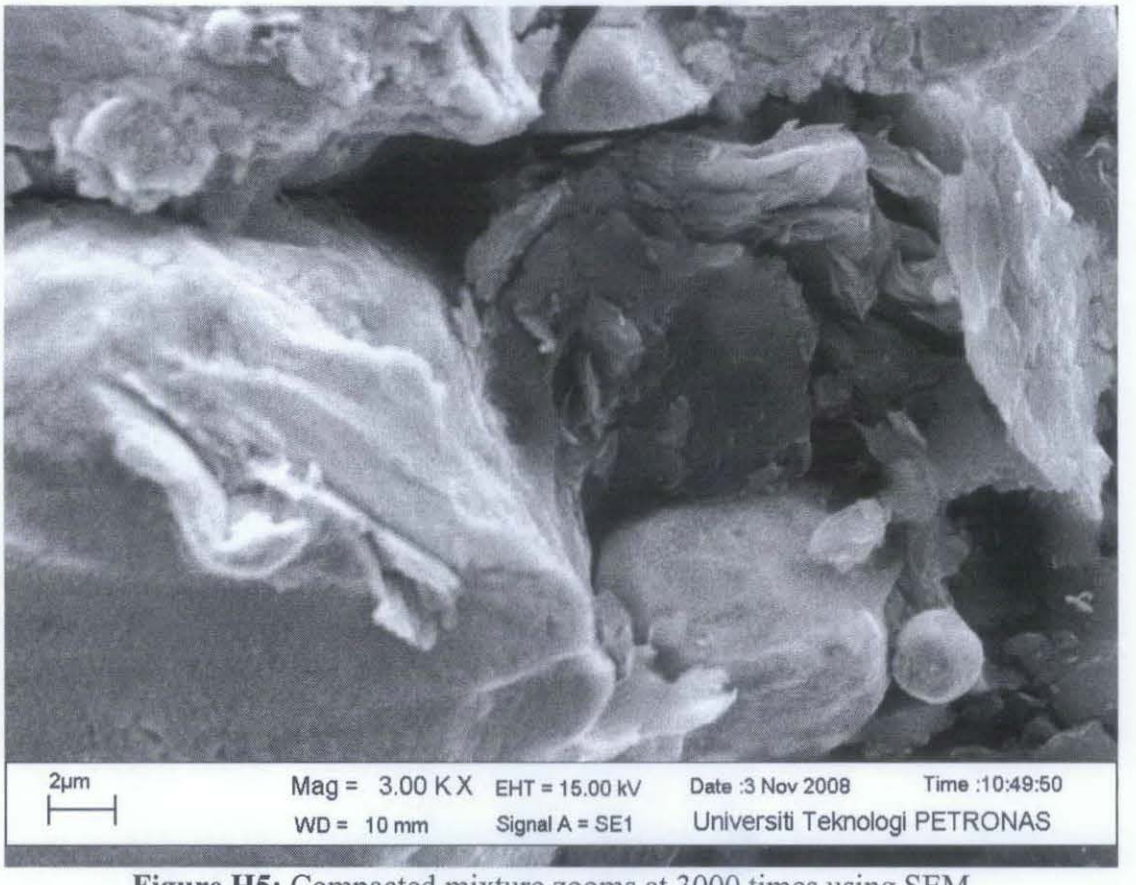

Figure H5: Compacted mixture zooms at 3000 times using SEM.

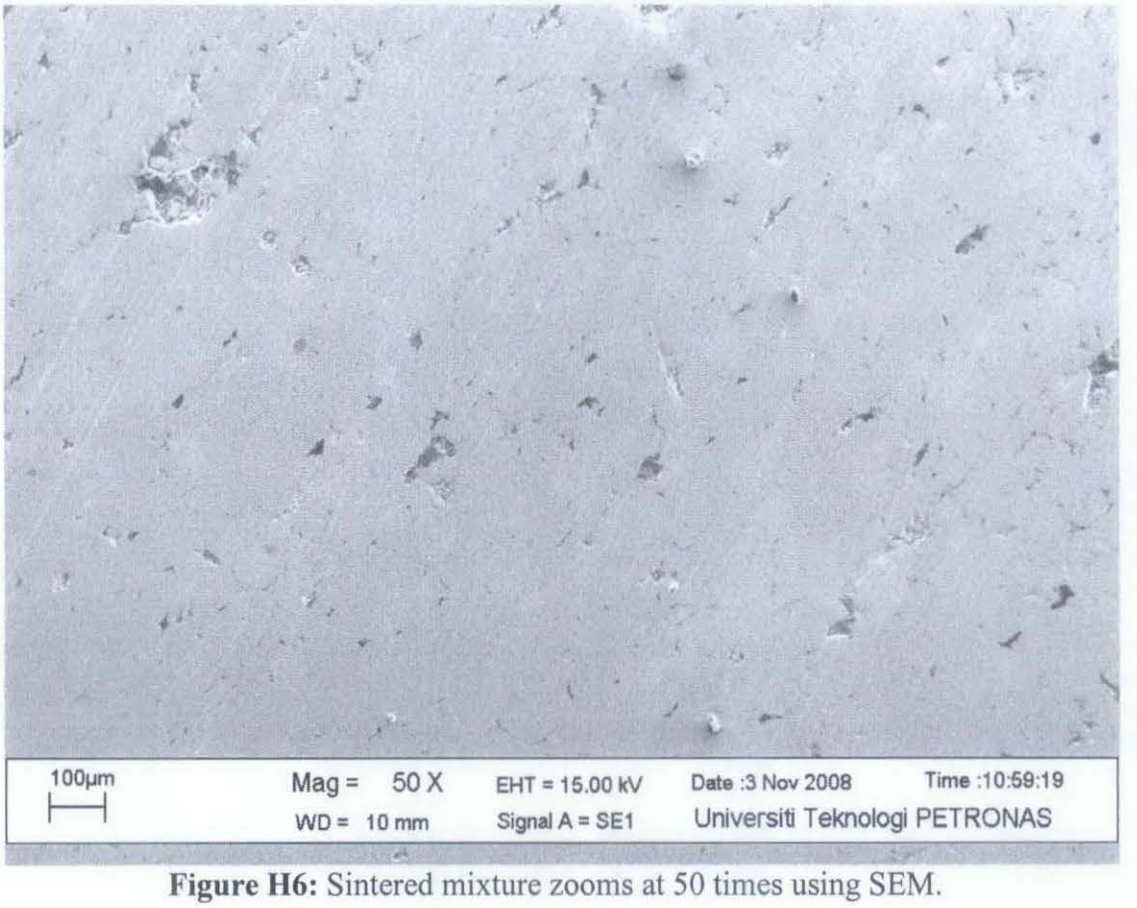

Figure H6: Sintered mixture zooms at 50 times using SEM.

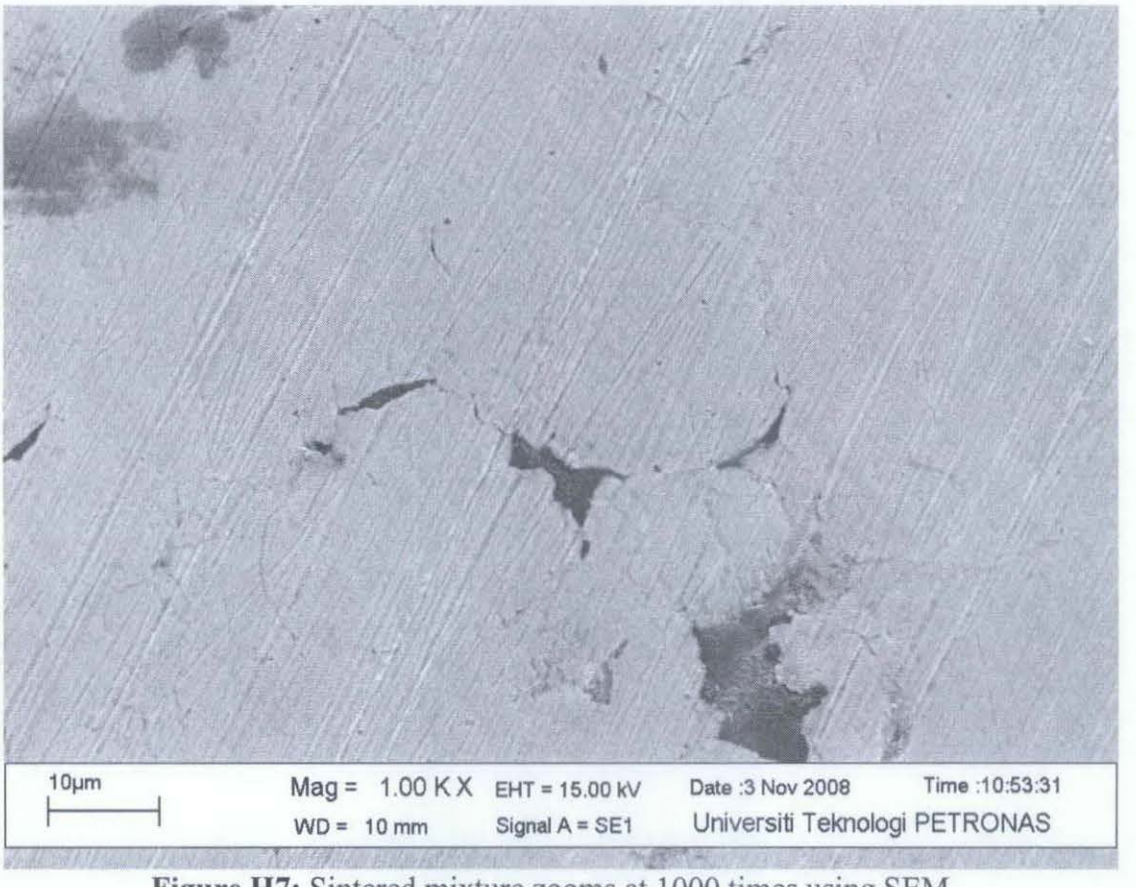

Figure H7: Sintered mixture zooms at 1000 times using SEM.

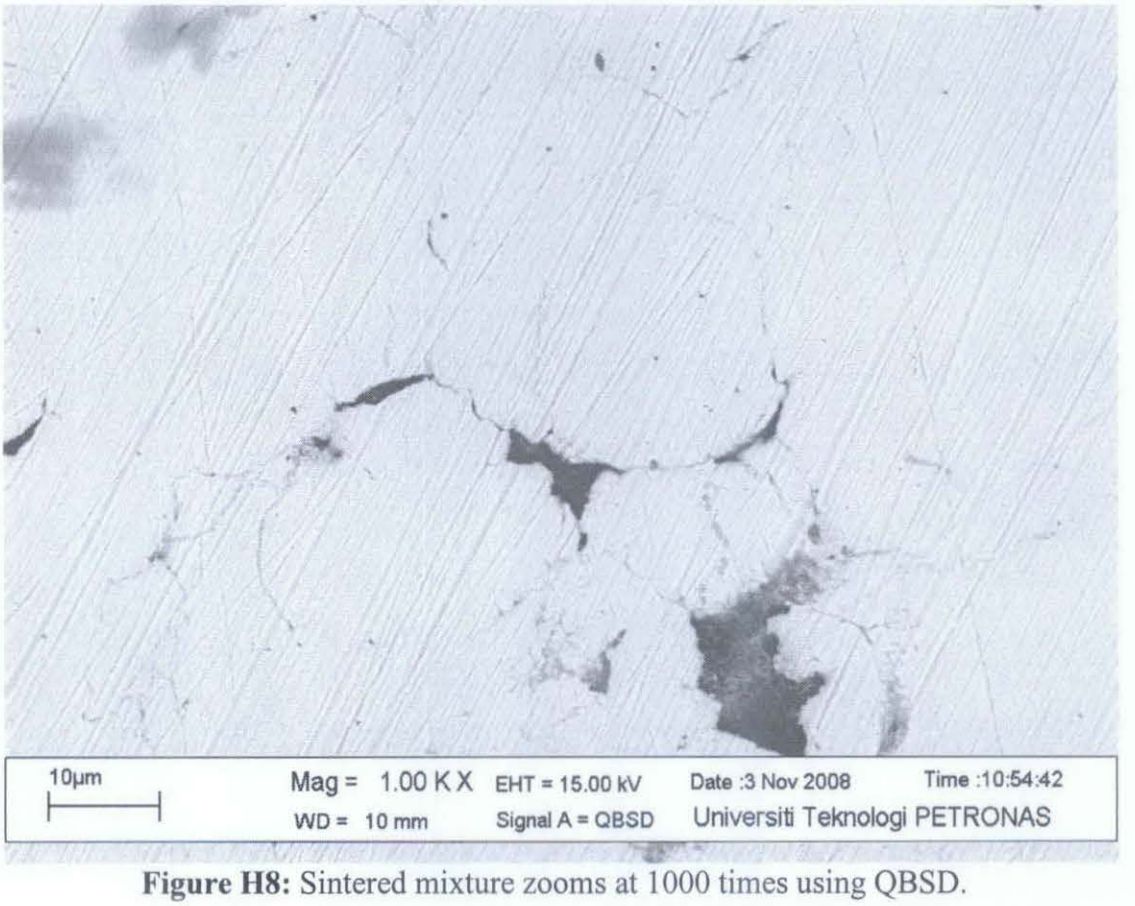

Figure H8: Sintered mixture zooms at 1000 times using QBSD.

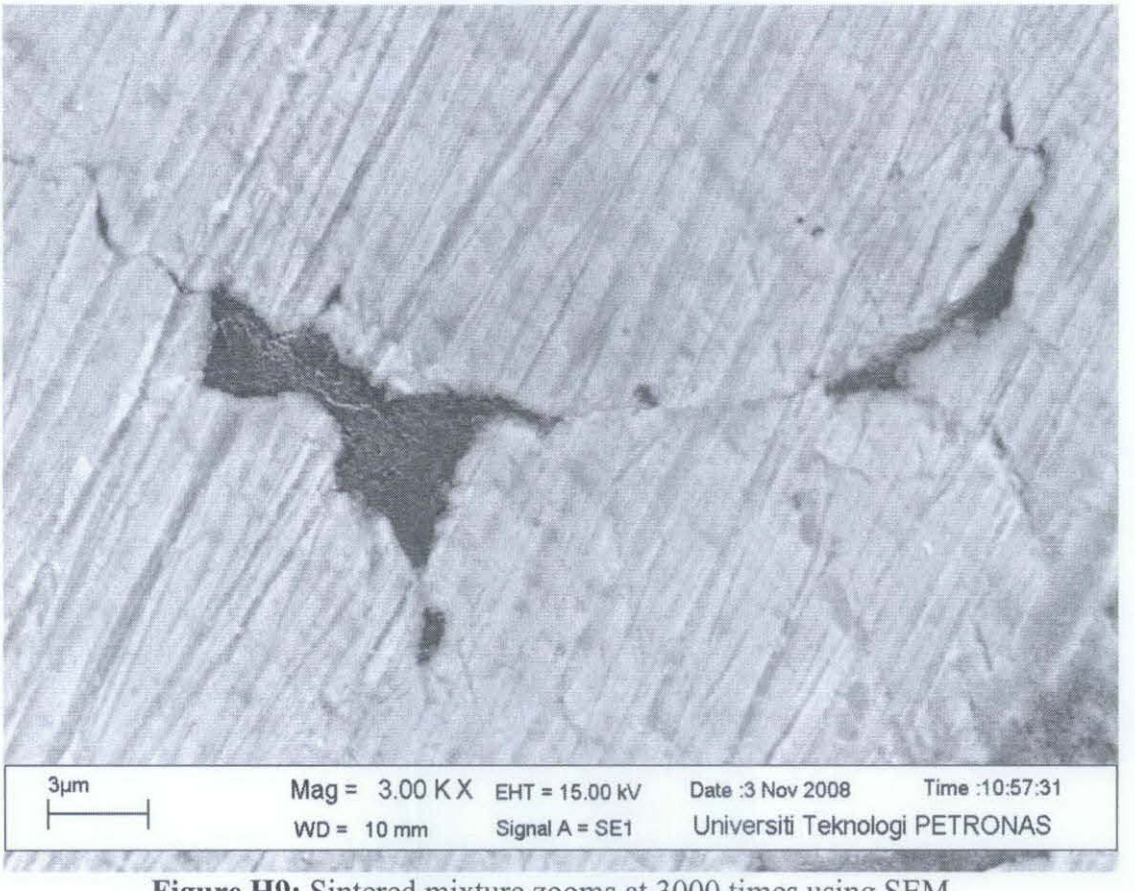

Figure H9: Sintered mixture zooms at 3000 times using SEM.