Characterization and Properties of Nickel Powder and Carbon Powder Mixture

by

Mohd Fasya b. Ramsah

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Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Mechanical Engineering Programme
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Approved by,

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AP DR OTHMAN BIN MAMAT

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

January 2008

CERTIFICATION OF ORIGINALITY

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

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MOHD FASYA BIN RAMSAH

ABSTRACT

This project is planned to provide new metal powders (mixture) with better properties compared to pure powders. P/M additive is a small amount of material or powder that normally being mixed into the base powder in order to improve certain property of P/M sintered part. 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 to the base powder mix prior to compacting and sintering of parts. Nickel powder as the base powder and graphite powder as the additive were used in the study. The objective of this project is to study the homogeneity, compressibility and sintering of the powder mixture. The homogeneity of the mixture was determined by using scanning electron microscope (SEM) whilst the compressibility of the mixture was determined based on the compressibility curve produced. The sintered properties were determined from the sintered density curve as well. From this study, the powder mixture was found homogeneous. Compressibility of mixed powders was increased with pressure. The maximum green density and sintered density were 6.66 g/cm3 and 7.43 g/cm3 respectively.

ACKNOWLEDGEMENT

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To all the technicians especially Mr Anuar, I would like to extend a heartfelt thank you for the guidance and effort in helping me with all their technical expertise and facilities.

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 individuals who have contributed in one way or another in initiating this project.

Thank you.

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CHAPTER 1
INTRODUCTION

1.1 Background of Study

Metal powders are used in industry for a diversify of products, including catalysts, welding electrodes, paints, printing inks, explosives, etc, in all of which the powder particles retain their identities. Traditional powder metallurgy, on the other hand, in its simplest definition, is a process whereby a solid metal, alloy or ceramic in the form of a mass of dry particles, normally less than 150 microns in maximum diameter, is converted into an engineering component of pre-determined shape and processing properties which allow it to be used in most cases without further processing. The basic steps in the traditional process are those of powder production; compaction of the powder into a handleable preform; and 'sintering', which involves heating the perform to a temperature below the melting point of the major constituent when the powder particles lose the identities through inter-diffusion processes and the required properties are developed [1].

Powder metallurgy (P/M) is one of several commercial metalworking processes for the fabrication of parts, components, or semi-finished products. Metal parts can be made by compacting metal powders in tool-steel or carbide dies and sintering (heating without melting) through P/M process. Its first use was in the early 1900s to make the tungsten filaments for incandescent light bulbs. This process is suitable for many applications because of the availability of a wide range of powder production, the capability to produce parts to net dimensions (net-shape forming), and the economics of the overall operation. P/M produces high performance components for instance gears, cams, bushings, cutting tools and automotive components such as piston rings and connecting rods [2].

1.2 Problem Statement

1.2.1 Problem Identification

In this project, graphite powders provide in the laboratory become additive to P/M nickel powders. Within the time constraint, the experiment will focus on establishing the sintered P/M samples and analyze the characteristics. The microstructure images of both powders are determined using SEM as attached in **Appendix A (Figure A1, A2)**.

1.2.2 Significant of the Project

The experimental project is significant in producing new metal powders (mixture) with better properties compared to pure nickel powder. The study gives the understanding on P/M production and properties. P/M can be used as the auto parts such as gears, cams and also globally used in fabrication industry.

1.3 Objective and Scope of Study

The objectives of the project are:

- To study the mixture of both powders/homogeneity of the mixture
- To study the compressibility of the mixture.
- To study the sintered properties of the mixture.

The scope of study for this project is to analyze the characterization and properties of the new metal powders (mixture). Moreover, the student has to determine the microstructure images of the powders by using SEM.

1.3.1 The Relevancy of the Project

The project is a platform to produce new metal powders by using P/M technique. From this project, the characterization and properties of the new mixture will be analyzed and being compared to the pure powders.

1.3.2 Feasibility of the Project within the Scope and Time Frame

Mechanical engineering students need to complete the project within 2 semesters. It is presumed that the project is feasible within the scope and time frame if there are no issues with regard to equipment function and material availability. Research work is allocated to be done first and the experimental work will started as early as possible to avoid the unforeseen shortcomings. The Gantt chart for second semester is attached in **Appendix A (Table A).**

CHAPTER 2
LITERATURE REVIEW

2.1 Extra Fine Nickel Powder

The use of fine powders in powder metallurgy could provide several benefits: higher strength, faster sintering, and increased diffusion of alloying elements leading to higher density and higher performance PM parts. However, fine powders often pose processing difficulties with particle agglomeration. Generally as the particle size decreases, the greater the tendency to agglomerate and the poorer the flow of material [3].

The benefit of increased diffusion of nickel (Ni) and improved distribution of fine nickel has to be weighed against the perceived benefit in relatively large, randomly dispersed austenitic nickel-rich phases obtained with standard nickel powder in typical sintered nickel steel microstructures. With varying Ni powder size, a range of microstructures in sintered Ni steel can be envisioned, from austenitic Ni-rich phases with standard Ni powder to the complete absence of Ni-rich phases with prealloyed Ni steel powders [3].

The optimum properties for a given application may lie in between these boundaries, in order to improve a combination of hardenability associated with prealloyed Ni steels and sufficient toughness and ductility normally associated with admixed Ni steels [3].

Alloying elements are often added to iron powder mixes to increase mechanical properties of sintered steels by allowing martensite formation at slower cooling rates and lower carbon content. There has been an increasing trend in recent years to use prealloyed molybdenum-ferrous powders for higher performance / higher density applications. Prealloyed molybdenum (Mo) is particularly effective at increasing hardenability without a loss in compressibility of the Fe powder. Unlike Mo, which promotes the formation of ferrite, Ni stabilities austenite and is a ferrite streghtener. Prealloyed Ni therefore hardens Fe powders, resulting in a loss of compressibility. A combination of prealloyed Mo and admixed Ni has been found to be particularly effective in sinter-hardening alloys. While admixed Ni imparts added ductility and toughness to sinter hardening alloys, maximum performance of Ni is difficult to achieve in sinter-hardened condition as compared to quenched and tempered condition due to limited diffusion and retained austenite. To overcome this limitation, the trend has been to use hybrid mixes with some prealloyed and admixed Ni [4].

Two nickel powders were admixed to the base Fe powder to produce FLN-4400 alloys: Inco T123 PM (standard Ni powder) and T110 PM (extra fine Ni powder). These 2 Ni powders have average particle sizes of approximately 7mm to 1.5mm respectively, as measured by laser scattering particle size analysis (see **Figure 2.1, 2.2**).

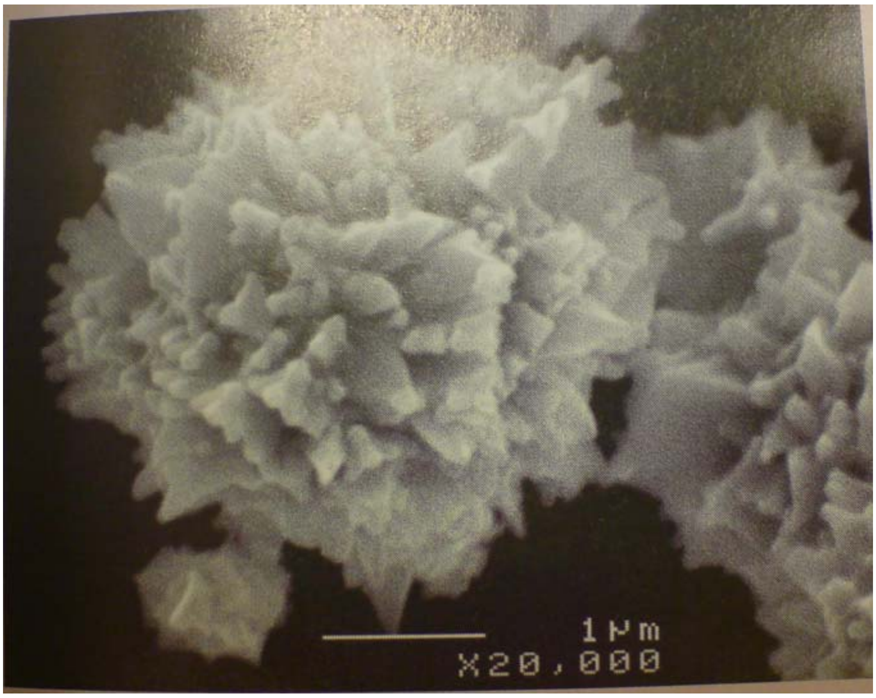


Figure 2.1: Standard Ni powder [4]

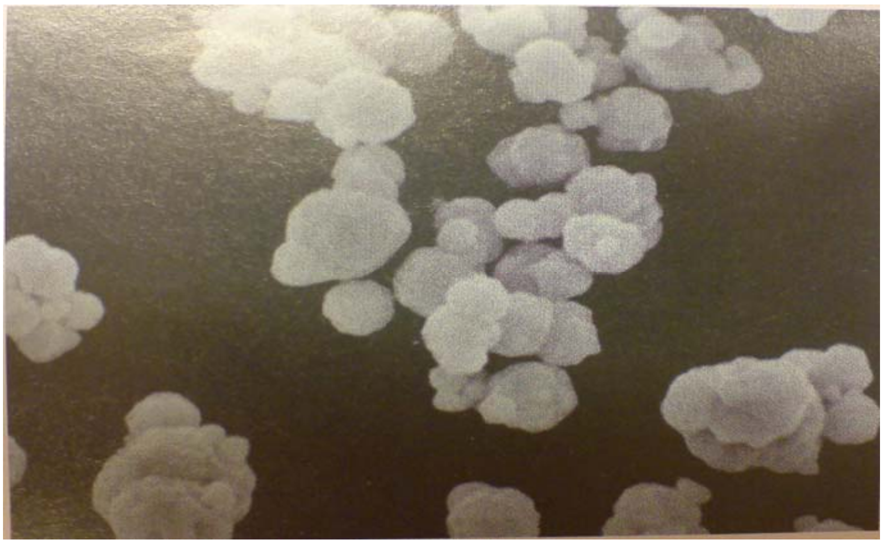


Figure 2.2: Extra fine Ni powder [4]

Tensile and Charpy impact tests were performed in accordance to with MPIF standards. Tensile properties were determined from a set of 15 tests bar per test condition. Hardness and density measurements were made on sets of five Charpy bars. Fatigue testing on heat-treated samples was performed by the rotating beam method. The fatigue endurance limit was determined for sets of 15 RBF samples by staircase method according to MPIF Standard 56. The physical testing results are summarized in **Table 2.1**.

Table 2.1: Physical properties comparison of FL-4405, FLN-4405 and FLN4-4405 in the as-sintered and heat-treated condition [4].

Material	**UTS (Mpa)**	**Elongation (%)**	**Hardness HRB/C**	**Impact Energy**
FLN2-4405 (XF)	765	0.74	95	35
FLN2-4405 (S)	775	0.95	94	40
FLN4-4405 (XF)	945	0.7	104	41
FLN4-4405 (S)	920	0.94	101	44
FL-4405	610	1.14	83	27
FLN2-4405HT (XF)	1430	0.2	41	23
FLN2-4405HT (S)	1500	0.36	41	19
FLN4-4405HT (XF)	1390	0.3	41	20
FLN4-4405 HT(S)	1390	0.3	40	17

Note XF = extra fine Ni powder; S = standard Ni powder

Fatigue endurance limit (FEL) determined by the rotating beam method are listed in **Table 2.2**. The FEL of Ni-containing alloys was more than 10% higher than the base FL-4405 alloy in the heat treated condition. The UTS of the heat-treated alloys was similar for both nickel-containing and base alloys, reporting the FEL as a percentage of UTS cannot be applied to these results. Statistically there was a little difference at FEL between the alloys containing admixed nickel powder [4].

Table 2.2: Rotating beam fatigue endurance limit (Mpa) of FL-4405HT alloys containing extra-fine (XF) and standard (Std) Ni powder for 10, 50 and 90% confidence intervals [4].

MIX	S_A^{10}	S_A^{50}	S_A^{90}
FLN2-4405HT XF Ni	424	407	389
FLN4-4405HT XF Ni	409	384	379
FLN2-4405HT Std Ni	430	422	391
FLN4-4405HT Std Ni	453	422	391
FLN-4405HT	387	365	343

The cutectoid in the as-sintered microstructure of FL-4405 steels has relatively coarse carbide plates with difficult-to-resolve ferrite. Addition of standard nickel powder results in a microstructure consisting of Ni-rich phases and pearlite (see **Figure 2.3**). There is some formation of martensite at the outer boundaries of these nickel-rich phases, while the centers are typically retained austenite. Extra-fine nickel powder addition results in the formation of martensitic Ni-rich phases in the as-sintered microstructure (see **Figure 2.4**). Extra-fine Ni-rich phases are more uniformly dispersed in the sintered steel than with the standard Ni powder. Bainitic regions were prevalent in the FLN4-4405 steels admixed with extra fine Ni powder [4].

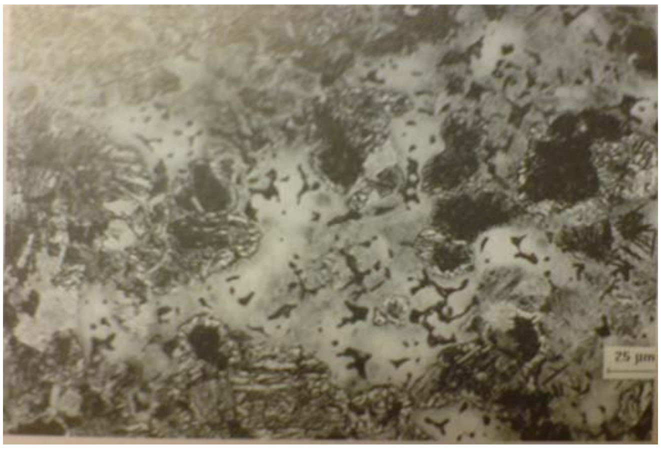


Figure 2.3: FLN4-4405 standard Ni powder [4]

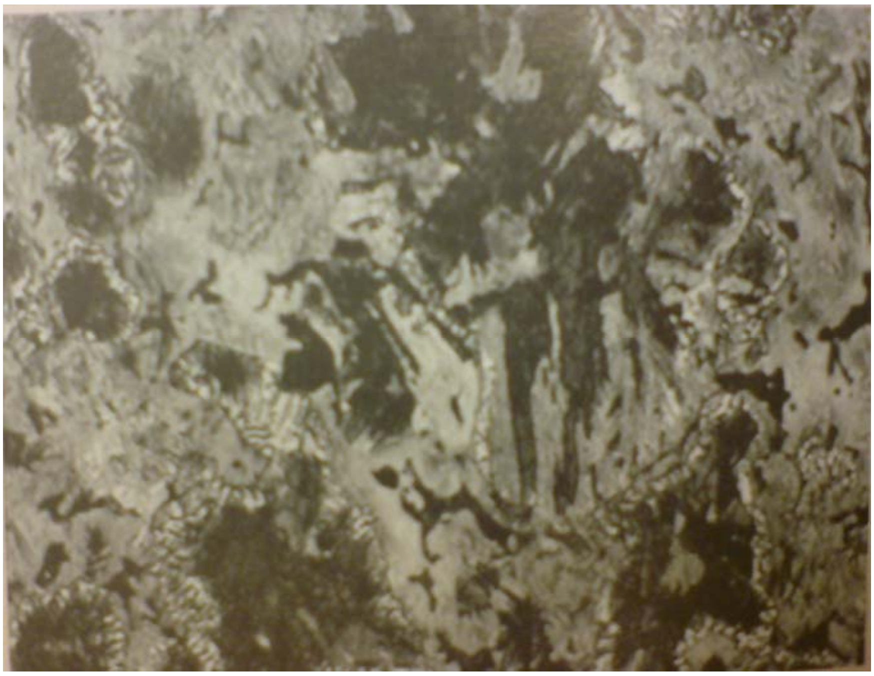
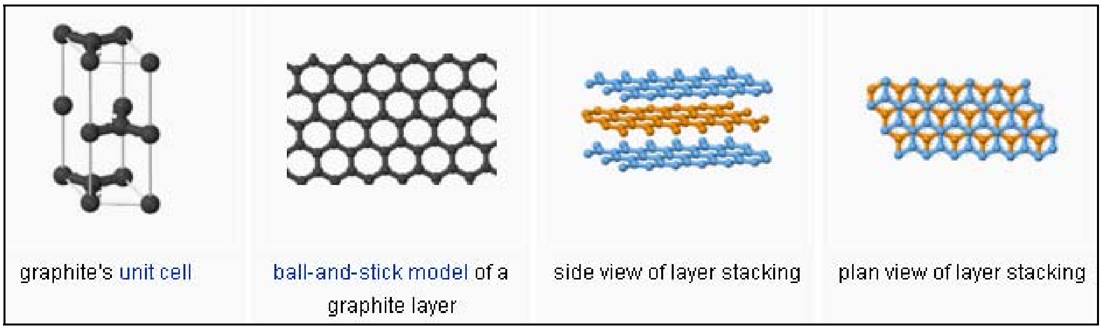


Figure 2.4: FLN4-4405 extra fine Ni powder [4]

2.2 Graphite Powder

Graphite is an electrical conductor, and can be used, for instance, in the electrodes of an arc lamp. Graphite holds the distinction of being the most stable form of carbon under standard conditions. Therefore, it is used in thermochemistry as the standard state for defining the heat of formation of carbon compounds. Graphite may be considered the highest grade of coal, just above anthracite and alternatively called meta-anthracite, although it is not normally used as fuel because it is hard to ignite [5].

There are three principal types of natural graphite, each occurring in different types of ore deposit: (1) Crystalline flake graphite (or flake graphite for short) occurs as isolated, flat, plate-like particles with hexagonal edges if unbroken and when broken the edges can be irregular or angular; (2) Amorphous graphite occurs as fine particles and is the result of thermal metamorphism of coal, the last stage of coalification, and is sometimes called meta-anthracite. Very fine flake graphite is sometimes called amorphous in the trade; (3) Lump graphite (also called vein graphite) occurs in fissure veins or fractures and appears as massive platy intergrowths of fibrous or acicular crystalline aggregates, and is probably hydrothermal in origin. **Figure 2.5** below shows the unit cell of graphite and graphite layer in several views [5].



graphite's unit cell

ball-and-stick model of a graphite layer

side view of layer stacking

plan view of layer stacking

Figure 2.5: Graphite's unit cell and graphite layer in several views [5]

2.3 Methods of Powder Production

Atomization produces a liquid metal stream by injecting molten metal trough a small orifice. The stream is broken up by jets of inert gas, air, or water. The size of the particles formed depends on the temperature of the metal, the rate of flow, nozzle size, and jet characteristics. Melt-atomization methods are widely used for production of powders for P/M. In one variation of this method, a consumable electrode is rotated rapidly in a helium-filled chamber. The centrifugal force breaks up the molten tip of the electrode, producing metal particles [2].

2.3.1 Atomization

Atomization produces a liquid metal stream by injecting molten metal trough a small orifice. The stream is broken up by jets of inert gas, air, or water. The size of the particles formed depends on the temperature of the metal, the rate of flow, nozzle size, and jet characteristics. Melt-atomization methods are widely used for production of powders for P/M. In one variation of this method, a consumable electrode is rotated rapidly in a helium-filled chamber. The centrifugal force breaks up the molten tip of the electrode, producing metal particles [2].

2.3.1.1 Gas Atomization

Gas atomization is a production process that gives spherical steel powders. A melt is poured through a gas expansion nozzle to form droplets that solidify into powder. Most powders are in the 15-300 µm range, but smaller powders are possible. The greater the energy in the gas, the smaller the powder will be. Typical gas used is nitrogen or argon. Melting can be performed in air for alloys less sensitive to oxidation [6].

2.3.1.2 Water Atomization

Water atomization is the most common technique for producing elemental and alloy ferrous powders. At the core of water atomization, high pressure water jets are directed against the melt stream, forcing disintegration of the melt into droplets that solidify into irregular shapes. The water can be directed by a single jet, multiple jets, or an annular ring.

The molten metal is transported to a water atomization facility where the melt is granulated by water jets, dried, and subsequently decarburized (carbon is extracted by heat and atmosphere reactions) to produce iron powder. Oxidation is useful in removing the carbon, but a final stage reduction is necessary to remove oxygen. Since the reduction process causes the particles to sinter-bond, subsequent milling is required to obtain discrete particles.

The powder shape is irregular and rough with some oxidation because of rapid cooling. Oils or other liquids have been used instead of water to gain better control of particle shape and oxidation. Pressure is the main process control variable in water atomization. Higher water pressures result in higher water velocities and small particle sizes [6].

2.3.1.3 Centrifugal Atomization

A spinning disk or other means is used to impart centrifugal force to a melt in centrifugal atomization. Droplets are thrown off as a fine spray that solidifies into spheres. The consumable electrode must be made from the desired alloy, since there is no alloying in the thin melt layer on the end of the electrode. The size of powders depends on the electrode size and spinning rate. Usually, the process is performed under inert gas to protect the powder from oxidation. Benefits of centrifugal atomized powders are the purity, spherical shape, and uniform apparent density. The disadvantages are a low production rate, high equipment, processing costs, and coarse particle size [6].

2.3.2 Oxide Reduction

Reduction of metal oxides uses gases, such as hydrogen and carbon monoxide, as reducing agents; thus, very fine metallic oxides are reduced to the metallic state. The powders produced by this method are spongy and porous and have uniformly sized spherical or angular shapes [2].

2.3.3 Electrolytic Deposition

Electrolytic deposition uses either aqueous solutions or fused salts. The powders produced are among the purest of all metal powders [2].

2.3.4 Carbonyls

Metal carbonyls, such as iron carbonyl ($Fe(CO)_5$) and nickel carbonyl ($Ni(CO)_4$), are formed by letting iron or nickel react with carbon monoxide. The reaction products are the decomposed to iron and nickel, producing small, dense, and uniform spherical particles of high purity [2].

2.3.5 Mechanical Communition

A simple means for making powder is to grind, file or hammer piece of steel. Mechanical communition (pulverization) involves crushing milling in a ball mill, or grinding brittle or less ductile metals into small particles. A ball mill is a machine with a rotating hollow cylinder that is partly filled with steel or white cast-iron balls. When made from brittle materials, the powder particles have angular shapes, whereas when they are made from ductile metals, they are flaky and are not particularly suitable for powder metallurgy applications [2].

2.3.6 Mechanical Alloying

Mechanical alloying where powders of two or more pure metals are mixed in a ball mill. Under the impact of the hard balls, the powders repeatedly fracture and weld together by diffusion, forming alloy powders [2].

2.3.7 Other Methods

Other methods that are less used include precipitation from a chemical solution, production of fine metal chips by machining, and vapor condensation. New developments include techniques based on high-temperature extractive metallurgical processes. Metal powders are being produced using high-temperature processing techniques based on the reaction of volatile halides (a compound of halogen and an electropositive element) with liquid metals and the controlled reduction and reduction/carburization of solid oxides [2].

2.4 Powder Characterization

2.4.1 Particle Size

Fine particles have it owns advantages. The surface finish would be smooth, because the pores would be small, and the fine particles, with their high surface area, would sinter rapidly, producing a higher density part [7]. For tool wear reasons, fine powders are better than powders with large particle size. During compaction, the flexible tool material is extruded into the voids between the particles causing a tearing effect. Larger particles give larger voids which increase the tearing [8].

The obvious way to measure particle size is using the little micron rulers and check the largest dimensions of each of the particles in SEM photos. Particle size is measured usually by screening that is by passing the metal powder through screens (sieves) of various mesh sizes. Screening is achieved by using a vertical stack of screens with increasing mesh size as the powder flows downward through the screens. The larger the mesh size, the smaller is the opening in the screen. The powder manufacturers use sieves that made from finely woven wire cloth. The size of the holes in the cloth or screen is controlled by the size of the wires and the number of wires/in. Besides screen analysis, several other methods are also used for particle-size analysis, particularly for powder finer than 45μm [2]:

- **Sedimentation** – involves measuring the rates at which particles settle in a fluid
- **Microscopic analysis** – including the use of transmission and scanning electron microscopy
- **Light scattering** – from a laser that illuminates a sample consisting of particles suspended in a liquid medium. The particles caused the light to be scattered, which is then focus on a detector that digitizes the signals and computes the particle-size distribution
- **Optical means** – such as having particles blocking a beam of light, which is then sensed by a photocell
- **Suspension of particles** – in a liquid a subsequent detection of particle size and distribution by electrical sensors

2.4.2 Particle Shape

Particle shape varies significantly between nickel powders. Further, shape influences packing, flow, and powder compaction. As measuring particle shape is difficult, qualitative descriptors are used to convey the particle shape. A full set of shape descriptors includes the following: spherical, angular, irregular, dendritic, sponge, cubic, polygonal, flake, ligamental, acicular, and rounded.

Particular shape varies with particle size and manufacturing technique (see **Figure 2.6, 2.7**). However, most particle size analyses assume a spherical shape. This is often invalid. This is because, normally particle shape is irregular and the cross section can be large differences in pore structure [6].

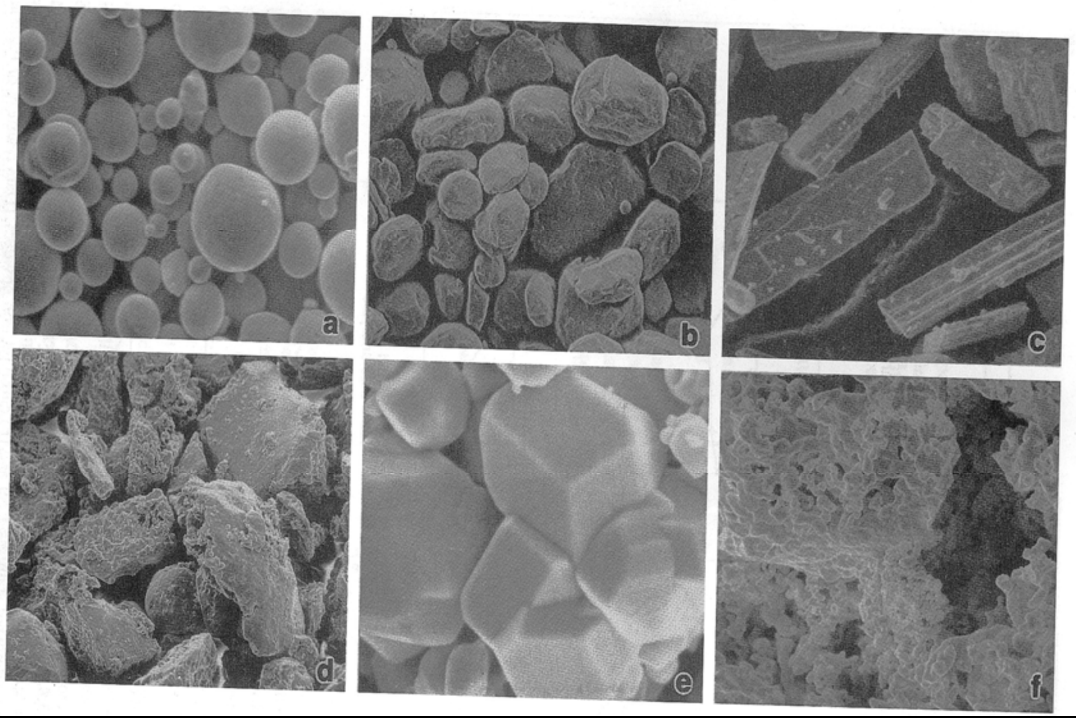


Figure 2.6: Diversity shapes in powders using SEM; (a) spherical; (b) rounded; (c) angular; (d) irregular; (e) polygonal; (f) sponge. [1]

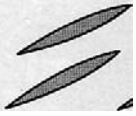
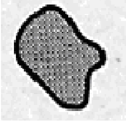
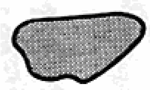
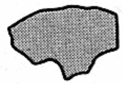
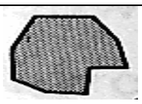
Particle shapes		Process Produced
Acicular		Chemical decomposition
Irregular rodlike		Chemical decomposition, mechanical comminution
Flake		Mechanical comminution
Dendritic		Electrolytic
Spherical		Atomization carbonyl (Fe), precipitation from a liquid
Rounded		Atomization, chemical decomposition
Irregular		Atomization, chemical decomposition
Porous		Reduction of oxides
Angular		Mechanical disintegration, carbonyl (Ni)

Figure 2.7: Particle shapes in metal powders and the processes by which they are produced [3].

2.4.3 Particle Packing and Flow

Small powders or irregular-shaped particle do not pack or flow easily. The small particles have a high surface area, and the irregular particles have many surface asperities. Sharp corners are a source of friction between particles that inhibit flow and packing. Both attributes are important to powder shaping, since the powder needs to fill out a die cavity rapidly and uniformly. The packing density decreases because of poor flow past neighbouring particles [6].

2.5 Mixing Metal Powders

Mixing powder is 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.

Powder mixing must be carried out under controlled conditions in order to avoid contamination and deterioration. Deterioration is caused by excessive mixing, which may alter the shape of the particles and work harden them; thus making the subsequent compacting operation more difficult. Powders can be mixed in air, in inert atmospheres (to avoid oxidation), or in liquids, which act as lubricants and make the mix more uniform. Several types of blending equipment are available such as V blender and double cone mixer (see **Figure 2.8, 2.9**). These operations are being increasingly controlled by microprocessors to improve and maintain quality.

Figure 2.8: V blender [9]

Figure 2.9: Double cone mixer [10]

Metal powders are explosive because of their high surface-area-to-volume ratio. Safety has to be considered during blending and during storage and handling. Precautions include grounding equipment, preventing the creation of sparks (by using non-sparking tools and avoiding the use o friction as a source of heat), and avoiding dust clouds, open flames, and chemical reactions [2].

2.6 Compaction

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. The powder must flow easily to feed properly into the die cavity. Pressing is generally carried out at room temperature, although it can be done at elevated temperature as well.

In powder metallurgy, one refers to density at three different stages:

- As loose powder
- As a green compact
- After sintering

The particle shape, average size, and size distribution dictate the packing density of loose powder. Spherical powder with a wide size distribution gives a high packing density. But a compact made from such powder has poor green strength, and, hence such powder is unsuitable for P/M parts to be made by the die-pressing method. For P/M parts production, powders with some irregularity of shape are preferred, even though the fill density of powder in the die is somewhat lower than that of spherical powder. For reproducibility of part dimensions, the fill density of the powder should be consistent from one powder lot to the next. Spherical powder is preferred for hot **isostatic pressing**; a special technique.

The density after compaction (green density) depends primarily on the compaction pressure, the metal powder composition, and the hardness of the powder. Higher pressure and softer powder give a higher green density. The green density and its uniformity within a compact improve with addition of a small quantity of admixed (blended-in) lubricant.

The higher the density, the higher will be the strength and elastic modulus of the part. The reason is that the higher the density, the higher will be the amount of solid metal in the same volume; hence the greater will be the part's resistance to external forces. Because of friction between the metal particles in the powder and between the punches and the die walls, the density can vary considerably within the part. This variation can be minimized by proper punch and die design and by friction control.

The compacting pressure required depends on the characteristics and shape of the particles, the methods of the blending, and the lubrication. The selection of the press depends on part size and configuration, density requirements, and production rate. The higher the pressing speed, the greater is the tendency to trap air in the die cavity.

Therefore, good die design, including provision of vents, is important so that trapped air does not hamper proper compaction [2].

Other compacting and shaping processes used in powder metallurgy include:

- Metal injection molding (MIM)
- Rolling
- Extrusion
- Pressureless compaction
- Ceramic molds
- Spray deposition

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. Before sintering, the compact is brittle, and its strength, known as green strength, is low. The nature and strength of the bond between the particles, depend on the mechanisms of diffusion, plastic flow, evaporation of volatile materials in the compact, recrystallization, grain growth, and pore shrinkage.

The sintered density of a part depends on the parts green density and on the sintering conditions, in terms of temperature, time, and furnace atmosphere. The sintered density increases with increasing values of temperature and time and usually with a more deoxidizing type of furnace atmosphere. For structural P/M part, a higher sintered density is very desirable, as it leads to better mechanical properties. Better properties and accuracy can be achieved by using a powder with a high compressibility.

Sintering temperatures are generally within 70% to 90% of the melting point of the metal or alloy. Continuous sintering furnaces are used for most production today. These furnaces have three chambers (see **Figure 2.10**):-

- A burn-off chamber to volatilize the lubricants in the green compact in order to improve bond strength and prevent cracking.
- A high temperature chamber for sintering
- A cooling chamber

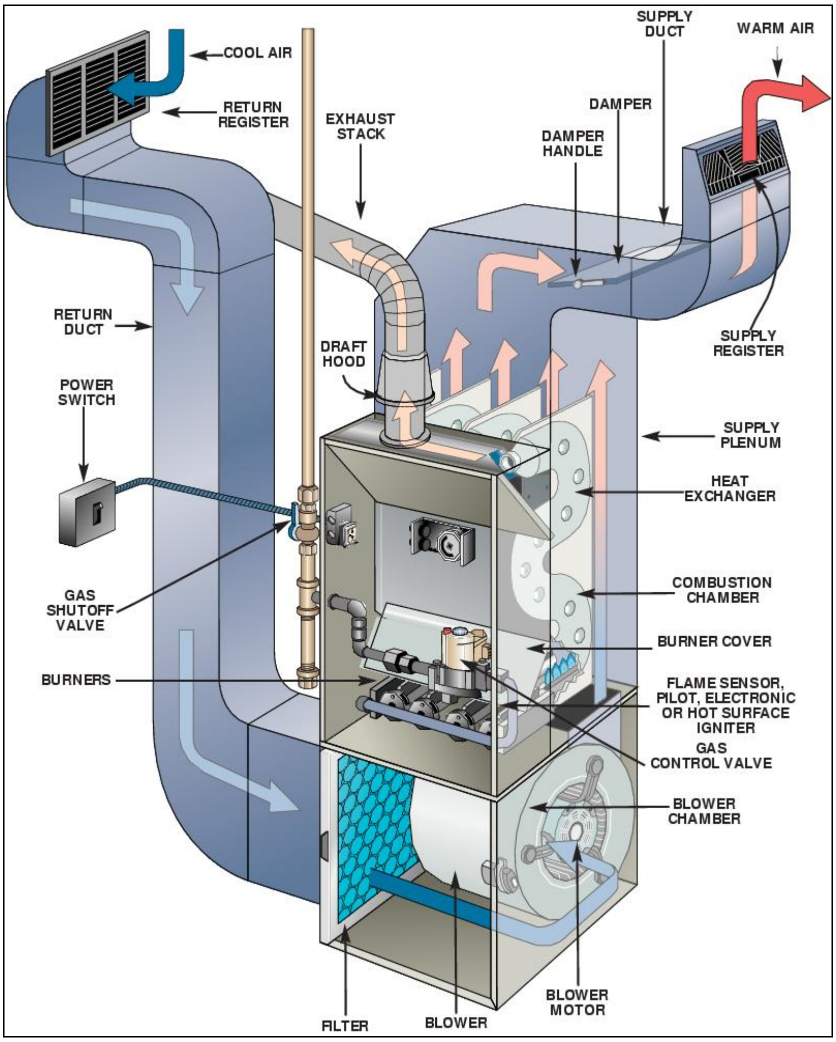


Figure 2.10: Components in a furnace [11]

Proper control of the furnace atmosphere is essential for successful sintering and to obtain optimum properties. Oxide inclusions have a detrimental effect on mechanical properties. For the same volume of inclusions, the smaller inclusions have a larger effect because there are more of them per unit volume of the part. 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.

Sintering mechanisms are complex and depend on the composition of the metal particles as well as the processing parameters. As the temperature increases, two adjacent particles begin to form a bond by diffusion (solid-state bonding) as shown in **Figure 2.11** below. As a result, the strength, density, ductility, and thermal and electrical conductivities of the compact increased. At the same time, however, the compact shrinks; hence, allowances should be made for shrinkage.

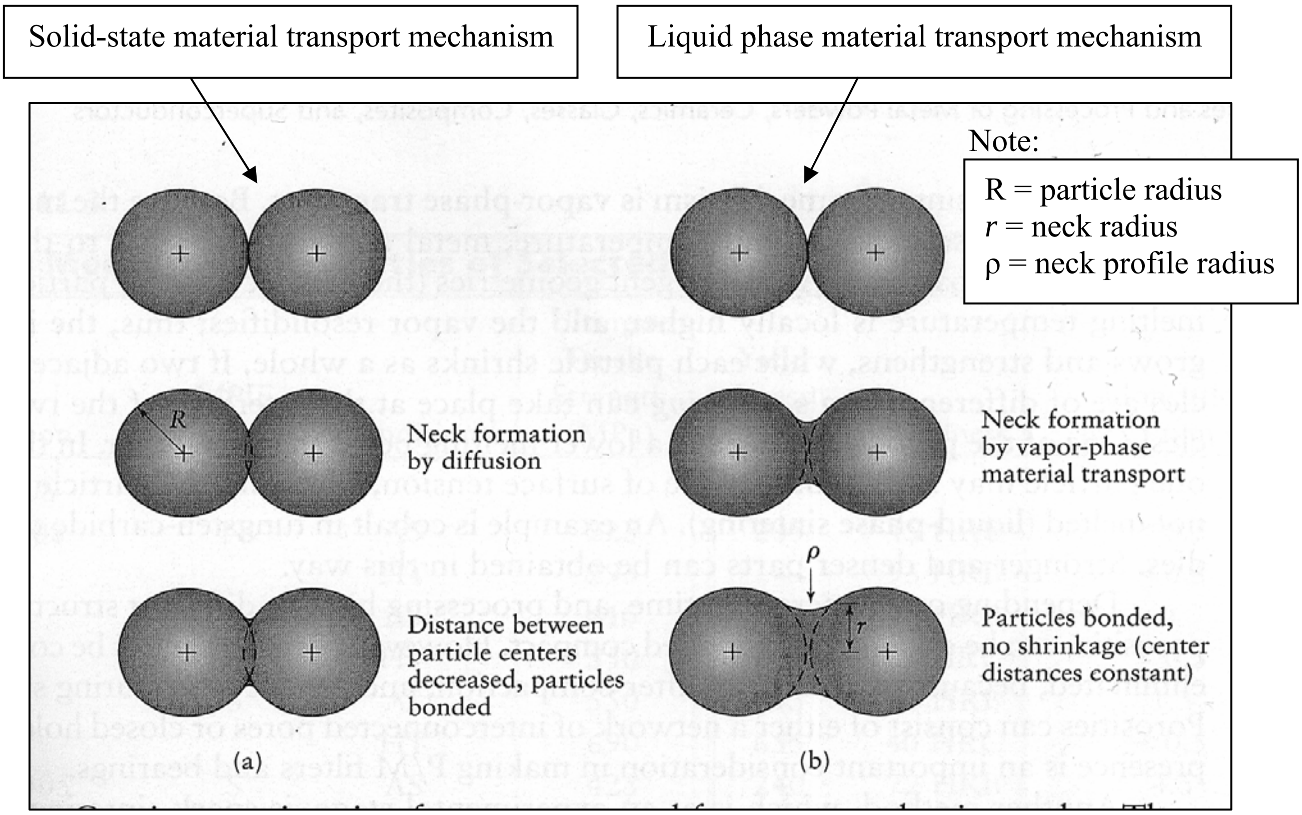


Figure 2.11: Schematic Illustration of 2 mechanisms for sintering metal powders [3]

A second sintering mechanism is vapour-phase transport. Metal atoms will release to the vapour phase from the particles because the material is heated very close to its melting temperature. At convergent geometries (the interface of two particles), the melting temperature is locally higher, and the vapour resolidifies; thus the interface grows and strengthens, while each particle is shrink as a whole. If two adjacent particles are of different metals, alloying can take place at the interface of the two particles. One of the particles may have a lower melting point than the other. In that case, one particle may melt and because of surface tension, surround the particle that has not melted (liquid-phase sintering).

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 [2].

CHAPTER 3

METHODOLOGY

3.1 Procedure Identification

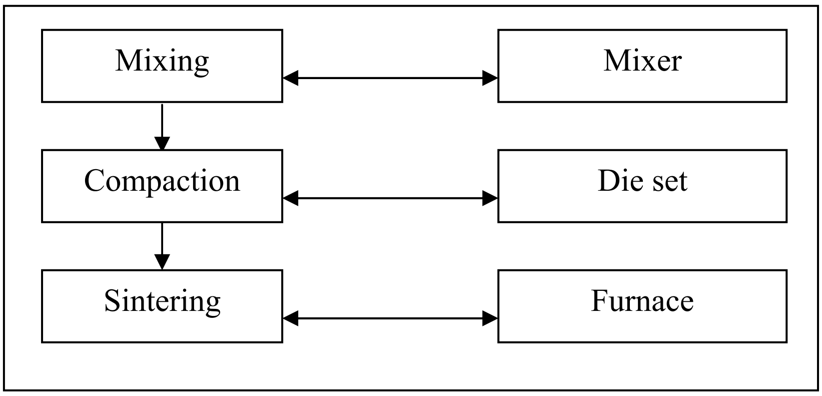
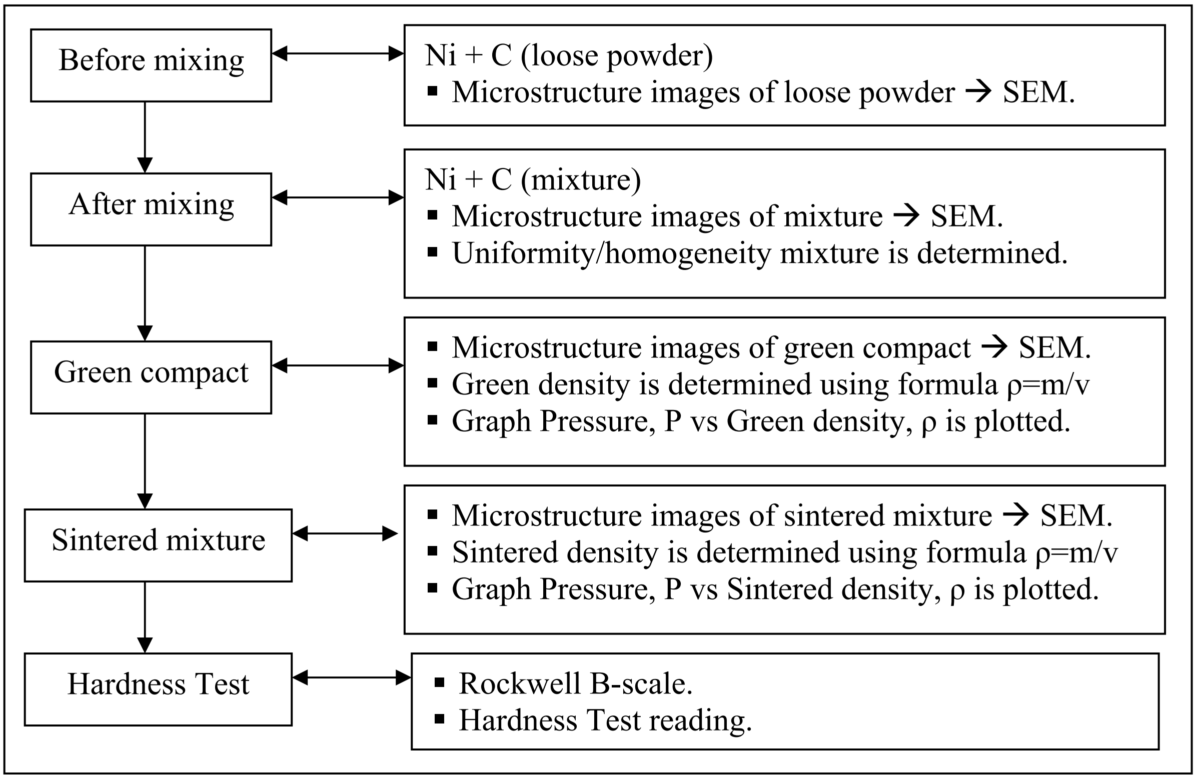


Figure 3.1: Flow chart of procedures and tools for (P/M) process



Ni + C (loose powder)
- Microstructure images of loose powder → SEM.

Ni + C (mixture)
- Microstructure images of mixture → SEM.
- Uniformity/homogeneity mixture is determined.

- Microstructure images of green compact → SEM.
- Green density is determined using formula ρ=m/v
- Graph Pressure, P vs Green density, ρ is plotted.

- Microstructure images of sintered mixture → SEM.
- Sintered density is determined using formula ρ=m/v
- Graph Pressure, P vs Sintered density, ρ is plotted.

- Rockwell B-scale.
- Hardness Test reading.

Figure 3.2: Flow chart of the process details

In producing the samples using powder metallurgy technique, firstly before mixing, microstructure images of the loose powders (nickel powder and graphite powder) are determined using SEM. Second, both powders are mixed manually together with a binder (2.02 wt %) which is phenolic thermosetting powder in order to bind nickel powder and graphite powder together. The microstructure images of the mixture are determined using SEM. Next, compaction process; where the mixture is 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. The microstructure images of the green compact are determined using SEM. A graph of Pressure, P vs Green Density, ρ is plotted whereby the green density is determined by using formula $\rho=m/v$. Besides, sintering process is applied to the green compact. Sintering is the process in which a 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 [2]. The microstructure images of the sintered pallet are determined using SEM and a graph of Pressure, P vs Green Density, ρ is plotted. Lastly, hardness test (Rockwell B-scale) is done on the pallets and the results are discussed.

3.2 Tools Required

In this project, tools required for the experiment are Mortar Grinder, Autopallet Press Machine, furnace, grinder and polisher, balancing beam, SEM and Hardness Testing Machine.

Mortar Grinder is a tool used to crush, grind, and mix substances. Grinding time and pressure of the mortar can be adjusted and fixed easily and work force can be saved [5]. In this project, mortar grinder is used to crush and grind the powders into fine particles.

Figure 3.3: Mortar Grinder

Autopallet Press Machine is used to compact the mixture into shapes in a die with controlled variable.

Figure 3.4: Autopallet Press Machine

Furnace is used to melt the metals and sintered the compacted mixture. There are several common type of furnaces; electric-arc furnaces, induction furnaces, crucible furnaces and cupolas.

Figure 3.5: Furnace

Grinder is a machine tool used for producing very fine finishes or making very light cuts and polisher is a machine tool which create a smooth and shiny surface by using rubbing or a chemical action, leaving a surface with significant specular reflection and minimal diffuse reflection [5].

Figure 3.6: Grinder and Polisher

Balancing beam (analytical balance) is an instrument that is used to measure mass to a very high degree of precision. The weighing pan(s) of a high precision (.01 mg or better) analytical balance are inside a transparent enclosure with doors so dust does not collect and so any air currents in the room do not affect the delicate balance [5].

Figure 3.7: Balancing Beam

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.

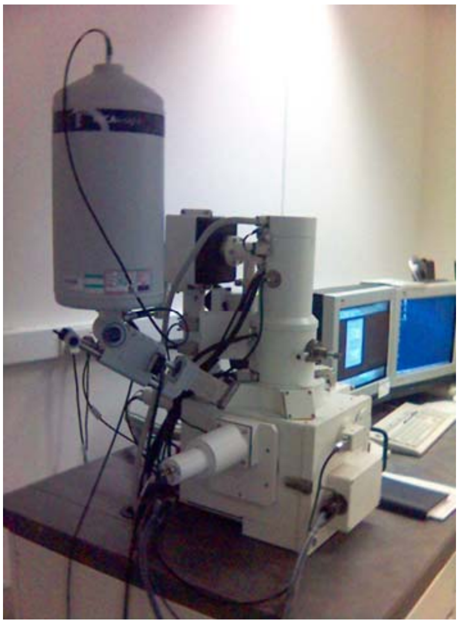


Figure 3.8: Scanning Electron Microscope

Hardness Testing Machine is a machine which determines the hardness by measuring the depth of penetration of an indenter under a large load compared to the penetration made by a preload [5].

Figure 3.9: Hardness Testing Machine

CHAPTER 4

RESULT AND DISCUSSION

4.1 Material Selection

This project is planned to provide new metal powders (mixture) with better properties compared to pure powders. P/M additive is a small amount of material or powder that normally being mixed into the base powder in order to improve certain property of P/M sintered part. At present, the student has developed the understanding on the process in powder metallurgy. The process involves three main stages, i.e. mixing, compaction and sintering. The P/M technique allows the blending (or mixing) of additives to the base powder mix prior to compacting and sintering of parts. In this project, the student has chosen nickel powder as the base powder (see **Figure 4.1**) and graphite powder as the additive (see **Figure 4.2**).

Figure 4.1: Nickel Powder (base powder)

Figure 4.2: Graphite Powder (additive)

Furthermore, the student has determined the particle shapes in both powders using SEM in order to know the processes by which they are produced. As for nickel powder, the particle shape obtained is rounded and hence, the powder is produced via atomization and chemical decomposition process (see **Figure 4.3**). Besides, graphite powder is in irregular shape; therefore, graphite powder is produced by atomization and chemical decomposition process (see **Figure 4.4**). The other microstructure images of different magnification (100X, 500X and 2000X zoom) of both powders are attached in **Appendix A (Figure A1, A2)**.

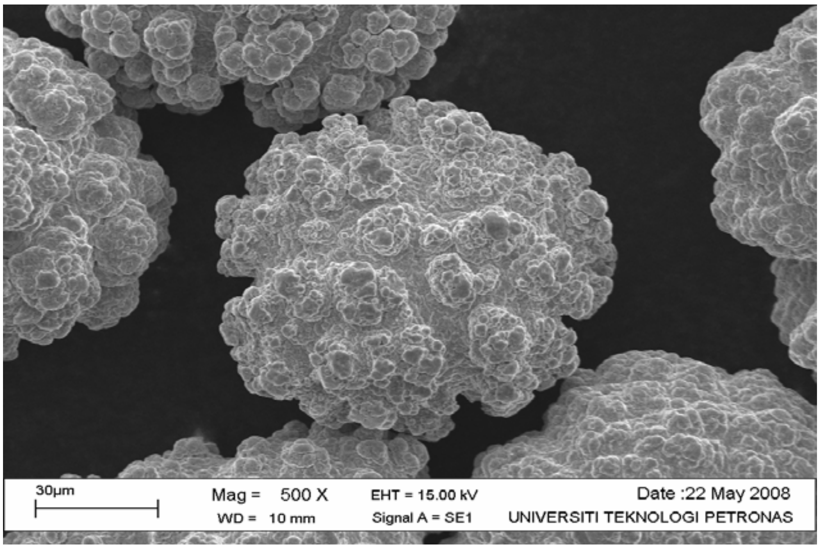


Figure 4.3: Nickel Powder (rounded shape) by which it is produced

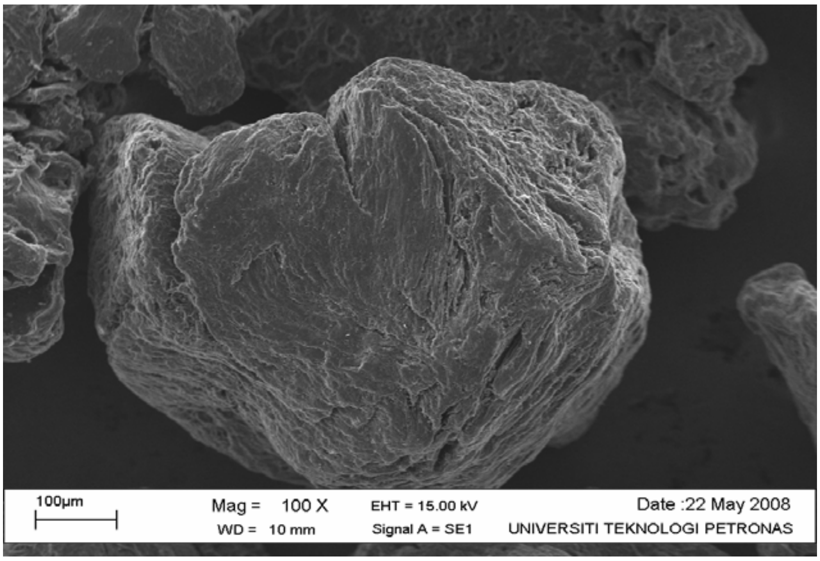
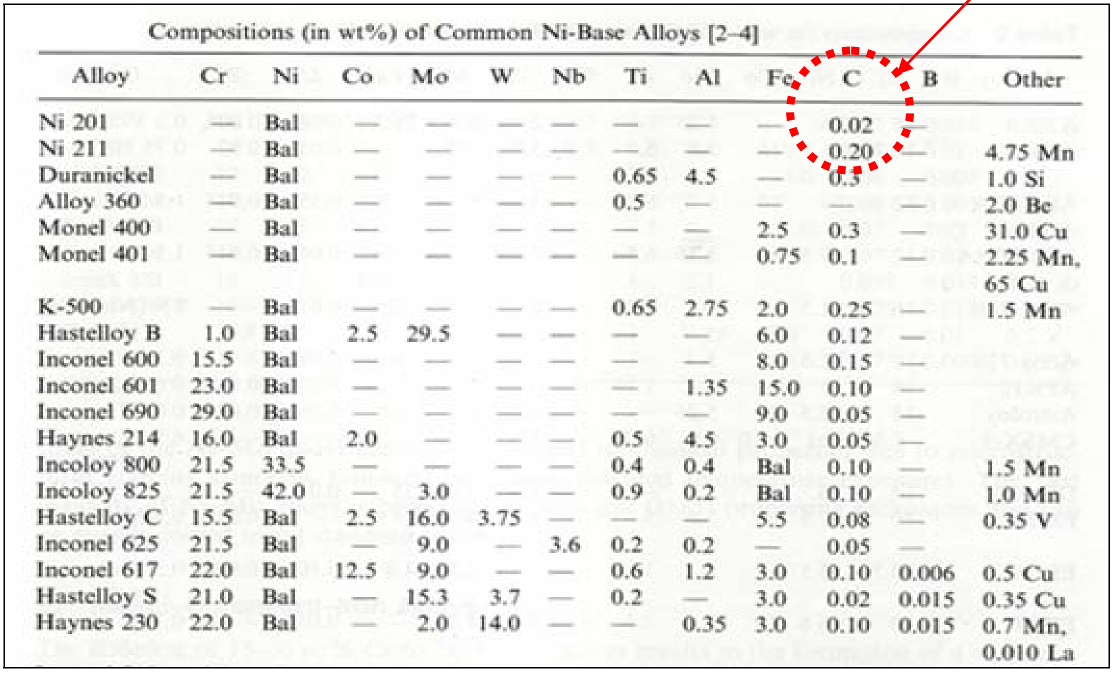


Figure 4.4: Graphite Powder (irregular shape) by which it is produced

4.2 Solubility

Moreover, the student has determined the minimum and maximum solubility for the Ni-C mixture. As referred to **Table 4.1** below, the minimum solubility of Ni-C mixture is 0.02 wt % whilst the maximum solubility of Ni-C mixture is 0.2 wt %. Hence, the student has established the optimum solubility of carbon (additive) which is 0.1 wt % and mix with the remainder 99.9 wt % nickel (base powder).

Table 4.1: Compositions (in wt %) of Common Ni-Base Alloys [3]



Compositions (in wt%) of Common Ni-Base Alloys [2–4]

Alloy	Cr	Ni	Co	Mo	W	Nb	Ti	Al	Fe	C	B	Other
Ni 201	—	Bal	—	—	—	—	—	—	—	0.02	—	
Ni 211	—	Bal	—	—	—	—	—	—	—	0.20	—	4.75 Mn
Duranickel	—	Bal	—	—	—	—	0.65	4.5	—	0.3	—	1.0 Si
Alloy 360	—	Bal	—	—	—	—	0.5	—	—	—	—	2.0 Be
Monel 400	—	Bal	—	—	—	—	—	—	2.5	0.3	—	31.0 Cu
Monel 401	—	Bal	—	—	—	—	—	—	0.75	0.1	—	2.25 Mn, 65 Cu
K-500	—	Bal	—	—	—	—	0.65	2.75	2.0	0.25	—	1.5 Mn
Hastelloy B	1.0	Bal	2.5	29.5	—	—	—	—	6.0	0.12	—	
Inconel 600	15.5	Bal	—	—	—	—	—	—	8.0	0.15	—	
Inconel 601	23.0	Bal	—	—	—	—	—	1.35	15.0	0.10	—	
Inconel 690	29.0	Bal	—	—	—	—	—	—	9.0	0.05	—	
Haynes 214	16.0	Bal	2.0	—	—	—	0.5	4.5	3.0	0.05	—	
Incoloy 800	21.5	33.5	—	—	—	—	0.4	0.4	Bal	0.10	—	1.5 Mn
Incoloy 825	21.5	42.0	—	3.0	—	—	0.9	0.2	Bal	0.10	—	1.0 Mn
Hastelloy C	15.5	Bal	2.5	16.0	3.75	—	—	—	5.5	0.08	—	0.35 V
Inconel 625	21.5	Bal	—	9.0	—	3.6	0.2	0.2	—	0.05	—	
Inconel 617	22.0	Bal	12.5	9.0	—	—	0.6	1.2	3.0	0.10	0.006	0.5 Cu
Hastelloy S	21.0	Bal	—	15.3	3.7	—	0.2	—	3.0	0.02	0.015	0.35 Cu
Haynes 230	22.0	Bal	—	2.0	14.0	—	—	0.35	3.0	0.10	0.015	0.7 Mn, 0.010 La

4.3 Mixing

Mixing is a process where nickel powder and graphite powder are mixed together using a mixer. Instead of using Ball Mill, a closed bottle is used as a mixer to mix the powders for 15 minutes as shown in **Figure 4.5** below. Ball Mill is not appropriate to mix both powders because it requires large amount of powders to balance its weight during operation.

The purpose of mixing is to:

- Achieve uniformity by mixing the powders that have different sizes and shapes.
- Impart special physical and mechanical properties and characteristics to the P/M product.

Figure 4.5: Conventional mixing process

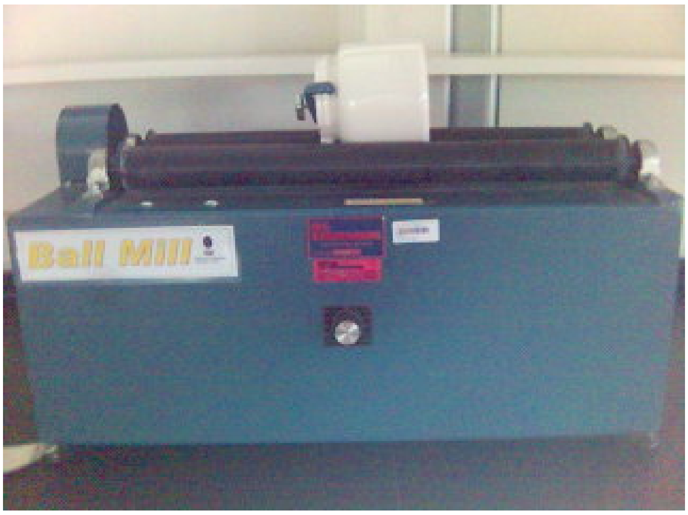


Figure 4.6: Ball Mill

As referred to the purpose of mixing, the student has determined the uniformity/homogeneity of the mixture using SEM. **Figure 4.7** below shows the microstructure image of the mixture.

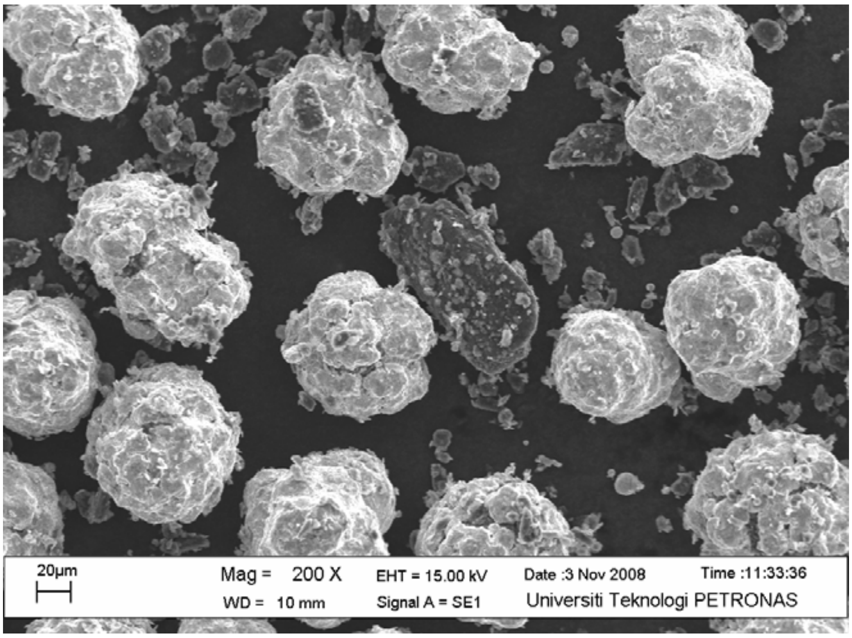


Figure 4.7: Uniformity/homogeneity of the mixture

Next, binder (2.02 wt %) which is phenolic thermosetting powder is added to the mixture. The purpose of binder used is to bind nickel powder and graphite powder together. **Figure 4.9** below shows the image of phenolic thermosetting powder. In this case, 2.02 wt% binder is used for a pallet weighted at 5g. The percentage of the binder is shown in calculation below:

Weight of Nickel = 4.996g

Weight of Carbon = 0.012g

Weight of Phenolic = 0.103g

Total weight of Ni-C mixture + Phenolic = 5.111g

$$\% \text{ Phenolic} = \frac{\text{Weight of Phenolic}}{\text{Total Ni-C mixture + Phenolic}} \times 100\%$$

$$= \frac{0.103}{5.111} \times 100\%$$

$$= 2.02\%$$

Figure 4.8: Calculation in producing 2.02wt% binder

Figure 4.9: Phenolic Thermosetting Powder

4.4 Compaction

Compaction is a process where the blended powders are pressed into shapes in dies using presses either hydraulically or mechanically actuated. The as-pressed powder is known as a green compact (see **Figure 4.10**). The purposes of compaction are:

- obtain the required shape
- obtain density
- obtain particle-to-particle contact
- to make the part strong enough to be processed further

Figure 4.10: Compacted powder (green compact) in pallet form

The student has completed the compaction process by producing 6 pallets with different compaction forces acted on them for 2 minutes (compression time). Each different force gives different dimension to each pallet. The student has used Vanier Calliper to measure the dimension (see **Figure 4.11**).The details of dimension of each pallet are described in **Table 4.2** below.

Table 4.2: Dimension of pallets after compaction

Pallet No.	Compaction Force (N)	Dimension	
		Diameter, d (cm)	Height, h (cm)
1	44482.22	1.31	0.62
2	53378.66	1.31	0.58
3	62275.10	1.31	0.57
4	71171.55	1.31	0.57
5	80067.99	1.30	0.56
6	88964.43	1.30	0.56

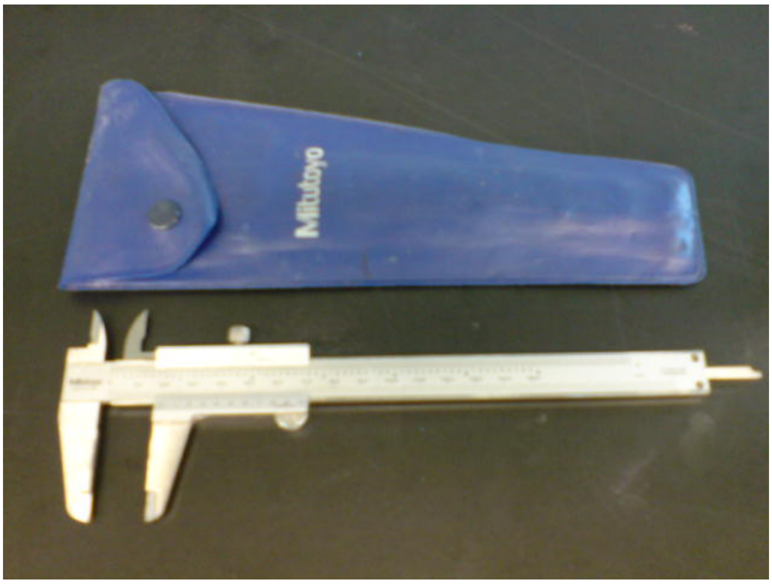


Figure 4.11: Vanier Calliper

From table above, the student concluded that the green density for each pallet can be achieved. This is done by using simple formula **ρ = m/v**, where ρ is density, m is mass of pallet and v is the volume of the pallet. The mass of pallet can be obtained by using balancing beam whilst volume of pallet can be obtained by calculation, π x (d/2) x (h). The green density of each pallet is described in **Table 4.3** below.

Table 4.3: Green density of the compacted pallets

Pallet No.	**Mass, m (g)**	**Volume, v (cm^3)**	**Green Density , ρ (g/cm^3)**
1	5.05	0.83	6.06
2	4.97	0.79	6.31
3	4.98	0.77	6.50
4	4.99	0.76	6.61
5	5.01	0.75	6.66
6	5.01	0.75	6.66

As observed from Table 4.3, the student concluded that the pallet starts to have constant density at force 80067.99N. This is because Pallet 5 and Pallet 6 approximately have same density. From here, the student established a graph of Green Density vs Pressure (see **Figure 4.12**).

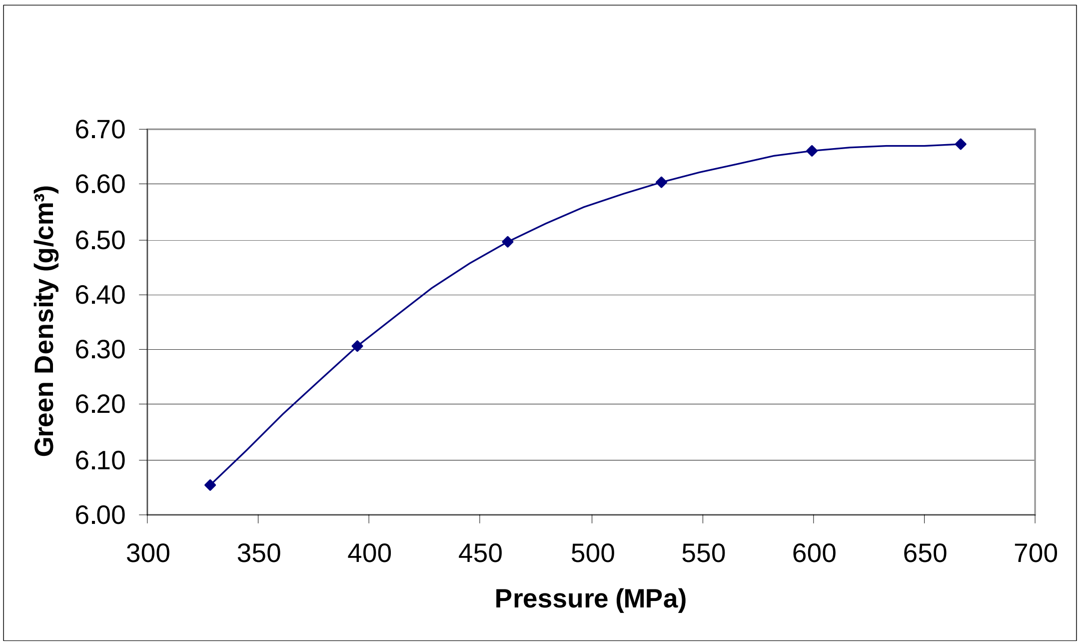


Figure 4.12: Green Density vs Pressure

Besides, the student has determined the microstructure image of the green compact using SEM as shown in **Figure 4.13** below. The report of green compact using EDX can be referred in **Appendix A (Figure A5).**

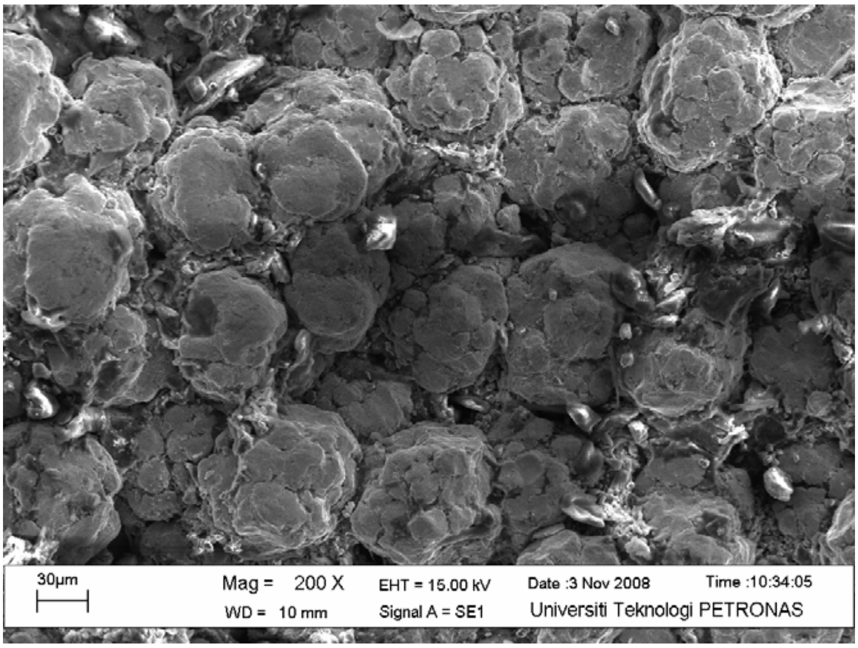


Figure 4.13: Ni-C (green compact) at 200X zoom

Instead of using SEM, the student also uses Quadrant Backscattered Electron Detector (QBSD). This device operates by emitting electron in the gaseous volume and thus generates additional ionization and avalanche amplification [5]. Below is the image of Ni-C (green compact) that uses QBSD. The dark color shows the existence of carbon in the compacted pallet. As refer to the image below, the carbon powder coated the nickel powder and also exists in the middle of the nickel to nickel contact.

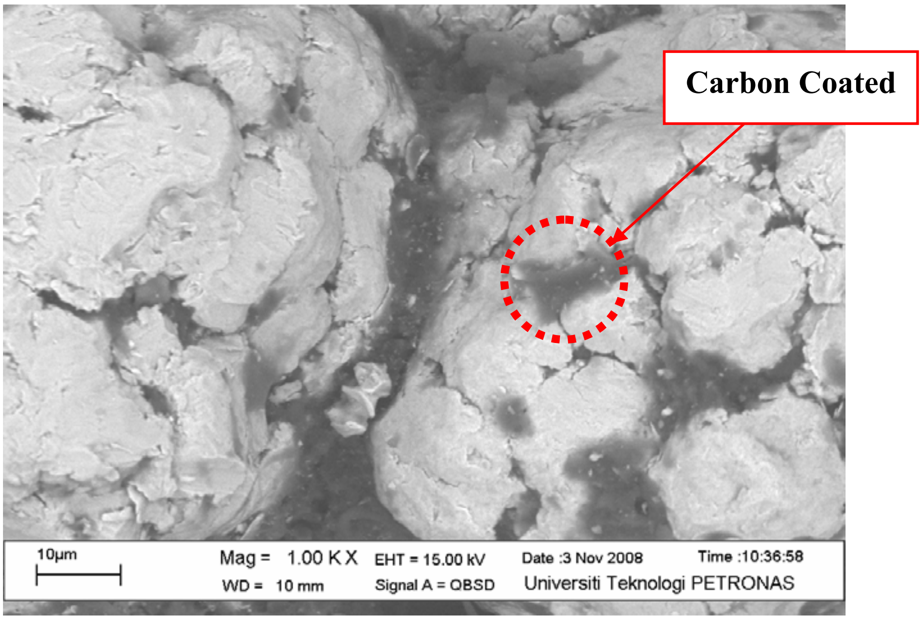


Figure 4.14: Ni-C (green compact) at 1000X zoom using QBSD

4.5 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. In this project, the green compact is heated at temperature 1010^{o}C for 30 minutes. The temperature is set according to **Table 4.4** below.

Table 4.4: Typical sintering temperature and time of nickel alloys and steels [3]

Material	**Temperature**		**Time (minute)**
	^{o}C	**^{o}F**	
Bronze	760-870	1400-1600	10-20
Copper	840-900	1550-1650	12-45
Brass	840-900	1550-1650	30-45
Iron	1010-1150	1850-2100	30-45
Nickel	1010-1150	1850-2100	30-45
Stainless Steel	1095-1285	2000-2350	30-60

After 30 minutes, the pallet is being cooled down in the furnace for 4 hours as the temperature of the furnace decreases naturally. The cooling process takes long time because to avoid crack to the pallet and to have smooth surface finish. **Figure 4.15** below shows the sintered pallet produced.

Figure 4.15: Sintered pallet

The densities of the sintered specimens were determined using Archimedes' principle: the specimens were weighed in air, weighed after coating with a lacquer to seal the porosity; and finally weighed when suspended in distilled water. **Table 4.5** below shows the mass measured prior to different conditions.

Table 4.5: Mass measured prior to different conditions

Pallet No.	Mass in air (g)	Mass with lacquer coating (g)	Mass when suspended in distilled water (g)
1	5.17	5.44	4.74
2	5.16	5.43	4.73
3	5.15	5.42	4.72
4	4.89	5.17	4.47
5	4.83	5.10	4.44
6	4.83	5.10	4.44

As sintered densities (ρ) were calculated in g/cm^3 using the folowing relationship:

$$\text{Density, } \rho = \frac{A}{A-B} \times (\rho_0 - \rho_L) + \rho_L$$

Where:
ρ = density of sample
A = mass of sample coated in air
B = mass of sample coated in distilled water
ρ_0= density of distilled water (0.99777 g/cm^3)
ρ_L= air density (0.0012 g/cm^3)

By inserting the mass of sample coated in air (A) and mass of sample coated in distilled water (B) into the equation above, the results of the sintered densities of each pallet are described in **Table 4.6** below.

Table 4.6: Sintered densities of the pallets

Pallet No.	Sintered Densities , ρ (g/cm^3)
1	6.97
2	7.14
3	7.26
4	7.35
5	7.41
6	7.43

As observed from table above, the student concluded that the pallet starts to have constant density at force 80067.99N (Pallet 5). This is because Pallet 5 and Pallet 6 approximately have same density. From here, the student established a graph of Sintered Density vs Pressure (see **Figure 4.16**).

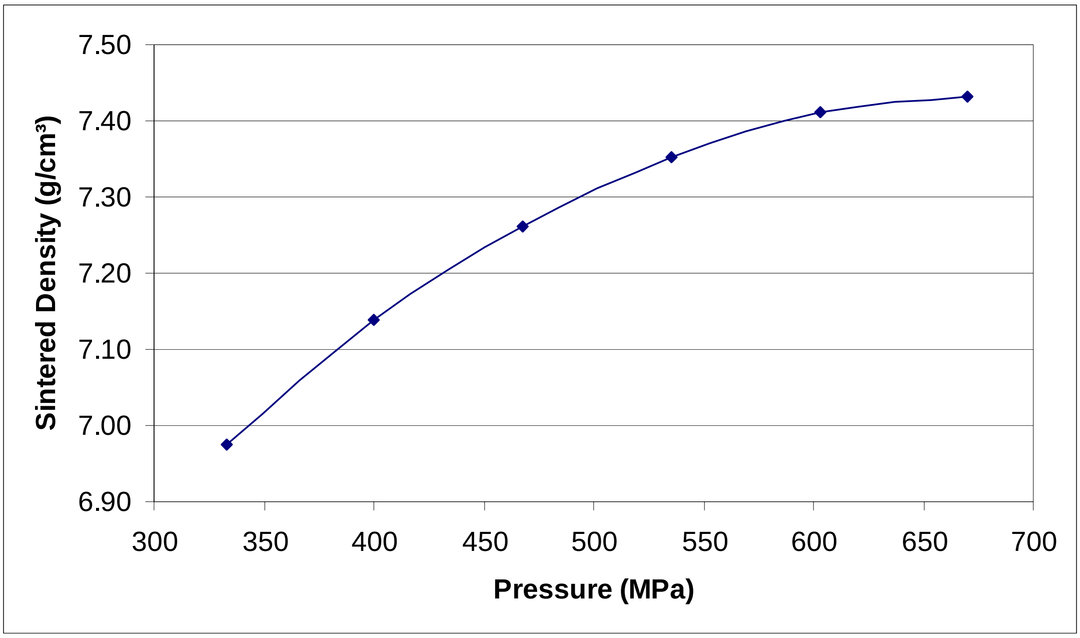


Figure 4.16: Sintered Density vs Pressure

Besides, the student has determined the microstructure image of the sintered mixture using SEM as shown in **Figure 4.17** below. Furthermore, the student has performed QBSD to the sintered pallet as well. The QBSD image is shown in **Figure 4.18**.

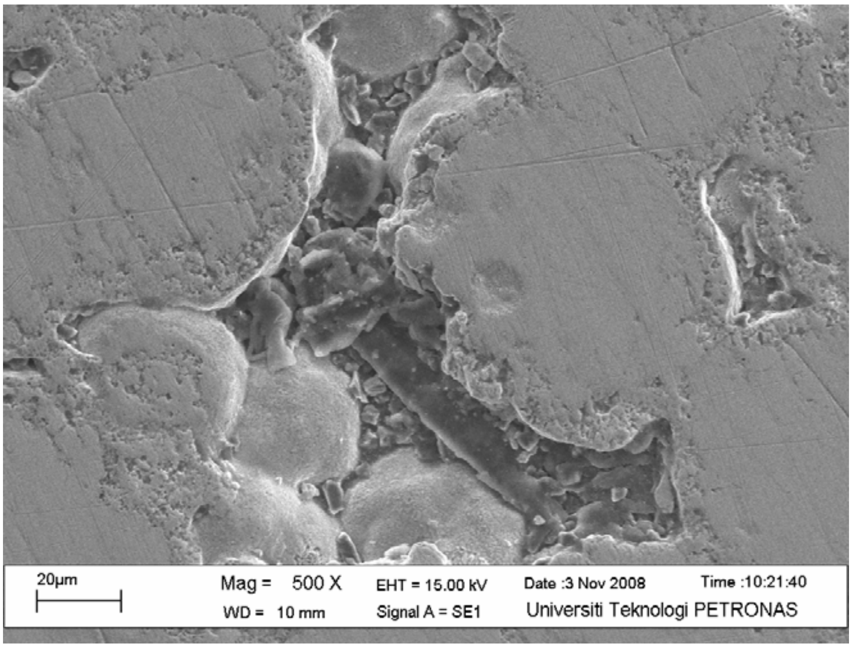


Figure 4.17: Ni-C (sintered mixture) at 500X zoom

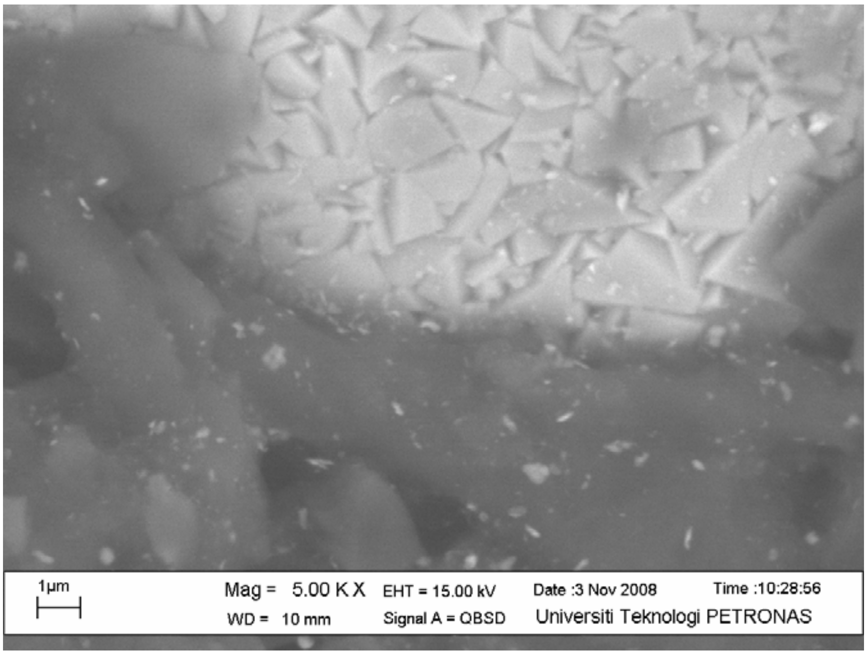


Figure 4.18: Ni-C (sintered mixture) at 5000X zoom using QBSD

After completed the 3 main processes in P/M techniques, the pallets are to be tested under hardness testing machine. The results and outcomes of the samples will be discussed under sub topic 4.6 (Hardness Test).

Figure 4.19: Ni-C sintered pallet ready to be tested

4.6 Hardness Test

Hardness of every pallet has been measured by using hardness machine. Rockwell hardness with B scale that uses ball indenter has been chosen. The load applied to the pallet is 100 kgf. **Figure 4.20** below shows the hardness machine and its measurement that has been used by the student. In performing hardness measurement, the distance between each point of hardness test should be taken into consideration. According to standard, the distance between the measured points should be three times more than the measured point diameter in order to get the accurate value of hardness.

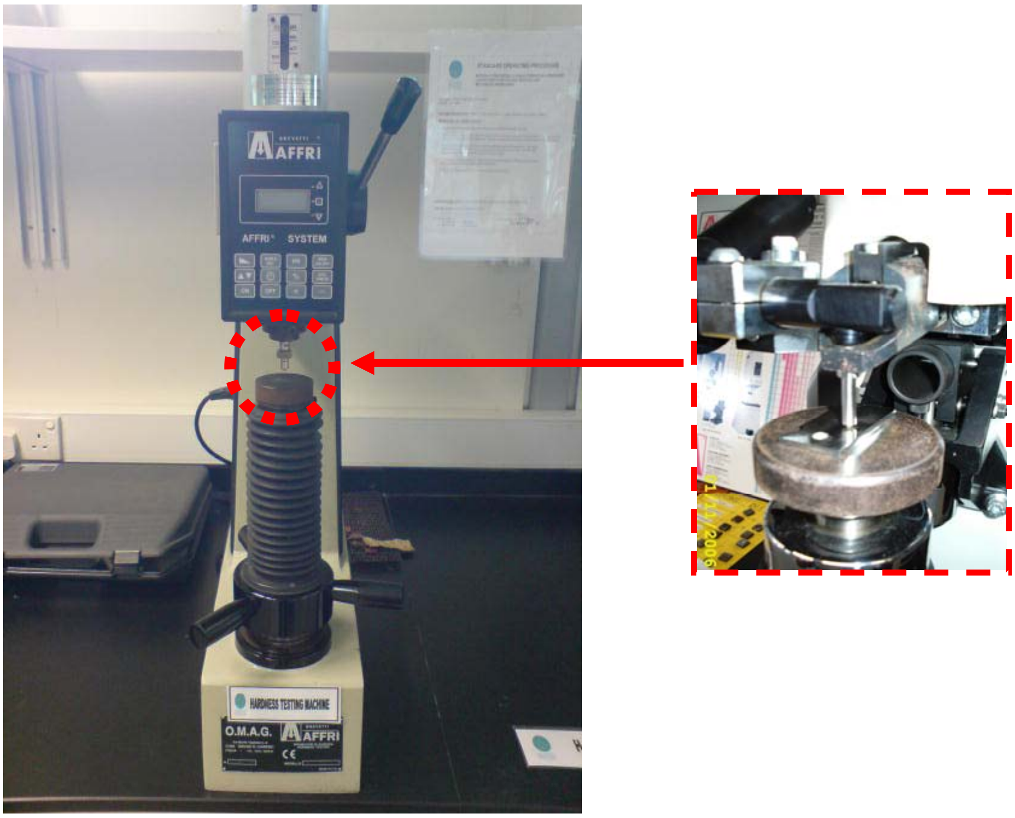


Figure 4.20: Hardness Testing Machine and ball indenter

Table 4.7 shows the result obtained from the hardness measurement using Rockwell Hardness Test. The strength of the sample pallet increases with the increasing of force exerted during compaction. As the force becomes greater, the particle to particle contact between atoms will become great as well. Thus, it gives a better homogeneity to the additives and base particle distribution. However, the reading for pallet number 6 is much preferable because it has reached steady state. Pallet number 5 has reached slightly steady state but for safety factor, the student chose pallet 6 (refer to graph in previous pages). The hardness machine gives a different value at a different point. The point with high hardness value is due to the present of the additives.

Table 4.7: Hardness Test Reading

Pallet No.	**Compaction Force (N)**	**Rockwell Hardness Scale B (HRB)**		
		1	**2**	**3**
1	44482.22	9.31	8.04	9.26
2	53378.66	26.73	27.71	29.58
3	62275.10	25.94	26.81	29.57
4	71171.55	33.22	40.76	32.86
5	80067.99	34.68	41.24	38.41
6	88964.43	34.46	41.45	39.62

CHAPTER 5

CONCLUSION

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

Compressibility of the mixture increased with pressure and become constant at 600MPa. The densities of the compacts were found increases after sintering process. The highest densities of green and sintered were 6.66 g/cm3 and 7.43 g/cm3 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 as well with temperature and pressure until the sintering plateau is reached. The highest hardness of the mixture was 41.45HRB.

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11. http://garrityhvac.com/Furnace.jpg (27/11/08)

APPENDICES

Appendix A

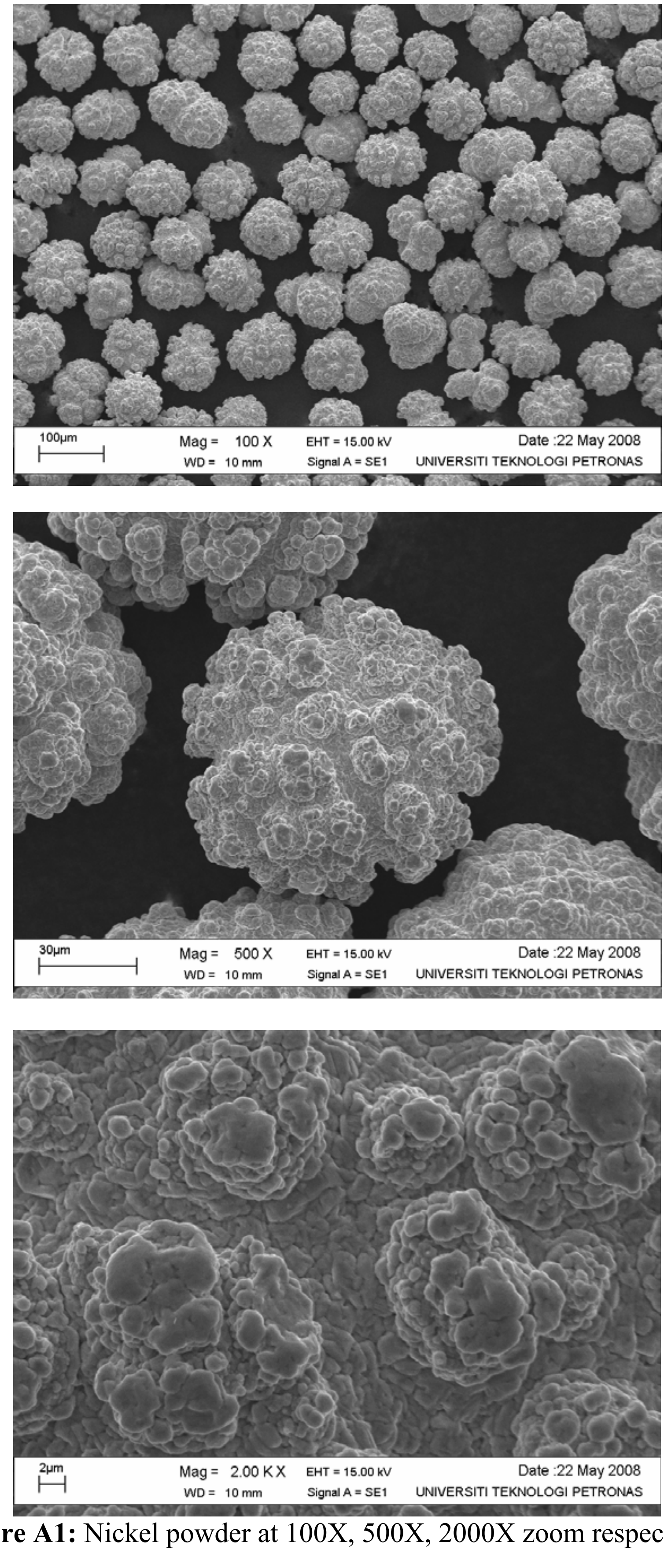


Figure A1: Nickel powder at 100X, 500X, 2000X zoom respectively

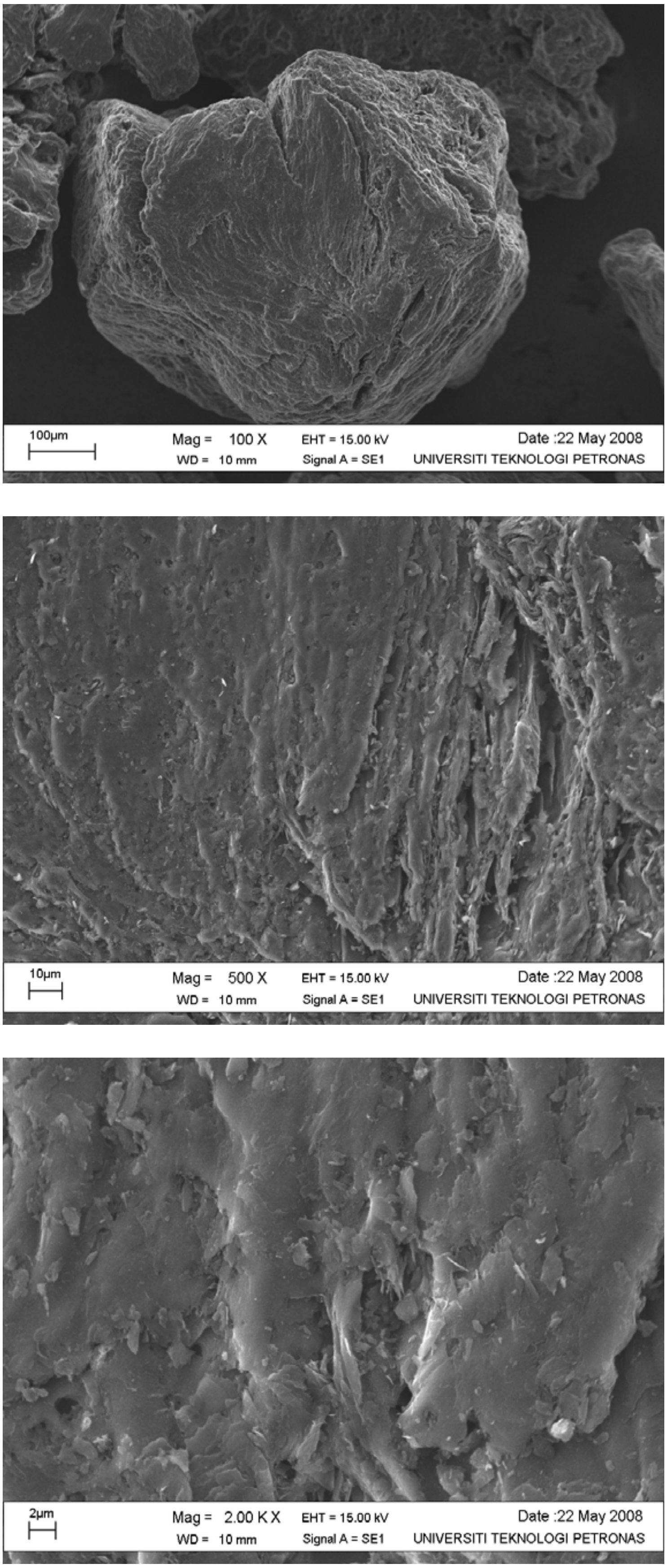


Figure A2: Graphite powder at 100X, 500X, 2000X zoom respectively

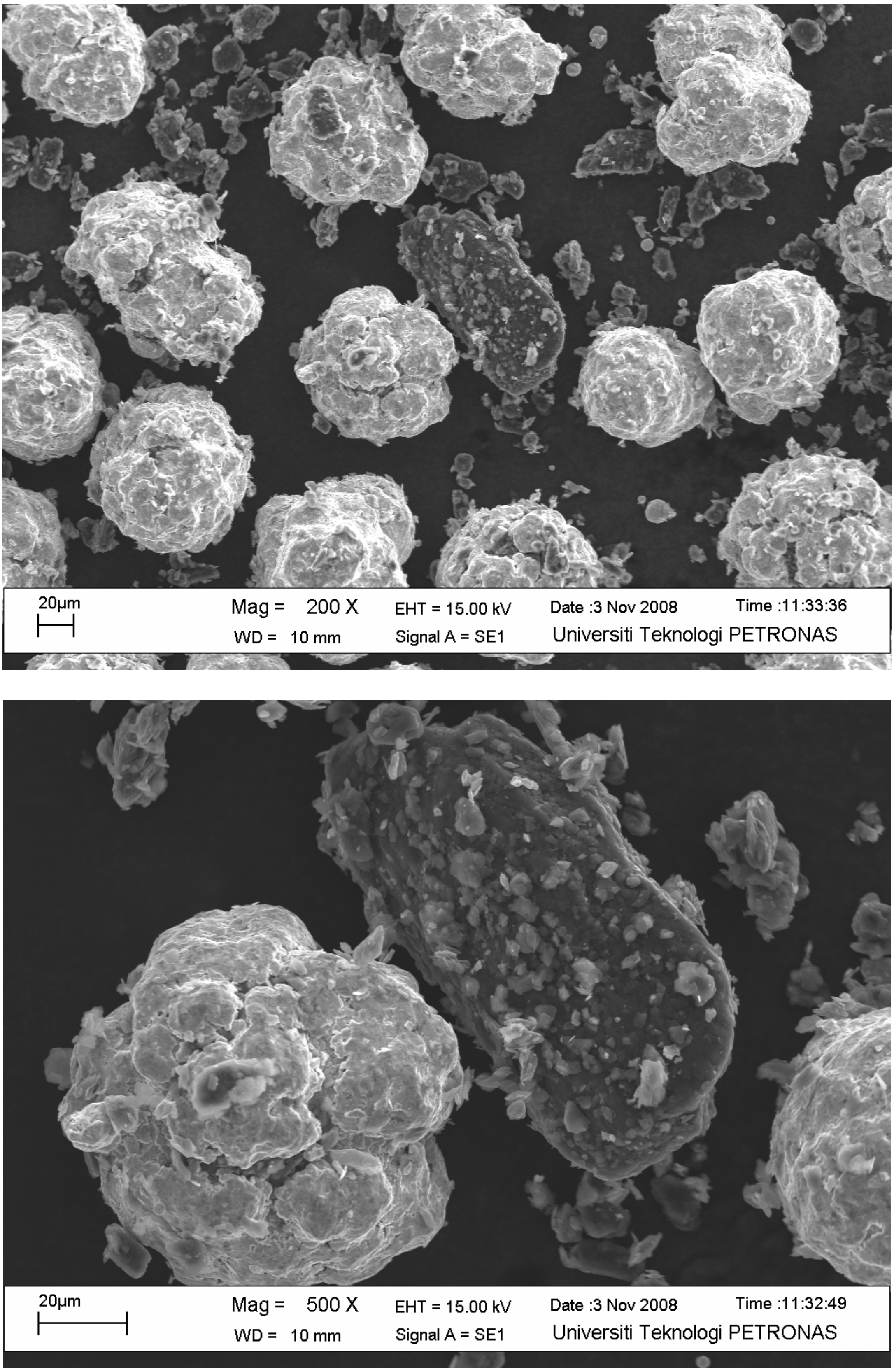


Figure A3: Ni-C mixture at 200X and 500X zoom respectively

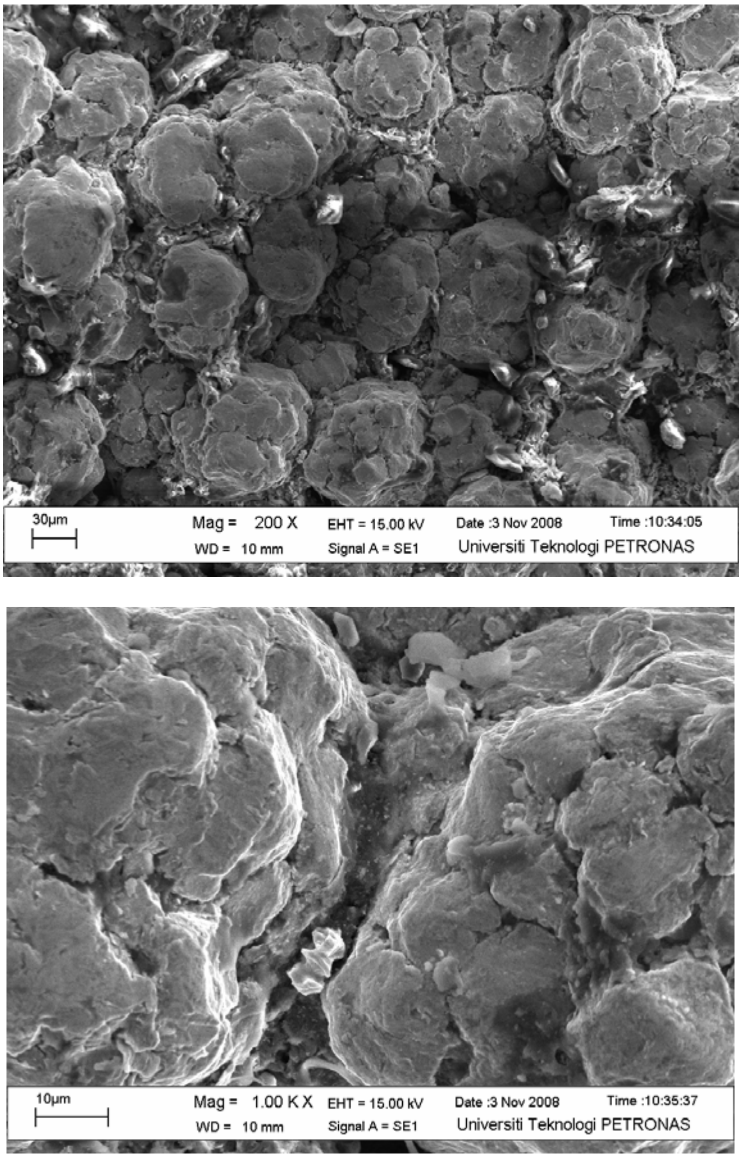


Figure A4: Ni-C (green compact) at 200X, 1000X and 5000X zoom respectively

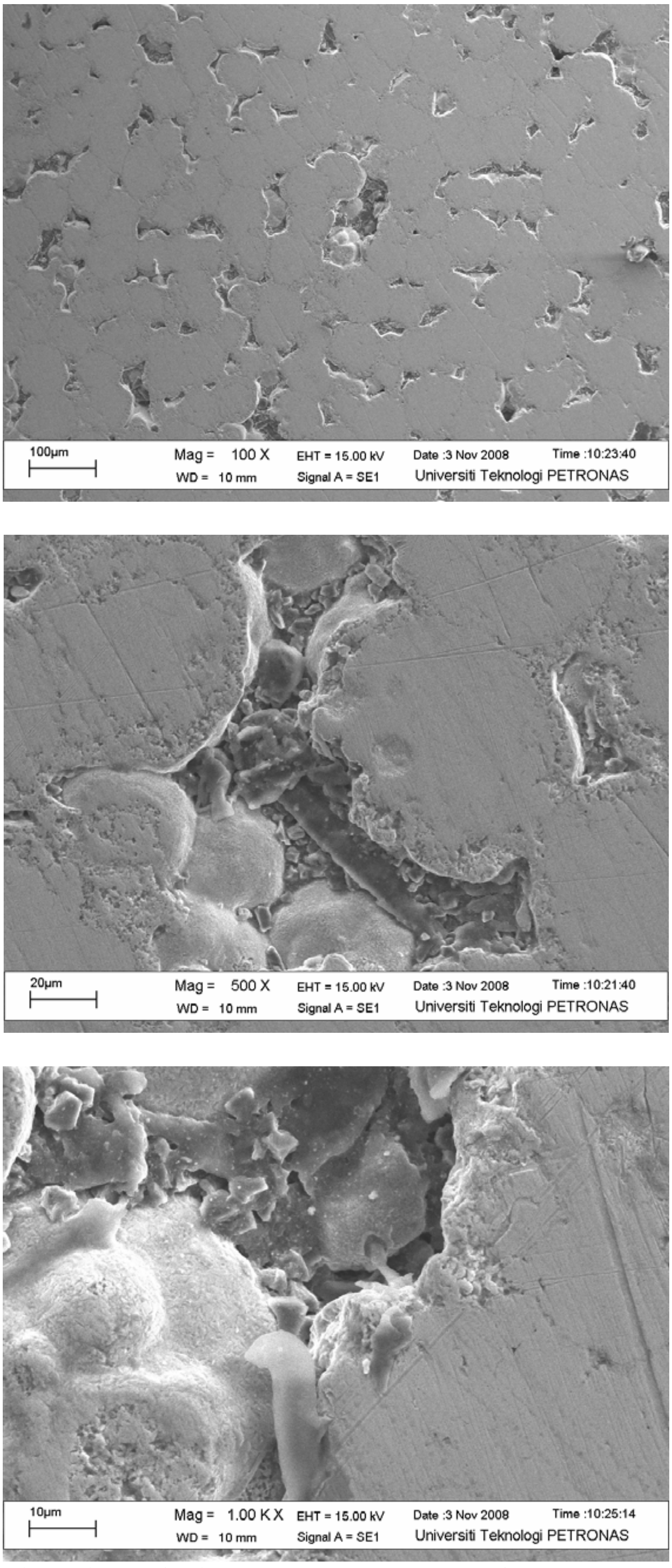
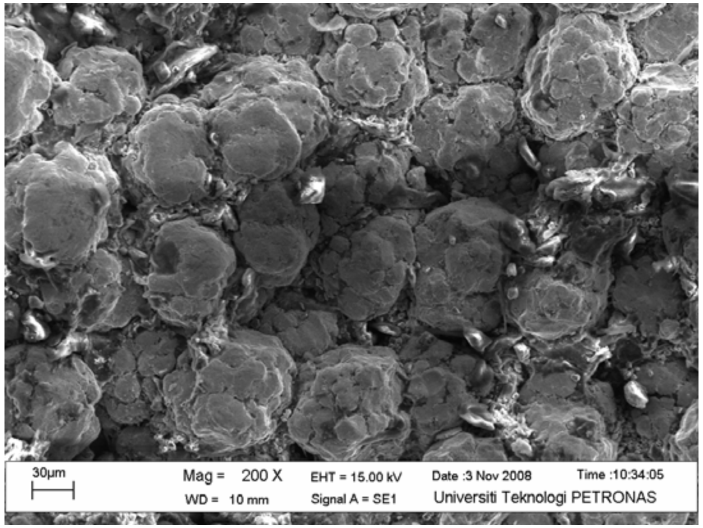


Figure A5: Ni-C (sintered mixture) at 100X, 500X and 1000X zoom respectively

Figure A6: EDX report on Ni-C green compact at 200x zoom



Spectrum processing:
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 4

Standard:
C CaCO3 1-Jun-1999 12:00 AM
O SiO2 1-Jun-1999 12:00 AM
Ni Ni 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	25.27	56.68
O K	7.38	12.43
Ni K	67.35	30.90
Totals	100.00	

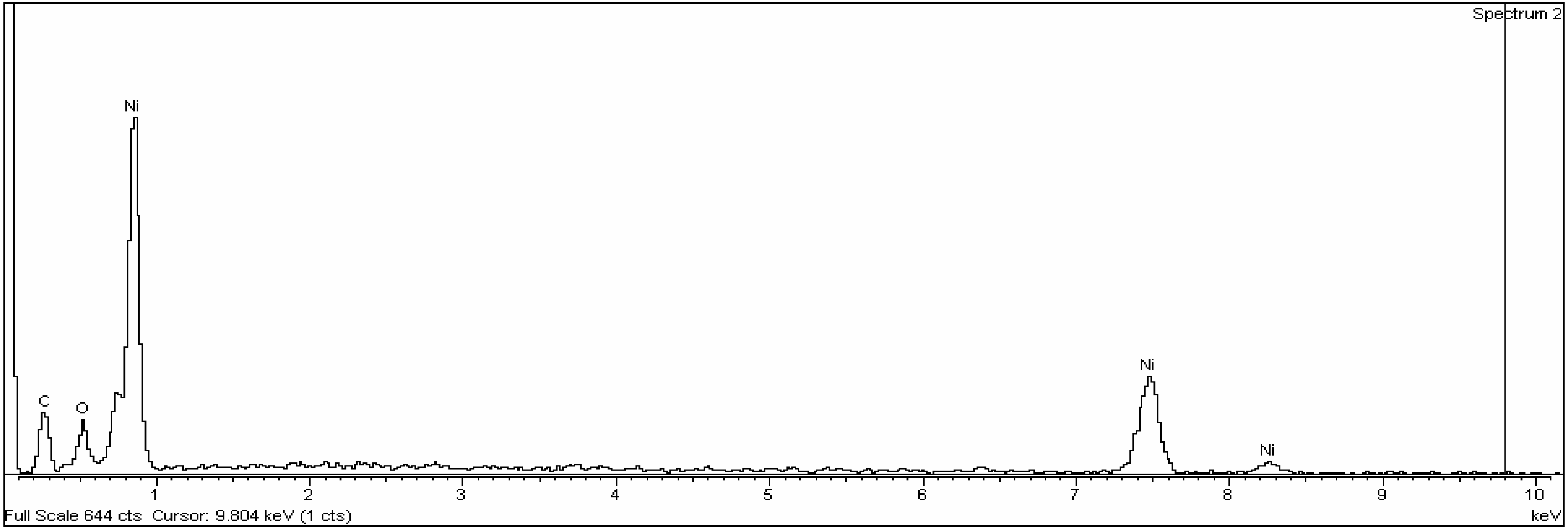


Table A: Gantt chart for July 2008

No.	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Project Work Continue	🟪	🟪	🟪												
2	Submission of Progress Report 1				●											
3	Project Work Continue				🟪	🟪	🟪	🟪								
4	Submission of Progress Report 2									●						
5	Seminar (compulsory)									●						
5	Project work continue								🟪	🟪	🟪	🟪	🟪			
6	Poster Exhibition											●				
7	Submission of Dissertation (soft bound)													●		
8	Oral Presentation														●	
9	Submission of Project Dissertation (Hard Bound)															●

● Suggested milestone

🟪 Process

Appendix B

General Information	
Form: Color: Odor:	Powder Silver-colored Odorless
Change in condition Melting point / Melting range: Boiling point / Boiling range: Sublimation temperature / start:	 1453°C (2647°F) 2730°C (4946°F) 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):	8.908 g/cm^3
Solubility in / Miscibility with water:	Insoluble

Table B1: Physical and chemical properties of nickel powder stock number 10579 based on Material Safety Data Sheet (MSDS).

General Information	
Form: Color: Odor:	Solid Black Odorless
Change in condition	
Melting point / Melting range:	3652 - 3697°C (6606 - 6687°F)
Boiling point / Boiling range:	Not determined
Sublimation temperature / start:	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:	Not determined
Upper:	Not determined
Vapor pressure:	Not determined
Density at 20°C (68°F):	2.25 g/cm^3
Solubility in / Miscibility with water:	Insoluble

Table B2: Physical and chemical properties of graphite powder stock number 10131 based on Material Safety Data Sheet (MSDS).