Microstructural Evolution and Physical Properties Changes during Liquid Phase Sintering of Fe-Cu-C Compacts

by

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Dissertation submitted in partial fulfilment of
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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
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BACHELOR OF ENGINEERING (Hons)
(MECHANICAL)

Approved by,

____
(Dr. Mazli Mustapha)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
January 2015

CERTIFICATION OF ORIGINALITY

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

MUHAMMAD ASROLDIN BIN MOHD ISHAK

ABSTRACT

Deeper understanding on the powder metallurgy method and the sintering process have granted in advanced development in the powder metallurgy industries. Fe-Cu-C is the leading materials in powder metallurgy method because of its ability for liquid phase sintering theoretically above melting point of copper; 1083 ˚C, thus strengthening the structure. The sintering temperature is important as it affect the diffusion of copper and carbon into iron matrix. Therefore the objective of this project was to study the microstructural evolution and the mechanical properties under different sintering temperature of mixed iron, copper, and carbon (Fe-Cu-C) compacts during liquid phase sintering. The effect of compaction pressure to the densification of Fe-Cu-C powder was also discussed in this study. Raw materials of pure iron, copper and carbon powder were mixed to the standard of Metal Powder Industry Federation (MPIF) of FX2008 (79 wt.% Fe, 20 wt.% Cu, and 1 wt.% C). The Fe-Cu-C powders were compacted into a pellets using hand press machine at different compaction pressures from 180 MPa to 600 MPa and sintered in a 5% H2 + 95% N2 gas atmosphere at different sintering temperature in the range of 1000ºC to 1200ºC. This study showed that the optimum sintering temperature that result in highest density of 6.76 g/cm^3 and highest hardness value of 200 HV was at 1200 ˚C. The compact pressure was at 600 MPa to minimise the porosity of the sample.

Keywords: *Microstructural, sintering, density, hardness*

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CHAPTER 1

INTRODUCTION

According to Murphy, et al (2004) Fe-Cu-C material has become the major material in Powder Metallurgy (PM) industries for many years. The reasons of the success of these compositions are the liquid phase from melting of the copper improves the sintering of the matrix iron and acceptable properties can be developed with less than optimal conditions. Greater stability and consistency in production of parts through the improvement of a more homogeneous microstructure can be achieved by increasing the sintering times. Additionally, from material properties standpoint, the ferrite was strengthened by the alloyed copper while the carbon diffuses and forms pearlite.

1.1 Background of the Project

The most widely practiced consolidations process in Powder Metallurgy (PM) is press and sintered method. In this process, loose powder is compacted in a die cavity shaped like the desired component, to produce green part which is then heated in a furnace to sinter the particle together. The powder metallurgy process is neither energy nor labor intensive, it conserves material, it is ecological clean and it produces net-shape components with homogeneous and reproducible properties. Currently, premixed iron-copper-carbon powders are increasingly being used for producing a variety of near-net-shape carbon steel components by powder metallurgy method. These components are pressed from iron-copper-carbon powders and sintered in the controlled atmosphere furnace. Copper is added to iron in order to increase the mechanical properties through solution hardening. Carbon has been found to be an excellent additive to iron-copper, since it reduces swelling and increases the strength of the alloy.

1.2 Problem Statements

The usage of powder metallurgy method in manufacturing industries have increase recently. Fe-Cu-C is the leading materials in powder metallurgy method because of its ability for liquid phase sintering theoretically above melting point of copper; 1083 ˚C, thus strengthening the structure. However, powder metallurgy sintered parts usually have residual porosity which influences the mechanical strength and other properties. The porosity can minimize by applying optimum sintering temperature. The sintering temperature is important as it affect the diffusion of copper and carbon into iron matrix. Optimum sintering temperature is needed to produce the highest mechanical properties of Fe-Cu-C. Furthermore, the resulting microstructure and properties of Fe-Cu-C compacts were found to be dependent on the thermal history during fabrication process. As the temperature increases, the microstructure of Fe-Cu-C also changes. Therefore, the temperature during sintering of Fe-Cu-C is important to produce best result from the sintering.

1.3 Objectives

The objectives of this project is to study the effects of different sintering temperature on microstructural evolution and the mechanical properties of mixed iron, copper, and carbon (Fe-Cu-C) powders according to MPIF standard of FX-2008 composition during liquid phase sintering. This project also study the effect of compaction pressure to the densification of Fe-Cu-C powder.

1.4 Scope of Study

This work will present the investigation carried out to study the sintering characteristics of Fe-Cu-C compact. In the experiment, the readily available iron, copper and carbon powder are mixed to the standard of Metal Powder Industry Federation (MPIF) of FX2008 which is 79 wt.% Fe, 20 wt.% Cu, and 1 wt.% C. The Fe-Cu-C powders will be compacted into a pellets using hand press machine at different compaction pressures and will be then sintered in a 5% H_2 + 95% N_2 gas atmosphere at different sintering temperature in the range of 1000°C to 1200°C. The relationship between the sintering temperature on sintered density and hardness will be presented and discussed. The effect of the compaction pressure on the density of the samples will also be discussed.

CHAPTER 2

LITERATURE REVIEW AND THEORY

2.1 Powder Metallurgy Method

"Diffusion of different metal powders as raw ingredients through process named sintering to create new materials and parts is a materials processing technology called Powder Metallurgy (PM).", (Tsutsui, 2012). According to, Dowson (1992), since the 1920s, PM has been used to produce a varied range of basic PM components, self-lubricating bearings and cutting tools. Tsutsui (2012) showed the diagram on basic process on how the product was created by using PM as in *Figure 2.1*:

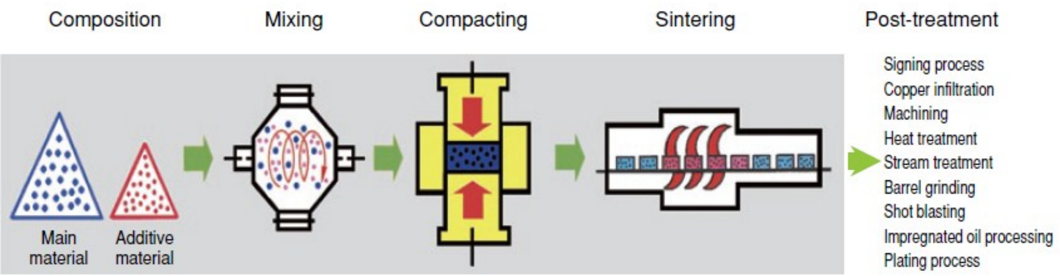

FIGURE 2.1. Fundamental process of powder metallurgy. (Tsutsui, 2012)

Dowson (1992) points that the PM process involves the processes as below:

1) Powder compression; generally in a container, to produce a compacted solid with enough cohesion to allow it to be handled securely.

2) Compact heating; typically in a controlled atmosphere, heated to below the melting temperature of the main component of the compact until individual particles stick to each other and have enough strength based on the intended material use for. This heating step also referred to as sintering process.

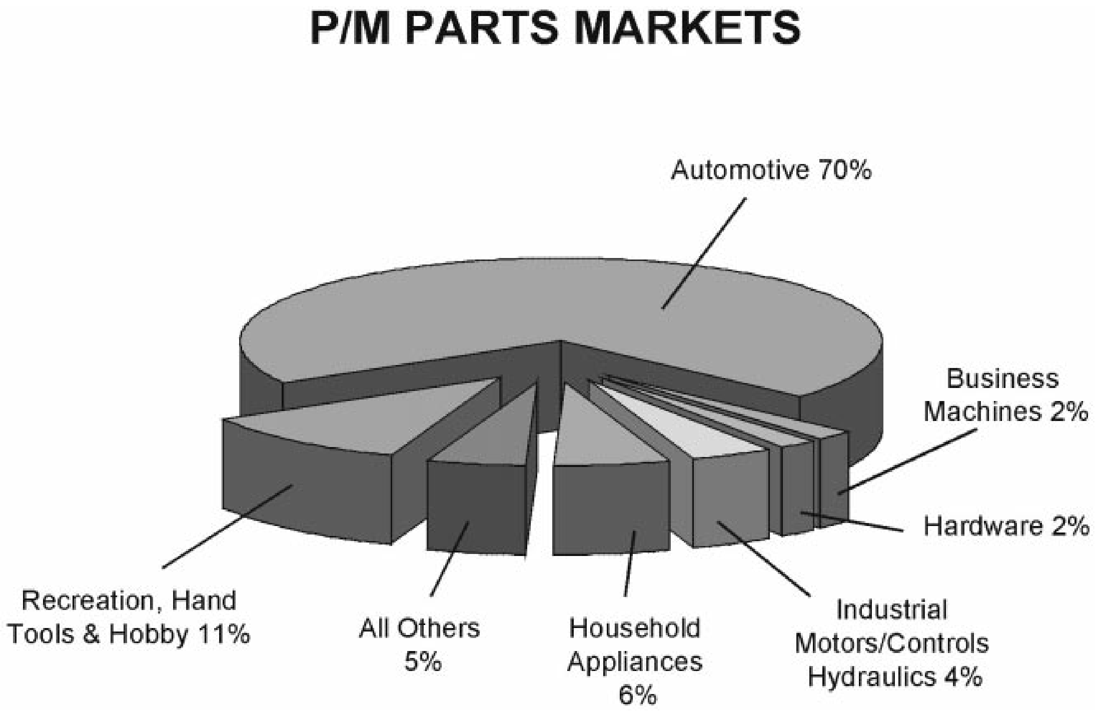

FIGURE 2.2. Powder Metallurgy Parts Market (Narasimhan, 2001)

Figure 2.2 shows the breakdown of the market of the parts produced by powder metallurgy method. The most application is on automotive parts which makes 70% of the applications.

2.2 Iron-Copper-Carbon Alloy

According to Marucci and Hanejko (2010) in their article entitled "Effect of copper alloy addition method on the dimensional response of sintered Fe-Cu-C steels", the most common alloy system used in press and sinter powder metallurgy is Fe-Cu-C. This system has many advantages especially excellent mechanical properties. "Whenever hardness, higher strength, and wear resistance are necessary, the Fe–Cu–C system is broadly used in the PM industry", (Narasimhan, 2001). The addition of copper in the Fe-C system enhances the properties of the alloys. According to Šalak (2005), alloying with copper not just increase the strength of the alloys and also upgrading the machinability. Narasimhan & Semel (2007) point that the Fe–Cu–C system shows typical LPS where the copper melts at 1082˚C and the solid iron grains dissolve in the liquid copper at the sintering temperature of 1120˚C thus the iron lattice expands for copper accommodation. Copper liquid pierces through the grain boundaries causing the swelling of the component. According to (Lawrock & Davies, 1990), carbon additions into Fe-Cu system lessen the growth by preventing the copper penetrating through the boundary of iron.

TABLE 2.1. Application of Fe-Cu-C powder (Narasimhan & Semel, 2007)

Material System	**Application**
Fe	Magnetic clutch housings Lock-up clutch collar Magnetic applications
Fe-Cu-C	Powder forged connecting rod Crankshaft belt drive Camshaft pulley Valve seat inserts Clutch adjustment ring Converter turbine sleeve Oil pump gears
Fe-Cu-C (double pressed-double sintered)	Synchronizer hubs

The composition of Fe-Cu-C used in this project is according to the Metal Powder Industry Federation Standard (MPIF) of FX-2008 which further detailed in ASTM B 783-04: “Standard Specification for Materials for Ferrous Powder Metallurgy (P/M) Structural” as copper infiltrated iron and steel with weight percentages of 0.6 – 0.9 wt.% of carbon, 15 – 25 wt.% of copper and the balance is the iron. According to ASTM B 783-04, the mechanical of FX-2008 material are stated in *Table 2.2*.

TABLE 2.2. Properties of FX-2008 material

Properties	FX-2008
Chemical Composition	0.6 – 0.9 wt.% C, 15 – 25 wt.% Cu, Bal. Fe
UTS	550 MPa
Hardness	90 HRB / 185 HV

2.3 Compressibility of Fe-Cu-C System

Before sintering process take place, the powder must be form into the desired shape. Therefore, the powder need to be compacted first. The compaction of the Fe-Cu-C powders is depending on the compressibility of the powder. The term compressibility is the measure of the ability of powder to deform under the applied compression and also described as pressure-density relationships (Anand & Kulkarni, 2013). Anand & Kulkarni (2013) further explain that the increase in compression load results in the increase of the compaction density and after certain level of densification, pressure has no effect.

2.4 Lubricant

Compacting different metals with different properties sure has its effect towards the die itself. As a protective measure to protect the die during compaction of metal powders, lubricant is used at the wall of the die. In short, lubricant are necessary in producing PM parts to minimize the die wear and reduce the ejection forces during pressing (Jonnalagadda, 2012). Furthermore, there are also lubricant that play a role as the binder to temporarily bind the powders after compaction. It is important that the lubricant should wet the metal surface completely to hold the shape and low value of shear coefficient of friction to protect the die (Jonnalagadda, 2012). There are many kinds of lubricants available in the market. For this study, the die wall lubricant used is zinc stearate.

2.5 Liquid Phase Sintering (LPS) Method

German, et al. (2008) briefly explained that liquid phase sintering (LPS) is a process of producing components from powders that have high performance and multiple-phase. They further explained that LPS involves sintering in situations where solid grains coexist with a wetting liquid. Variety of LPS are applied to application of materials engineering. For example, the automotive industries engine “connecting rods” and “high-speed metal cutting inserts”.

According to German, et al. (2008), LPS is applied to mixtures and alloys that liquefy over a certain range of temperatures whereby usually the liquid dissolves the solid grains in them. Thus, a capillary force is that pulls the grains together is created from the liquid wetting the solid because of the solubility. Simultaneously, the densification of the compounds is assisted by the high temperature that softens the solid. Since liquid has high-diffusion rates, the sintering is faster and lower sintering temperature required. As the final sintered product is a compound with customized properties, LPS is the leading sintering process. *Figure 2.3* shows the changes in microstructure during LPS.

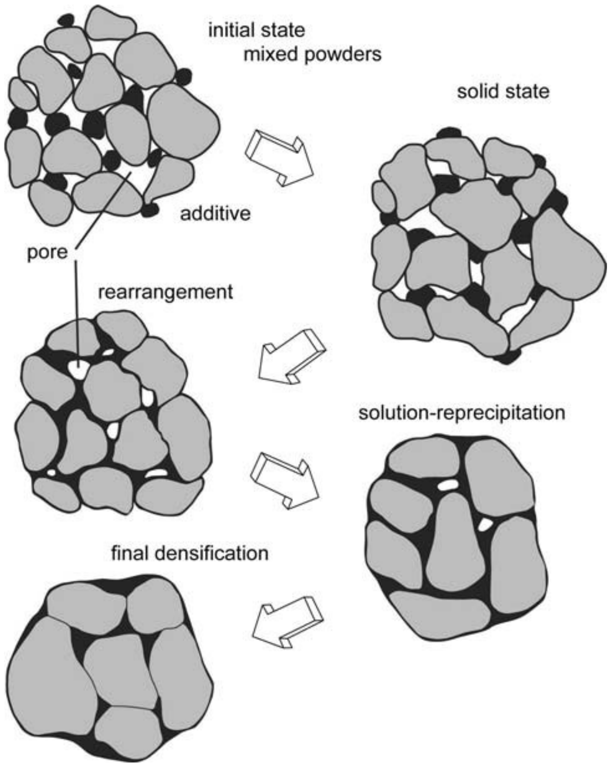

FIGURE 2.3. Microstructure changes during Liquid Phase Sintering (German, Suri, & Park, 2008)

2.6 Mechanical Testing Of Materials

Mechanical testing is required to determine the mechanical properties of the compacts. The example of mechanical properties that can be determine by mechanical testing are sintered density, hardness, tensile strength, impact and more. For this study, only focus on sintered density and hardness value of the compacts.

2.6.1 Sintered Density

Sintered density refers to the density of the sintered compact (Jonnalagadda, 2012). It should be less than the theoretical density because of evaporation of lubricant. Often, high sintered density is desirable except in the case of bearings and filters. Mechanical properties of the compact increase with its density.

2.6.2 Hardness

Hardness is defined as the resistance to wear and indentation (Jonnalagadda, 2012). It is measured by impressing an indenter test tip into the surface of the sample. Several hardness scales exist, Rockwell hardness with scales HRB, HRC and Vickers hardness with scale HV10 are popularly used. Rockwell hardness is a destructive test as the impression remains on the surface after testing. Vickers on the other hand is used for testing micro hardness and can be considered as a non-destructive test. The measurement is performed using a small indentation measured in a micro scope. For this study, because the sample is not viable for Rockwell hardness test, Vickers hardness has been used. High hardness value shows that the material have greater resistance to wear and abrasion. Hardness is directly proportional to the strength (Jonnalagadda, 2012).

CHAPTER 3

METHODOLOGY/PROJECT WORK

This study was done using three main raw materials, which were pure iron (Fe) powder, pure copper (Cu) powder and carbon (C) powder. All powders were mechanically mixed in a Turbular mixer. The weight percentage of the powders were fixed at 79 wt. % Fe, 20 wt. % Cu, and 1 wt. % C following the Metal Powder Industry Federation (MPIF) standards of FX2008. The mixed powders were compacted at 180 MPa, 360 MPa, 440 MPa, 520 MPa, and 600 MPa using 12mm diameter die for 6 different batches. The compacts then were sintered at temperature of 1000 ºC, 1040 ºC, 1080 ºC, 1120 ºC, 1160 ºC, and 1200 ºC per batch. The physical and mechanical properties and the microstructural observation will be further investigated. Below are the phases of the laboratory work conducted in the study and the details framework for this research shows in *Figure 3.1*.

PHASE I	: Raw material and preparation of alloy
PHASE II	: Physical properties measurement
PHASE III	: Microstructural observation
PHASE IV	: Mechanical testing (hardness test)

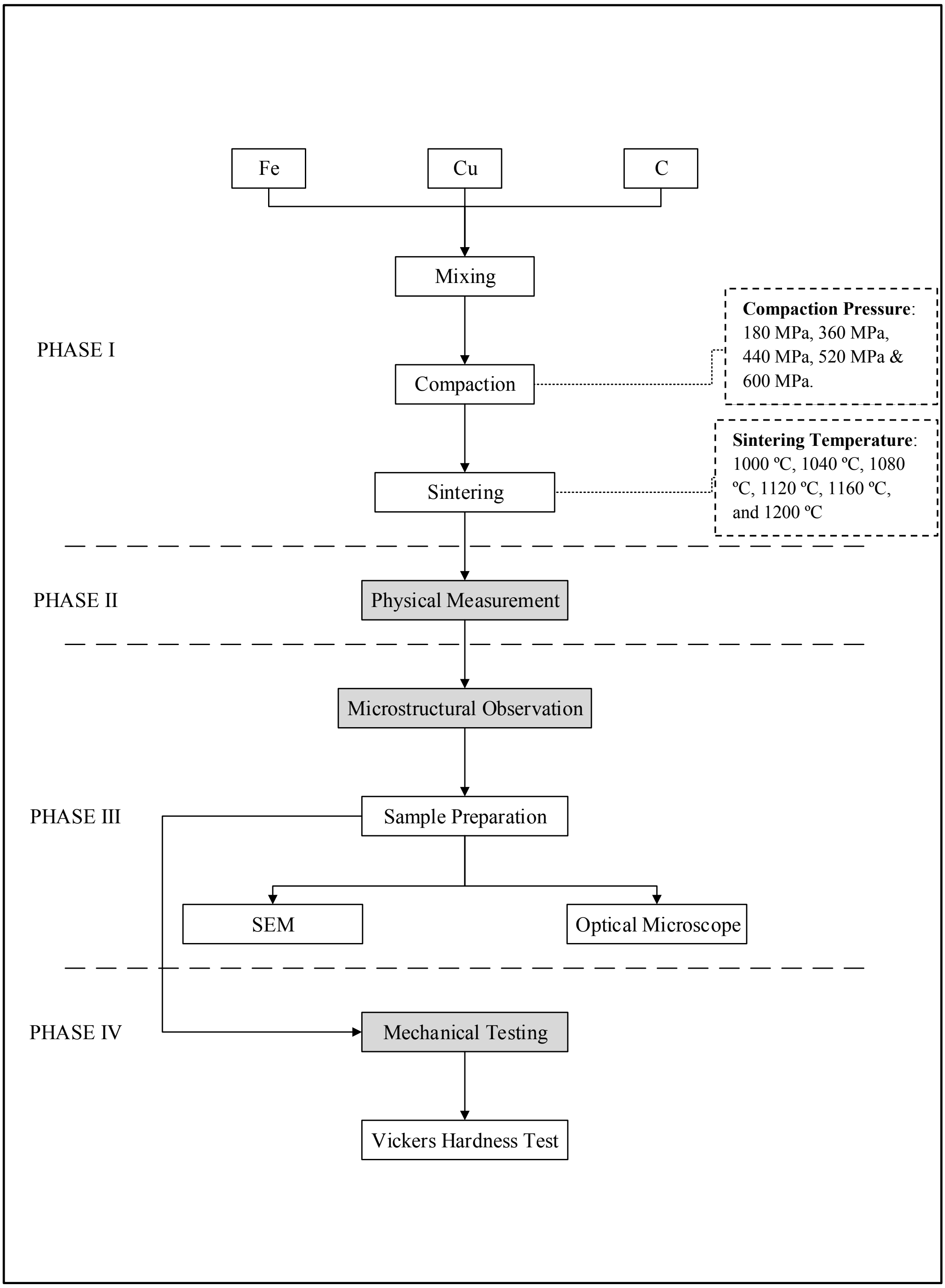

FIGURE 3.1. Research Framework of liquid phase sintering of Fe-Cu-C alloy

3.1 Raw Materials

3.1.1 Iron Powder

The pure iron powder that was readily available in the material laboratory were supplied from Merck KGaA, Darmstadt, Germany. The average size of the particle is 10 µm. Iron is a commonly used metal in manufacturing industries because of its high mechanical properties and resource availability. The alloying ability of iron with carbon to produce greater mechanical properties have made iron as the favorable metal for most manufacturing industries. Iron can be form to steel and cast iron depending on the weight percentage of carbon. In this study, iron was the based material with weight percentage of 79 wt. %. The color of the iron powder was grey and irregular spherical in shape. The iron powder melts at 1535 ˚C. *Table 3.1* shows the mechanical properties of iron powder.

TABLE 3.1. Properties of pure iron powder

Properties	Description
Formula	Fe
Molar mass	55.85 g/mol
Form	Solid Powder
Colour	Grey
Melting point	1535 ˚C
Relative Density	7.87 g/cm^3 @ 20 ˚C
Particle size	< 10 µm
Bulk density	3000 - 4000 kg/m^3
Hardness (Vickers)	608 MPa / 62 HV

3.1.2 Copper Powder

The pure copper powder that was readily available in the material laboratory were supplied from Merck KGaA, Darmstadt, Germany. The average size of the particle is 63 μm. Copper is added to iron in order to increase the mechanical properties through solution hardening. In this study, the weight percentage of copper used was 20 wt. %. The color of the copper powder was brown and flake in shape. The iron powder melts at 1083 ˚C. *Table 3.2* shows the mechanical properties of copper powder.

TABLE 3.2. Properties of pure copper powder

Properties	Description
Formula	Cu
Molar mass	63.55 g/mol
Form	Solid Powder
Colour	Brown
Melting point	1083 ˚C
Relative Density	8.96 g/cm^3 @ 20 ˚C
Particle size	< 63 μm
Bulk density	1290 kg/m^3
Hardness (Vickers)	343-369 MPa / 35-38 HV

3.1.3 Carbon

The carbon powder that was readily available in the material laboratory were supplied from Merck KGaA, Darmstadt, Germany. The average size of the particle was not specified. Carbon has been found to be an excellent additive to iron-copper, since it reduces swelling and increases the strength of the alloy. In this study, the weight percentage of carbon used was only 1 wt. % because the desired product was steel alloy. Iron alloy will become cast iron if the weight percentage of carbon is more than 2 wt. %. The color of the carbon powder was black and irregular in shape. *Table 3.3* shows the mechanical properties of copper powder.

TABLE 3.3. Properties of pure carbon powder

Properties	Description
Formula	C
Molar mass	12.01 g/mol
Form	Solid Powder
Colour	Black
Melting point	N/A
Relative Density	2.2 g/cm^3 @ 20 ˚C
Particle size	N/A
Bulk density	ca. 280 kg/m^3
Hardness (Vickers)	343-369 MPa / 35-38 HV

3.1.4 Zinc Stearate

The preparation of samples for powder metallurgy required a substance called lubricant. A lubricant can be either mixed together with the metal powder or applied on the surface of the die wall. Lubricant also function as binder to hold the shape of the metal powder after compaction. For this study, the lubricant used was zinc stearate. The lubricant was applied at the surface of the die wall before inserting the mixed metal powders. The lubricant will be compacted along with the powders and wet the surface part of the compacted powders to hold the shape. After sintering, the lubricant will be burnt out.

3.2 Raw Materials Processing

3.2.1 SEM Analysis

The nature of the powder size and distribution were further study using Phenom ProX Scanning Electron Microscope (SEM) to get a view of the structure and shape of the powders. The powders were pasted onto a carbon tape and then put on a holder. Then the holder was inserted into the SEM. The photomicrograph of the powder distribution at magnification of 1500x were taken for each powders.

3.2.2 Weighing and Mixing

Weighing of individual raw material powders has been carried out using Metler Toledo precision balance. The powders were weighed at 79g for iron, 20g for copper and 1g for carbon which total up to 100g with weight percentage of 79 wt. % Fe, 20 wt. % Cu and 1 wt. % C. Then all of the weighed powders were mechanically mixed into a container, and homogenously mixed using a tubular mixer for an hour. The tubular mixer will shake the container containing the mixed powders at different angles which made up to a 3 dimensional shake. This will homogenously and uniformly mix the powders. The process of mixing is important because the powders need to be evenly distributed so that the concentration of each powders will be uniform throughout the mixture. After mixing process done, the homogenously mixed powders will be weighed again to 30 different samples with each sample weight of 3 - 3.2 g.

3.2.3 Compaction of Mixed Powders

The forming of the samples using powder metallurgy method begins with the densification of the loose powder into green compact in a cavity rigid die. A high carbon steel die was used to make the cylindrical shape of the green compact. The diameter of the die used was 12mm. A single action compacting technique was used for uniaxial compression of the powders in the die. The technique involved the application of pressure only through the upper part of the punch, while the lower part and the die are fixed to the bottom support as shown in *Figure 5*. The wall of the die and the punch surface were coated with zinc stearate first before inserting the powders. This is to facilitate the compaction and ejection of the die punch, protecting the surface of the die from scratch and damage and also act as binder to hold the shape of the green compacts. The pressures applied for the compaction will be different. Since the press machine used only tolerate in kN unit, the load will be then calculated using *Equation 3.1 and 3.2*.

$$Load\ (N) = Pressrue\ (MPa)\ \times\ Area\ of\ die\ (mm^2) \tag{3.1}$$

$$Area\ of\ die\ punch\ (mm^2) = \frac{\pi\ \times ((12\ mm)^2)}{4} = 113.1\ mm^2 \tag{3.2}$$

Enerpac Hydraulic Hand Press was used to compact the powders. The weighed powders of approximately 3 – 3.2 g will be poured into the die and the powder will be compacted for 1 minute to ensure adequate pressure applied. After compacting, the lower punch was removed and a U-shaped steel frame was inserted under the mold to assist the ejection process. Load is then applied to the upper punch until the sample was ejected. *Figure 3.2* shows the setup of the uniaxial die. The procedures were repeated at pressure of 180 MPa, 360 MPa, 440 MPa, 520 MPa and 600 MPa which converted to load of 20.4 kN, 40.7 kN, 49.8 kN, 58.8 kN, and 67.9 kN for 6 different batches (Calculation refer to *Appendix 5*).

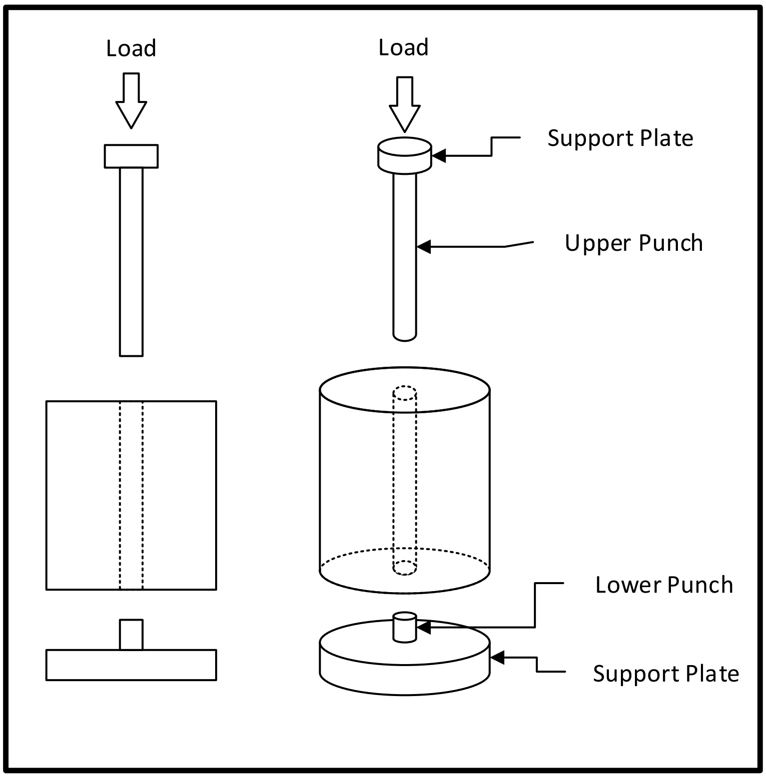

FIGURE 3.2. Schematic diagram of the uniaxial die pressing of powder

3.2.4 Sintering

Samples were sintered using a tube furnace (Carbolite) with maximum temperature of 1200 ˚C with gas flow ability. A ceramic crucible was used to support the samples while in sintering process. The samples were arranged appropriately on the ceramic crucible and placed at the center of the furnace to ensure the uniform heating of the samples. The furnace was closed and air is purged out by purging 95% N_2 – 5% H_2 gases into the heating chamber. The furnace temperature was ramped up at heating rate of 10 ˚C/min until 800 ˚C. Then the samples were hold for about 10 minutes to familiarize the temperature to the samples. Then the temperature was ramped up again at the same heating rate until the sintering temperature. The sintering temperatures were at 1000 ˚C, 1040˚C, 1080˚C, 1120˚C, 1160˚C, and 1200 ˚C respectively for each batch of samples (See Appendix 6). Each samples were hold for about 60 minutes at the sintering temperature to allow the flow and diffusion of the carbon and copper into iron matrix. After that, the samples were slowly cooled to room temperature by switching off the heating while maintaining the same atmosphere. This is important to make sure there were no air while in the process of cooling. *Figure 3.3* shows the heating curve for each sintering temperature.

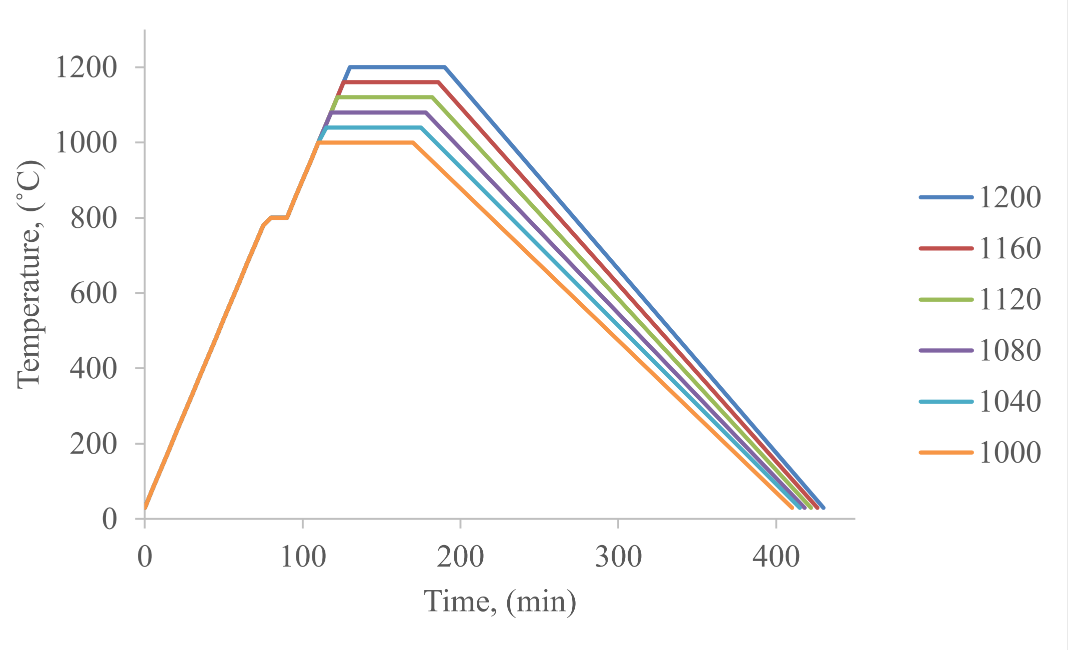

FIGURE 3.3. Heating curvers of sintering of the samples

3.3 Physical Properties Measurements

After the sintering process, the samples were physically measured for the physical properties such as the linear dimension (sintered diameter) and sintered density.

3.3.1 Linear Dimension

From the literature review, the higher the compaction pressure, the higher the densification of the compacts. Any pressure applied above the optimum compaction pressure will not affect the densification because the powders are already compacted at maximum possibility. Therefore, to study the linear dimensional changes of the samples, the samples compacted at the highest pressure (600 MPa) were chosen. The diameter of the samples were measured using Vernier caliper for several attempts at different points to get the average of the diameter. Then, the data were compared to the original diameter of the die and the percentage of dimensional changes were recorded.

3.3.2 Sintered Density

The sintered density of all samples were measured according Archimedes' water immersion principle via ASTM B 962-13 Standard: "Standard Test Methods for Density of Compacted or Sintered Powder Metallurgy (PM) Products Using Archimedes' Principle". The first step was to measure the mass of the sample in air (m_a). Then the samples were submerged in distilled water for about 30 minutes to allow water to enter the pores in the samples. The mass of the samples when immersed in water were recorded as m_i. The samples then were taken out and dried using absorbent paper. After that, the mass of the samples were measured again in the air (m_o). The sintered density of the samples were calculated using *Equation 2* where ρ_w was the density of water at room temperature (0.9997 g/cm^3).

$$Sintered\ Density\ \left(\frac{g}{cm^3}\right) = \frac{m_a}{m_o - m_i} \times \rho_w \tag{3.3}$$

3.4 Microstructure Observation

3.4.1 Sample Preparation

Sample preparations must be done before examining the microstructure of the sample. The main steps for the preparations are:

1. Mounting
2. Grinding
3. Polishing
4. Etching

3.4.1.1 Mounting

The samples were hot mounted using Automatic Mounting Press (SIMPLIMET 1000) to hold the samples for surface grinding. The mounting were made of phenolic resin with cylindrical shape (diameter: 30 mm) mold. The purpose of mounting is to get the flat surface of sample so that it would be easier for grinding process to take place.

Prior to mounting the samples, all oil, grease and debris were removed from the sample by washing it in acetone or ethanol, then drying it. The mounting press were inspected and cleaned before mounting process, especially cylinder and the upper and lower rams. Then the ram was raised to the top of the cylinder. The inner surfaces, rams and threads of the mounting was coated with release agent.

After the samples were placed on the center of the ram, the ram was lowered until it reached the lowest depth. The molding compound (phenolic powder) was added with an adequate amount. The cylinder was closed by inserting the upper ram and the fixture is tightened. The temperature, pressure, time were set 150 ˚C, 4000 psi and 15 minutes respectively.

3.4.1.2 Grinding

Once the sample has been mounted, the surface of the samples must be grinded to produce flat surface. Grinding process was performed by using Grinder and Polisher machine model Metaserv 2000.

The surface of the samples were mechanically grinded manually on the grinding machine using SiC paper with running water. The direction of grinding was changed 90° after each SiC paper. SiC paper used ranging from the coarsest grit paper, 200 grits to 1200 grits to eliminate the scratches from the previous grinding stage otherwise they will not be removed in polishing.

First 200 grit size was used. Samples were placed face down of abrasive surface, and being slide in a forward and backward motion. The water tap was opened and applied during the grinding process in order to act as a cooling agent. Samples is turned to 90 degrees and repeat above procedure on the 320, 400, 800 and 1200 grits.

3.4.1.3 Polishing

Polishing was used to create a flat, defect-free surface for examination of a microstructure under a microscope. Grinder and Polisher machine model Metaserv 2000 was consist of 2 rotating discs covered with soft cloth impregnated with diamond particles (1 micron and 6 micron size) and an oily lubricant. MetaDI fluid is applied on the polishing pad. 6 micron diamond paste is applied on the samples. The samples with diamond paste were being polished on the rotating polishing pad. The procedure was repeated using 1 micron diamond paste.

3.4.1.4 Etching

The 3% Nital etching solution for the samples was prepared by mixing 100 ml of ethanol and 10 ml of Nitric Acid. The etchant preparation and other activities were done on the table under the Fume Hood with the polished surface up and gloves were wore. The surface was cleaned with alcohol without touching the samples surface. The samples were dried by using dryer machine. The etchant was placed in a beaker. The etchant was applied on the polished surface by swab using cotton bud and about 30 seconds to 60 seconds. The samples were dried by using dryer machine without touching the samples surface.

3.4.2 Microstructure Observation before Etching

Before etching process, the polished samples were observed under optical microscope with magnification of 500x. This to obtain the photomicrograph of the unetched condition of the samples. Photomicrograph of the samples surface before etching were taken and recorded for analysis.

3.4.3 Microstructure Observation after Etching

After etching process, the etched samples were observed under optical microscope with magnification of 500x. Photomicrographs of the samples surface after etching were taken and recorded for analysis.

3.5 Mechanical Testing

3.5.1 Vickers Micro Hardness

Vickers hardness testing for sintered parts carried out using Vickers Micro Hardness Tester LM 247AT (LECO). The surface being tested generally required a grinding and polishing as to easily identifying the inherent porosity of samples and thus, can it can be avoided while placing the diamond indenter. A test load of 5 N or 0.5 kgf in accordance with the ASTM E384-11-e1 Standard; “Standard Test Method for Knoop and Vickers Hardness of Materials”. After the force has reached a static equilibrium condition, the force dwell for 15 seconds and further penetration ceases. Precision microscope was used to measure the resulting surface area of the measured square-based pyramidal produced by diamond indenter. For each samples, at least 5 indentations were carried out and the mean value were obtained and recorded as the micro hardness of the samples.

3.6 Final Year Project Flow Process

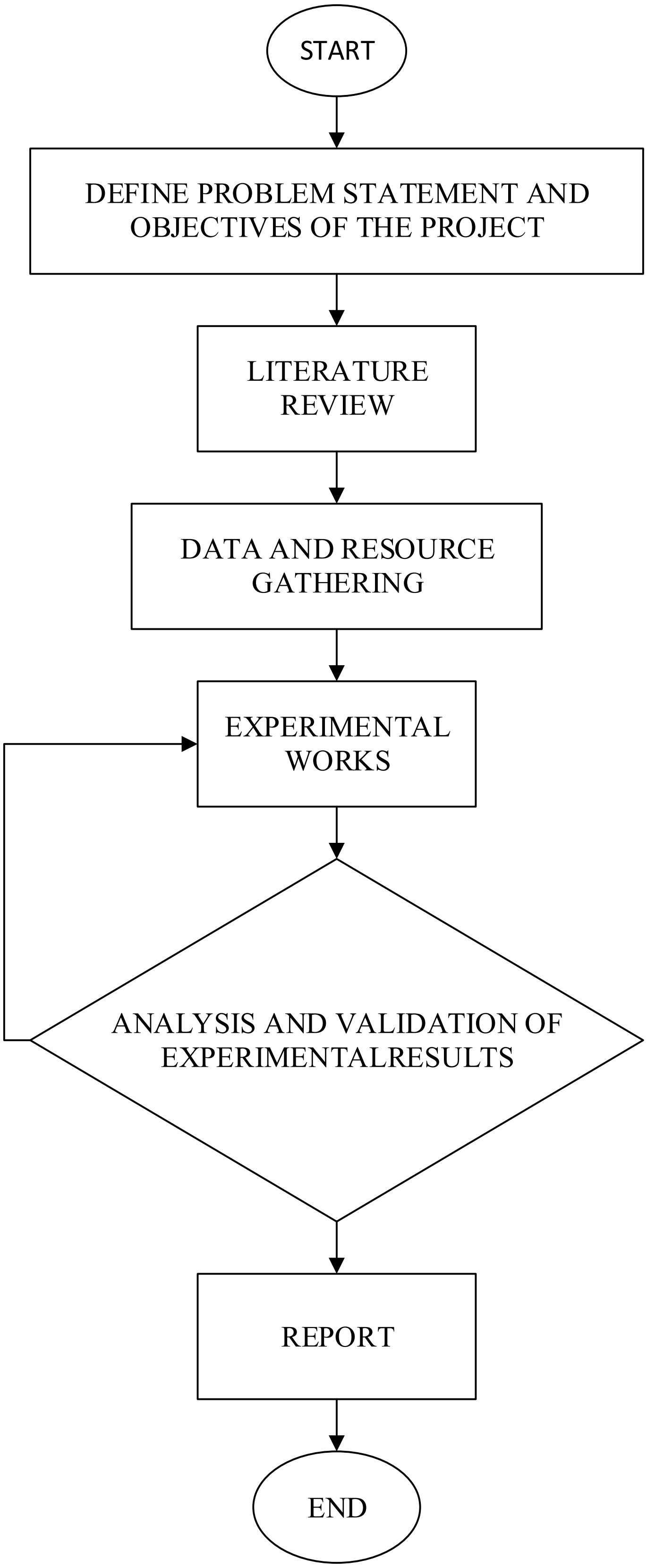

FIGURE 3.4. Project Flow Chart

3.7 Gantt Chart & Project Activities

TABLE 3.4. Overall Final Year Project Gantt chart

Project Activities	Weeks																											
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	1	2	3	4	5	6	7	8	9	10	11	12	13	14
	Final Year Project I														Final Year Project II													
1. Project Selection	🟩																											
2. Literature Review	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩	🟩										
3. Extended Proposal Submission				🟩	🟩	🟩																						
4. Proposal Defense							🟩																					
5. Approval for Usage of Resources				🟩	🟩	🟩	🟩	🟩	🟩																			
6. Raw Materails Analysis																												
- Raw Materials Acquisition								🟩	🟩																			
- SEM Analysis of Raw Materials										🟩	🟩																	
7. Interim Report Submission												🟩	🟩	🟩														
8. Sample Preparation																												
- Weighing of Raw Materials															🟩													
- Mixing of Raw Materials															🟩													
- Compacting Mixed Materials																🟩	🟩											
9. Progress Report Submission																				🟩	🟩							
10. Sintering of Compacted Samples																		🟩	🟩									
11. Physical Measurement on Sintered Samples																												
- Mass measurement																			🟩									
- Dimension measurement																			🟩									
- Density Measurement																			🟩									
12. Sample Preparation for Microstructure Analysis																												
- Mounting of Samples																				🟩								
- Grinding and Polishing of Samples Surface																					🟩	🟩						
- Samples Etching																					🟩	🟩						
13. Microstructure Observation of Samples																					🟩	🟩						
14. Hardness Measurement of Samples																					🟩	🟩						
15. Result Analysis																						🟩	🟩					
16. Pre-SEDEX Poster Presentation																							🟩	🟩				
17. Draft Final Report Submission																									🟩	🟩		
18. Dissertation (soft bound) Submission																										🟩	🟩	
19. Technical Paper Submission																											🟩	🟩
20. Viva Presentation																												🟧
21. Submission of Project Dissertation (Hard Bound)																												🟧

3.8 Key Milestones

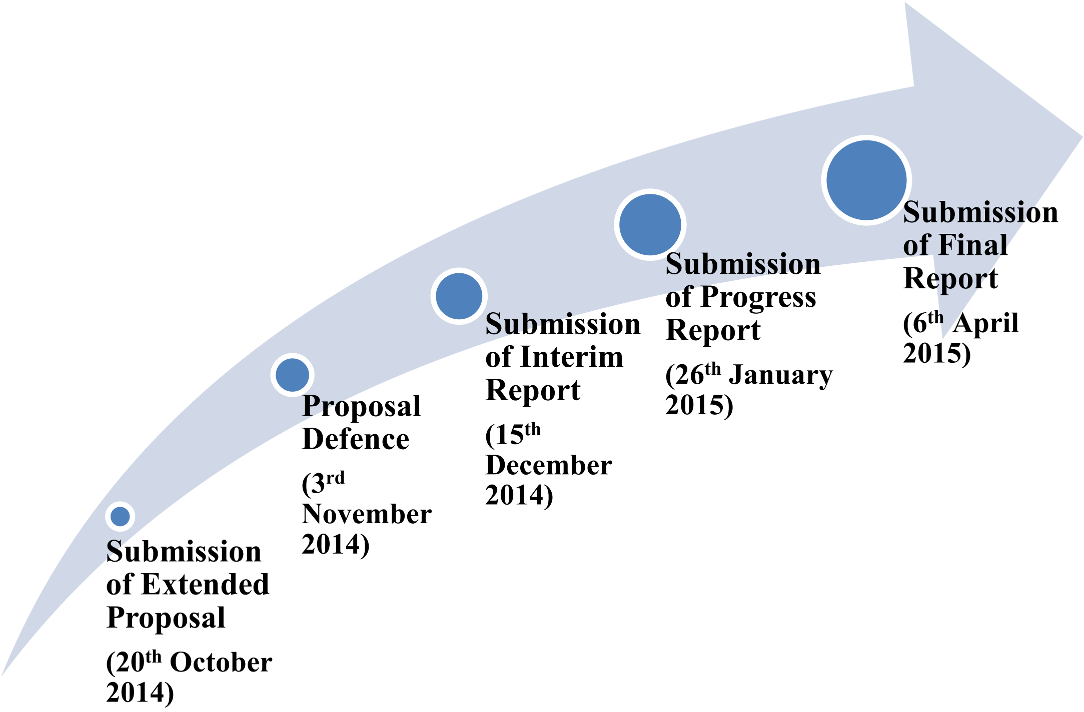

FIGURE 3.5. Project Key Milestones

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Powder Distribution

The readily available Fe, Cu and C powders were observed under a Scanning Electron Microscope (SEM) to get a view on shape and size of powder particles before mixing process is done. *Figures 4.1*, *4.2* and *4.3* show the microstructures of powders of pure iron, copper and carbon. Micrographs indicate the smooth and spherical shape of iron, flake shape of copper and the irregularly shaped carbon particles.

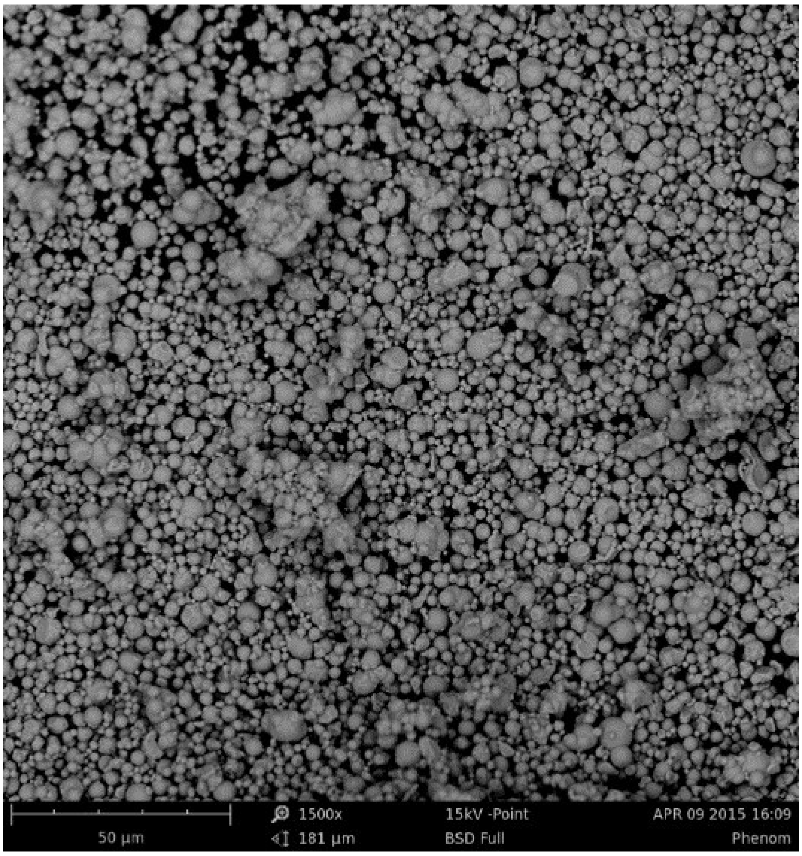

FIGURE 4.1. Powder distribution of Fe powder

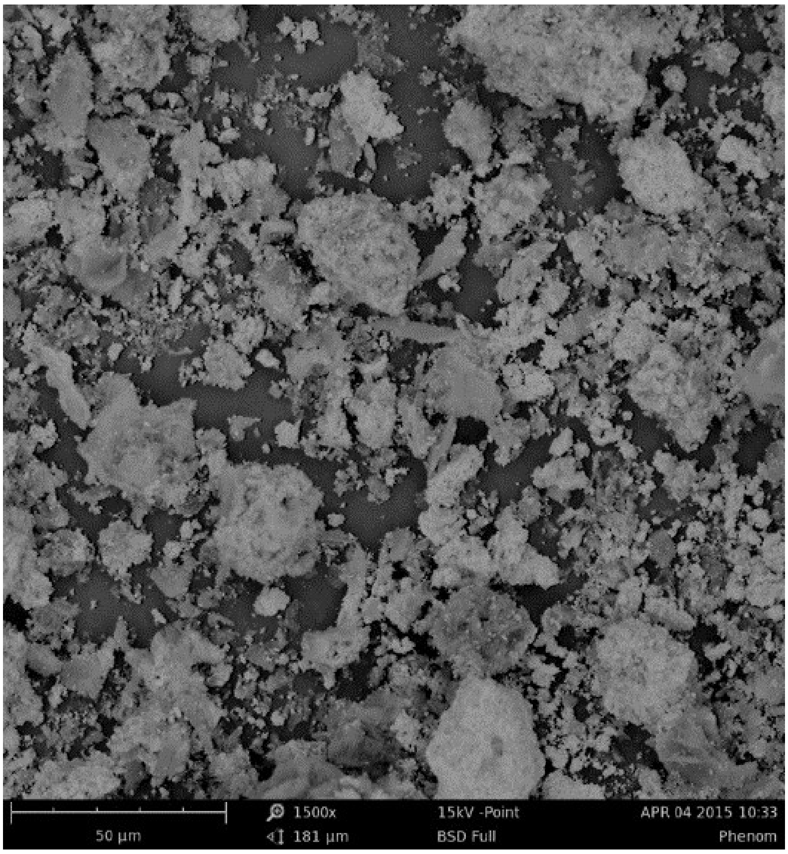

FIGURE 4.2. Powder distribution of Cu powder

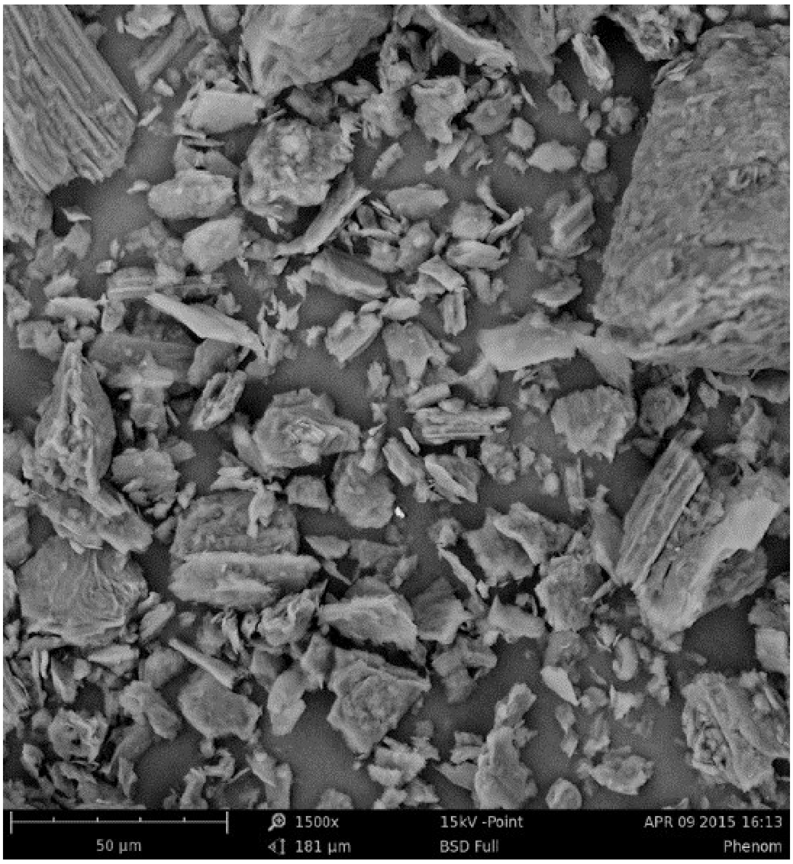

FIGURE 4.3. Powder distribution of C powder.

4.2 Dimensional Changes and Microstructure Evolution

The dimension of the samples may changes due to the reaction between the elements in the alloy during sintering process. For example, the diffusion of carbon into iron matrix to from cementite. Cementite in iron matrix (ferrite) is called pearlite. The samples sintered at 1200 ˚C and compacted at compaction pressure of 600MPa were measured for the linear dimensional changes. Average percentage of linear dimensional changes were recorded in *Table 4.1*.

TABLE 4.1. Dimensional changes of the samples sintered at 1200 ˚C and compacted at 600 MPa

Temperature /°C	Dimensional Change / %
1000	0.75
1040	0.80
1080	0.85
1120	0.70
1160	0.68
1200	0.65

From the result, the dimension of the samples were greater than the die size mostly due to growth. As the temperature increases from 1000 ˚C to 1040 ˚C, the sample linearly growth from 0.75 % and 0.80 % respectively. This growth is mainly due to the diffusion of carbon in the iron matrix. The presence of pearlitic structure, a lamellar or layered structure of two phases: α-ferrite and cementite (Fe_3C) in the cross-sectional photomicrograph of sample sintered at 1000 ˚C and 1040 ˚C respectively *Figure 4.4 (a)* and *4.4 (b)* confirmed the growth of the compacts. The photomicrograph showed presence of unmelted copper in the structure, further confirming that the growth is primarily due to the diffusion of carbon in the iron matrix. The presence of the unmelted copper in the compacts was due to the considerably lower sintering temperature than the melting temperature (1083 ˚C) of copper.

4.2.1 Etched condition photomicrograph of sintered Fe-Cu-C

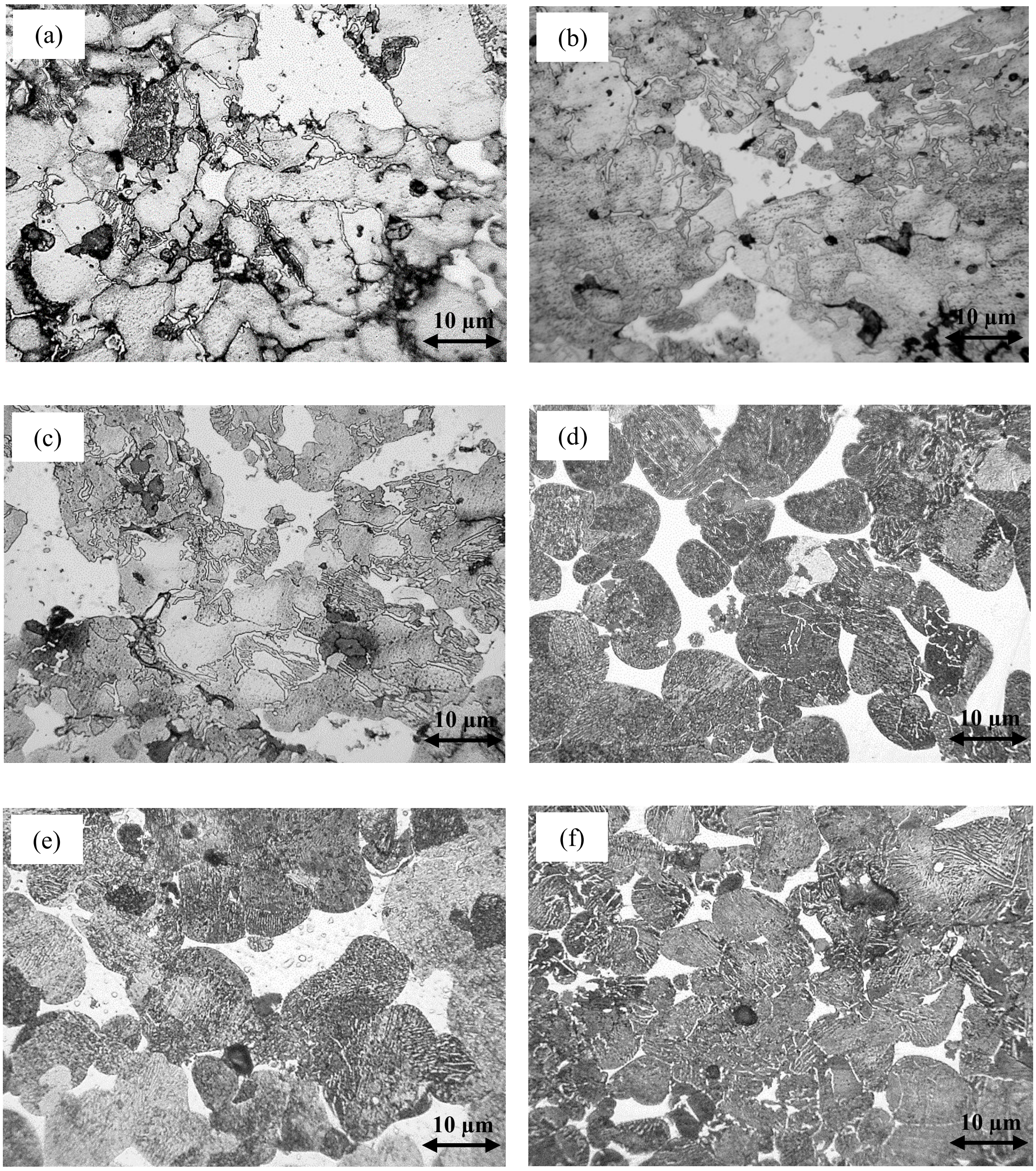

FIGURE 4.4. Photomicrographs of Fe-Cu-C compacts sintered at (a) 1000˚C (b) 1040 ˚C (c) 1080 ˚C (d) 1120 ˚C (e) 1160 ˚C (f) 1200 ˚C. Etched with 3% Nital.

A further increase in sintering temperature to 1080 ˚C caused an increase in linear growth to about 0.85 % of the die size. The increase in growth is due to the point that copper start to melt and dissolve in iron matrix. The cross-sectional photomicrograph of the compacts sintered at 1080 ˚C in *Figure 4.4 (c)* confirmed the presence of copper (white area) in the structure. The further increase in sintering temperature to 1200 ˚C caused the growth to decrease to 0.65% of the die size. This is due to near complete particle to particle bonding, as evidenced in *Figure 4.4 (f)*.

4.2.2 Unetched condition photomicrograph of sintered Fe-Cu-C

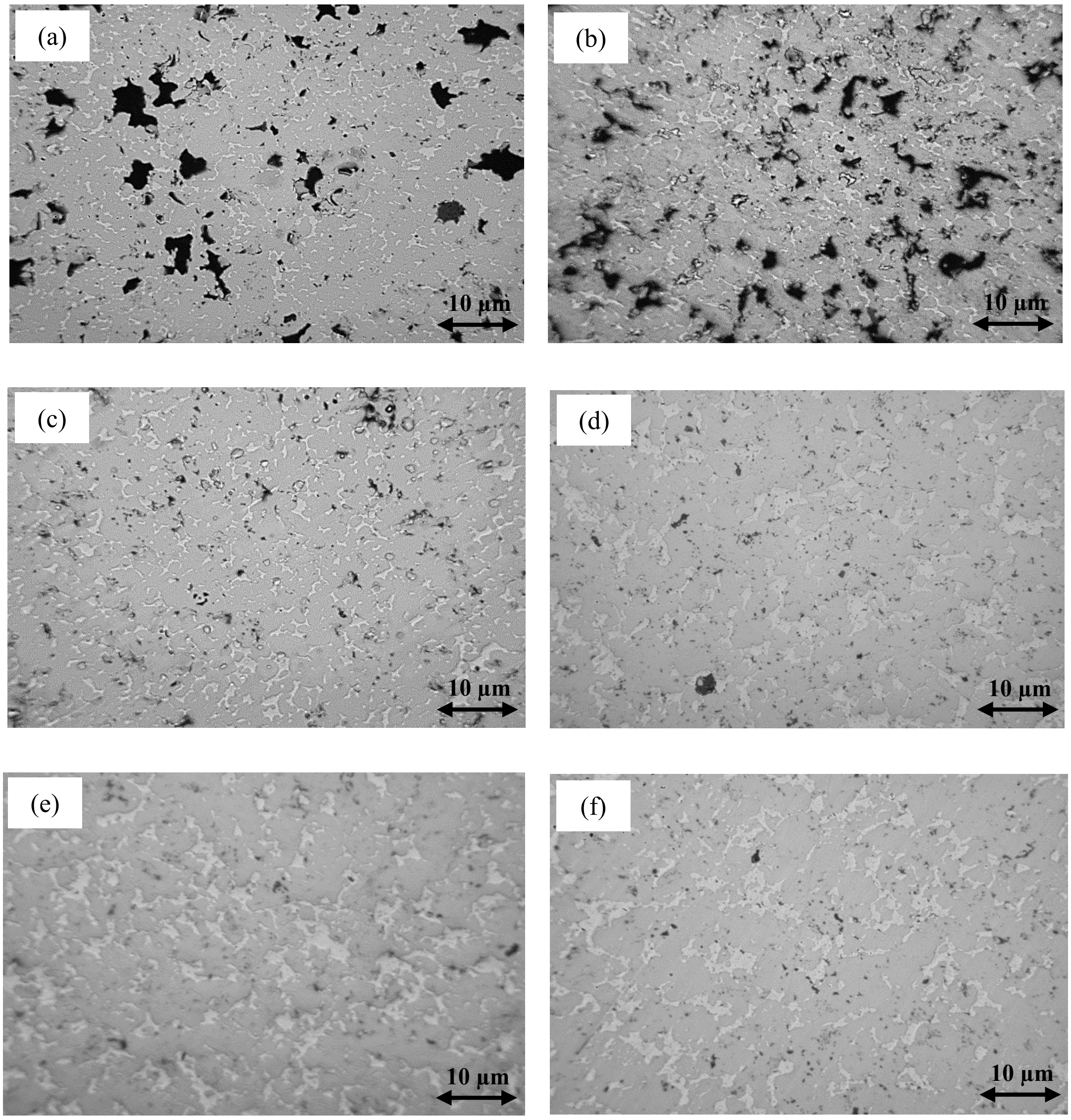

FIGURE 4.5. Photomicrographs of Fe-Cu-C compacts sintered at a) 1000˚C (b) 1040 ˚C (c) 1080 ˚C (d) 1120 ˚C (e) 1160 ˚C (f) 1200 ˚C. Unetched condition.

From *Figure 4.5 (a)* and *4.5 (b)*, the porosity of the samples can be clearly seen. During sintering temperature of 1000˚C and 1040 ˚C, copper inside the samples have not completely melt. Thus copper have not diffuse in the iron matrix to fill in the void causing the pores to be visible. Further increase in sintering temperature above the melting point of copper (1083 ˚C) cause copper inside the samples to melt and started to diffuse into iron matrix and fill the void and gap between the iron particles. This results in the decreased in the porosity as *in Figure 4.5 (c), 13 (d) 4.5 (e),* and *4.5 (f).*

4.3 Compaction Pressure Effect and Microstructure Overview

The data of the sintered density of different compaction pressure at various sintering temperatures is summarized in *Figure 4.6*. The increase in sintered density is due to the increase in the extent of sintering and diffusion of carbon in the iron matrix. The phenomena of copper began to melt and dissolved in the iron matrix above sintering temperature 1080 ˚C have also contributed to the increase in sintered density of the compact. In all cases the sintered density increases as sintering temperature increase.

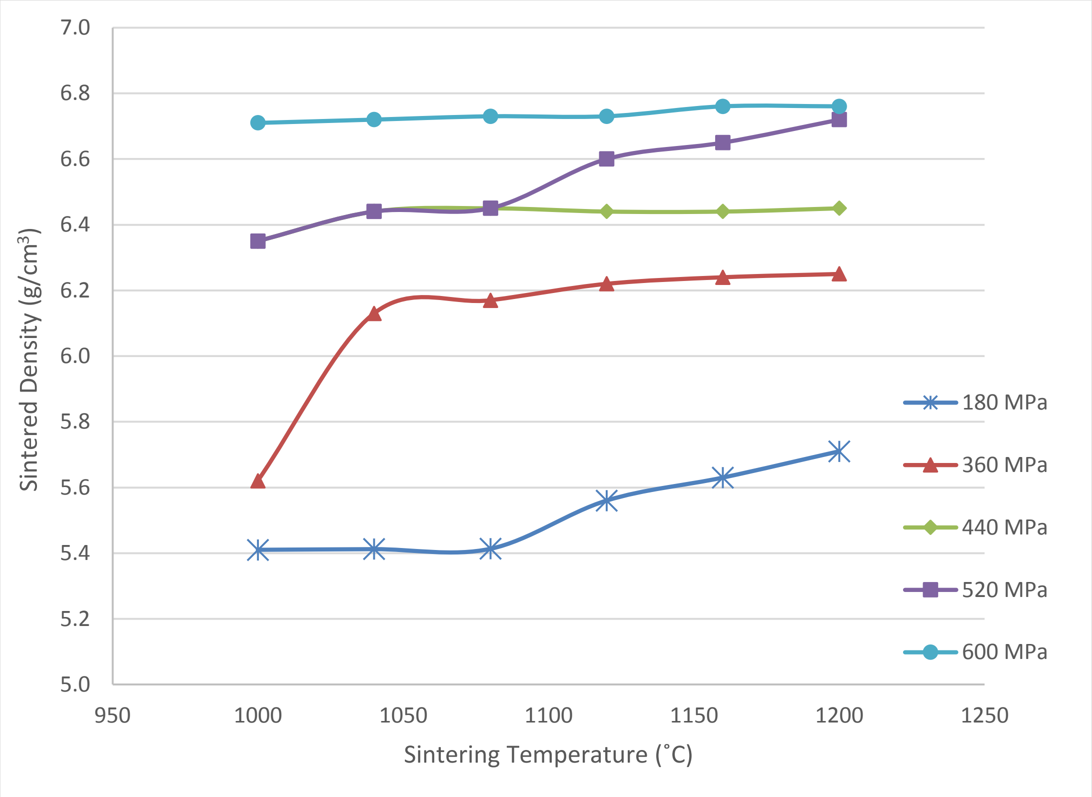

FIGURE 4.6. Variation of the sintered density of different compaction pressure at various sintering temperatures

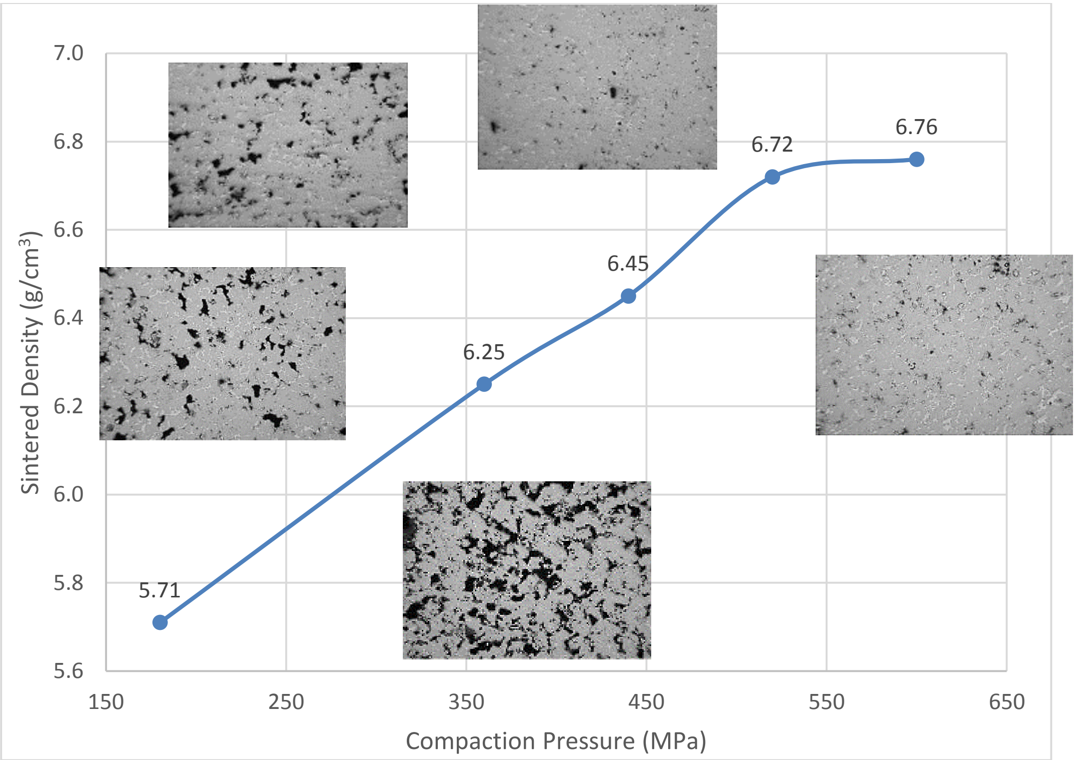

FIGURE 4.7. The sintered density of the compacts vs. various compaction pressure at sintering temperature of 1200 ˚C.

The compaction pressure also play a significant role in densification of the compact as shown in *Figure 4.7*. While maintaining the sintering temperature at 1200 ˚C, the existence of the pores was clearly visible in the compact which was compacted at 180 MPa. From the photomicrographs, it can be clearly seen that as the compaction pressure increase, the porosity level of the samples were reduced. From the results obtained, compaction pressure of 600 MPa is necessary to minimize the present of pores in the compacts.

4.4 Surface Hardness

The surface hardness data in *Figure 4.8* showed a slight increase in hardness value from 88 to 200 HV with an increase in sintering temperature from 1000 ˚C to 1200 ˚C. This increase in hardness was due to the initially compacted structure before sintering, increase in the extent of sintering and diffusion of carbon in the iron matrix. The mechanism of copper began to melt and dissolved in the iron matrix at sintering temperature above 1080 ˚C and near to complete bonding to bonding phenomena have also contributed to the increase in hardness of the compact. Sintering at higher temperature (>1000 ˚C) leads to formation of more sintered contacts, while the metal matrix structure was technically homogenous. Therefore, better hardness and sintered density was expected.

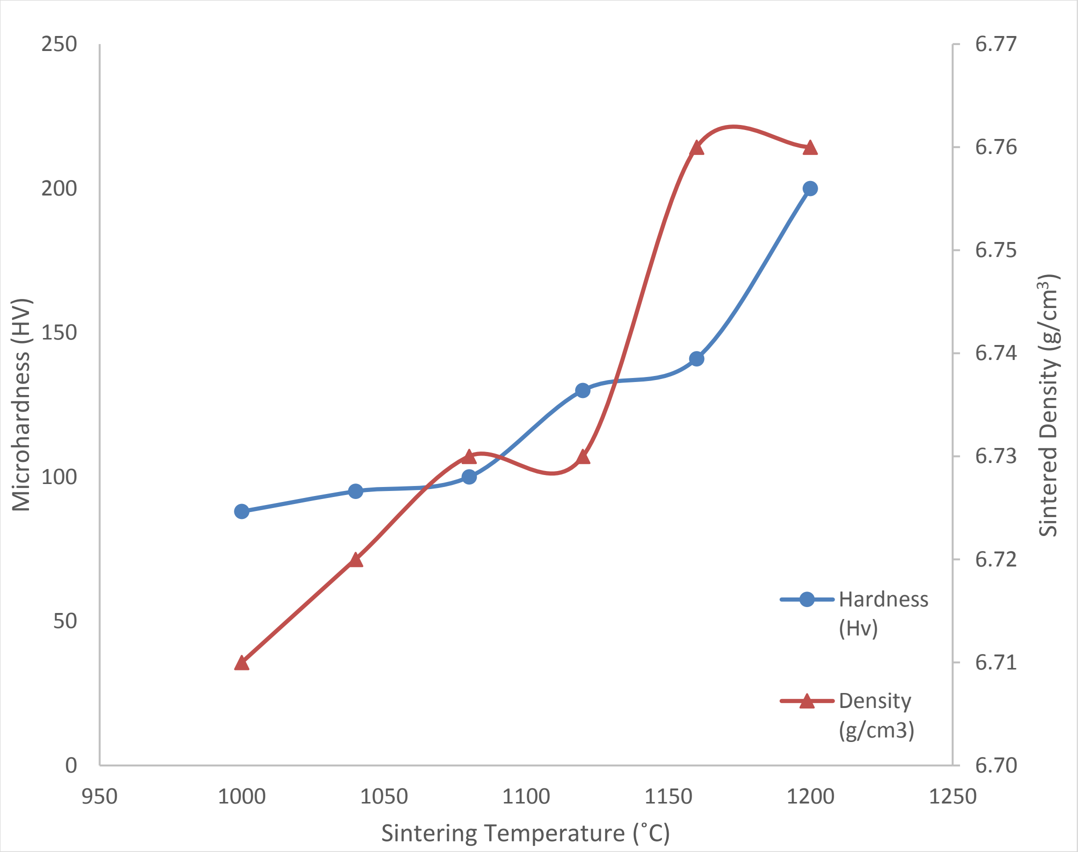

FIGURE 4.8. Variation of micro-hardness and sintered density with sintering temperature at compaction pressure of 600 MPa.

From *Figure 4.8*, the density of the sample increases as the sintering temperature increase. At sintering temperature between 1080 ˚C and 1120 ˚C, there is slight increase in density due to the process of melting of copper had taken place. As the copper started to melt, the liquid copper started to diffuse into iron matrix. This describe the sudden increase in the rate of density increase because molten copper started to fill in the voids thus making the samples denser.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The studies of the microstructure evolution in different sintering temperature can improve the understanding of the behavioral changes of the Fe-Cu-C system. From the study, the results showed that the sintering temperature was a critical factor influencing the sintered microstructure, densification behavior and hence the hardness of the Fe-Cu-C materials. The optimum sintering temperature for Fe-Cu-C was at 1200 ˚C. The physical properties of the compacts were as follow: sintered density of 6.76 g/cm^3; Maximum hardness value of 200 HV was also measured for samples sintered at 1200 ˚C. The optimum compaction pressure applied to ensure maximum compaction of Fe-Cu-C powders was at 600 MPa.

5.2 Recommendation

The developments in Powder Metallurgy (PM) method has given a great opportunity for the studies of more complex alloys and producing more sophisticated alloys with the customized properties. In future study, it is recommended that the study should include the effect of the different percentage of Fe-Cu-C composition to determine the effect of each elements towards the properties of the material.

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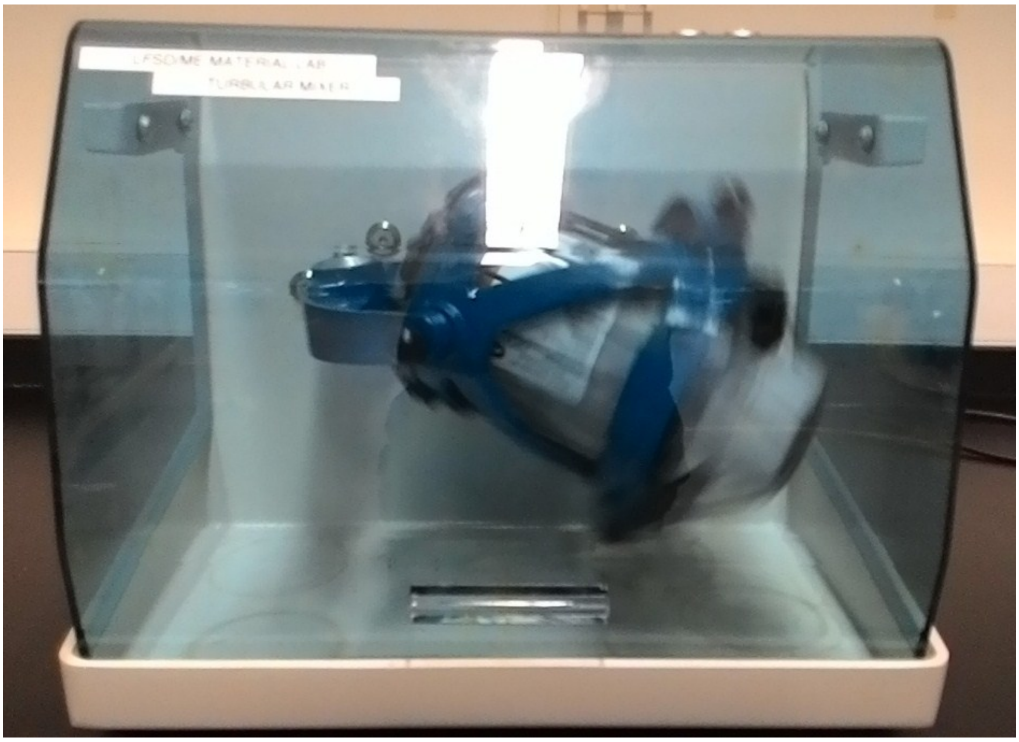
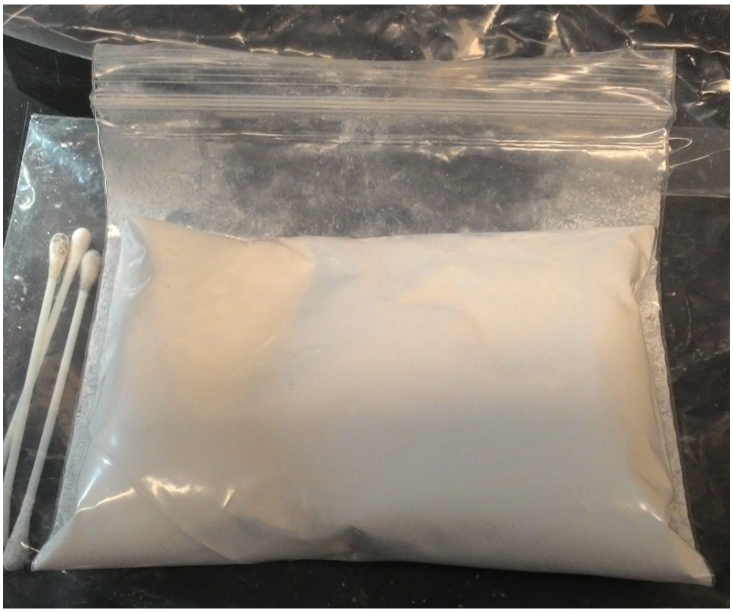
APPENDICES

APPENDIX 1. Iron, Copper and Carbon powders before mixing



APPENDIX 2. Mixing of the powder mixture in Tubular mixer

APPENDIX 3. Die in compaction process



APPENDIX 4. Zinc Stearate used as die wall lubricant

$$Load\ (N) = Pressure\ (MPa)\ \times\ Area\ of\ die\ punch\ (mm^2)$$

$$Area\ of\ die\ punch\ (mm^2) = \frac{\pi\ \times ((12\ mm)^2)}{4} = 113.1\ mm^2$$

Compaction Load, kN : = 180 MPa $\times\ 113.1\ mm^2\ =\ 20.4\ kN$

= 360MPa $\times\ 113.1\ mm^2\ =\ 40.7\ kN$

= 440 MPa $\times\ 113.1\ mm^2\ =\ 49.8\ kN$

= 520 MPa $\times\ 113.1\ mm^2\ =\ 58.8\ kN$

= 600 MPa $\times\ 113.1\ mm^2\ =\ 67.9\ kN$

APPENDIX 5. Calculation for converting compaction load to pressure

	Compaction Pressure, MPa	Sintering Temperature, ˚C					
		1000	1040	1080	1120	1160	1200
Samples Label	180	FX01	FX11	FX21	FX31	FX41	FX51
	360	FX02	FX12	FX21	FX31	FX41	FX51
	440	FX03	FX13	FX21	FX31	FX41	FX51
	520	FX04	FX14	FX21	FX31	FX41	FX51
	600	FX05	FX15	FX21	FX31	FX41	FX51

APPENDIX 6. Samples Labeled for sintering process

APPENDIX 7. SIMPLEMET Automatic Mounting Press

APPENDIX 8. Phenolic powder for mounting of samples

APPENDIX 9. Grinder and Polisher machine model Metaserv 2000

APPENDIX 10. SiC grit paper 180 (left) and 400 grit (right).

APPENDIX 11. Grinding process in progress

APPENDIX 12. Drying process of samples

APPENDIX 13. Etching process of samples

APPENDIX 14. LECO LM 247 AT Vickers Micro Hardness Tester