Study on the Effect of Annealing Temperature on the Compressibility and Microscopic Structure of Pure Graphite

by

Mohamad Anas Najmi Bin Abdul Razak

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

AUGUST 2011

Universiti Teknologi PETRONAS
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CERTIFICATION OF APPROVAL

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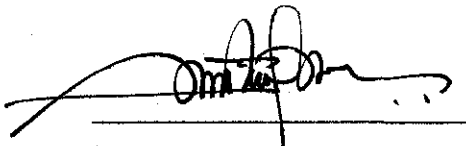
Approved by,



(AP Dr. Othman Bin Mamat)

UNIVERSITI TEKNOLOGI PETRONAS
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AUGUST 2011

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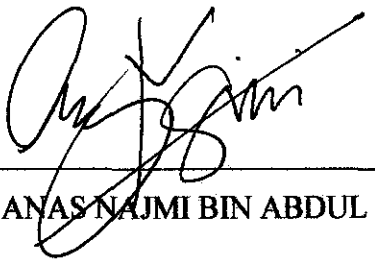

MOHAMAD ANAS NAJMI BIN ABDUL RAZAK

ABSTRACT

The problem of improving the physical and mechanical properties of graphite is not receiving considerable attention despite graphite being used widely in critical and dangerous applications in many industries such as in nuclear reactors, automotive components, rocket nozzles and high temperature heat exchangers. Studies have indicated that, if a metal powder is annealed, the compressibility of the powder will improve. Therefore, it is the purpose of this report to investigate and establish an extensive study to improve the compressibility of graphite powder, one of the many mechanical properties of graphite. The objective of this report is to study on the effect of annealing temperature of pure graphite powder on the compressibility and microscopic structure of its pellet produced by powder metallurgy process. The graphite powder will undergo powder metallurgy processes in order to produce the pellets; grinding, annealing and compacting. Four sets of samples comprising of 10 pellets each will be produced.1 set will be left unannealed as the datum, and the other 3 set of samples will be annealed at three different annealing temperatures; 700°C, 800°C and 900°C. The pellets will be compressed using compaction pressure between 14-140 MPa (2000psi-20000psi). Next, the density of all the pellets will be measured and compressibility curves comparing the compressibility of all set of samples will be plotted based on the densities recorded. After that, the microstructure of pellets compressed at 137.90 MPa (20000psi) at all annealing temperature (unannealed, 700°C, 800°C and 900°C) will be analyzed using SEM and recorded. The analysis carried out to the compressibility curve of the samples shows that, the compressibility of the samples increase as the annealing temperature increases. The microstructures of the samples indicated that necking and bridging structures formed vigorously as the annealing temperature increases. As a conclusion, the compressibility of graphite increases as its annealing temperature increases.

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CHAPTER 1
INTRODUCTION

This section will provide some background information of the project, discuss on the problem statement that leads to the establishment of the project and put forward the objective and scope of study of the project.

1.1 Project Background

Graphite uses have changed dramatically in the past 20 years. United States reported consumption of natural graphite increased by 27% to 49,800 tonne in 2008 from 39,200 tonne in 2007 (Olson, 2008, p. 32.1). Olson (2008) also reported that the four industries that dominated U.S. natural graphite use are brake linings, foundries, refractories and steelmaking., with brake lining, foundries, lubricants and refractories accounted for 42% of natural graphite consumption, while the production of batteries and pencils together made up another 3% of consumption. Table 1.1 summarized the reported natural graphite consumption by end use.

According to Olson (2008),

> Graphite has properties of both metals and non-metals, which makes it suitable for many industrial applications. The metallic properties include electrical and thermal conductivity. The non-metallic properties include high-thermal resistance, inertness, and lubricity. The combination of conductivity and high-thermal stability allows graphite to be used in many applications, such as in batteries, fuel cells, and refractories. Graphite's lubricity and thermal conductivity make it an excellent material for high-temperature applications because it provides effective lubrication at a friction interface while furnishing a thermally conductive matrix to remove heat from the same interface. Electrical conductivity and lubricity allow its use as the primary material in the manufacture of brushes for electric motors. A graphite brush effectively

transfers electric current to a rotating armature while the natural lubricity of the brush minimizes frictional wear. Today's advanced technology products, such as friction materials and battery and fuel cells, require high-purity graphite. Natural graphite is purified to 99.9% carbon content for use in battery applications.

Table 1.1: U.S. Reported Consumption of Natural Graphite, by End Use

End use	Crystalline		Amorphous[2]		Total	
	Quantity (metric tons)	Value (thousands)	Quantity (metric tons)	Value (thousands)	Quantity (metric tons)	Value (thousands)
2007:						
Batteries	W	W	--	--	W	W
Brake linings	489	929	4,050	3,100	4,540	4,030
Carbon products[3]	284	775	W	326	W	1,100
Crucibles, retorts, stoppers, sleeves, nozzles	W	W	W	W	W	W
Foundries[4]	W	W	474	323	W	W
Lubricants[5]	624	774	W	W	W	W
Pencils	W	W	W	W	W	W
Powdered metals	258	433	W	W	W	W
Refractories	6,490	3,190	W	W	W	W
Rubber	W	W	W	W	W	W
Steelmaking	W	W	W	W	W	7,410
Other[6]	8,820	9,660	2,150	2,410	11,000	12,100
Total	18,600	18,200	20,500	17,900	39,200	36,100
2008:						
Batteries	W	W	--	--	W	W
Brake linings	515	2,260	3,070	W	3,590	W
Carbon products[3]	266	693	W	W	W	W
Crucibles, retorts, stoppers, sleeves, nozzles	W	W	W	W	W	W
Foundries[4]	W	W	3,600	13,100	W	W
Lubricants[5]	844	2,910	W	W	W	W
Pencils	W	W	W	W	W	W
Powdered metals	561	1,770	4	8	565	1,780
Refractories	7,210	6,280	W	W	W	W
Rubber	24	W	W	W	W	W
Steelmaking	W	W	W	W	W	W
Other[6]	8,740	12,600	3,390	19,900	12,100	32,400
Total	20,200	30,900	29,600	125,000	49,800	156,000

W Withheld to avoid disclosing company proprietary data; included in "Total." -- Zero.

(Extracted from Olson, 2008, p. 32.4)

However, due to its structure, graphite possesses relatively low mechanical strength. For instance, graphite is a very soft material, having a Mohs hardness of 1-2 (Graphite-Mineral Properties and Uses, 2005) and bulk density ranging from 1.3-1.95 g/cm^3 (Graphite – Classification, Properties and Applications of Graphite, 2002). Therefore, the project will focus on improving the mechanical strength of graphite by varying the

annealing temperature for the graphite powder heat treatment to improve its compressibility.

1.2 Problem Statement

The growing interest in carbon nanostructure and their unique properties has led to many extensive studies on the mechanical processing involved, such as ball milling of graphite powder. Graphite, apart from its traditional used in the brake lining, foundries, refractories and steelmaking industries (Olson, 2008, p. 32.1), are vastly used to manufacture many components in recent technologies such as moderators and structural materials for nuclear reactors, automotive components, rocket nozzles and high temperature heat exchangers (Carreira, Sánchez-Coronado, Narciso, Martínez-Escandell, Rodríguez-Reinoso, 2008, p. 1)

Despite having numerous applications, graphite has low mechanical strength. According to Carreira et al. (2008), carbon materials such as graphite are claimed to possess certain disadvantages such as low mechanical strength and low degree of oxidation resistance at high temperature (p. 1). This is a source of concern as graphite is widely used in various industries; some of them are high-risk and potentially dangerous applications.

Various research papers presented generally focus on the improvement of electrical and thermal conductivity of graphite. One of the factors affecting the mechanical strength of graphite is the compressibility of its powder. Compressibility of a powder can be improved by annealing the powder for heat treatment purpose before the powder undergoes further processing. However, study of the effect of annealing temperature to the compressibility of graphite is not well established although it is very necessary to improve the mechanical strength of graphite. Papers on this study are scarce and very rare to find. Hence, it is the purpose of this research to establish an extensive study in this area.

1.3 Objective and Scope of Study

The objective of this research is to study on the effect of annealing temperature of pure graphite powder on the compressibility and microscopic structure of its pellet produced by powder metallurgy process. The specimens will undergo several powder metallurgy processes such as annealing, grinding and compacting. In order to meet the objective, this project will concentrate on varying the annealing temperature of the graphite powder and analyze its effect on the green density of the graphite pellets. This study aims to record the green density of the pellet for each annealing temperature and develop compressibility curves based on the data collected. Then, the microstructure of the specimens for each annealing temperature will be analyzed and compared. This project will be conducted in two semester's time (2010/2011).

Certain boundaries need to be defined so that this project will be time-feasible and achievable. The project is expected to take much of its time in reducing the size of graphite powder to a smaller, more appropriate size as the graphite powder initially received has a larger size and thus impossible to compact it to pellet form.

The scopes of study for this project are as follow:

Production of Graphite Pellets

Samples of the graphite pellets will be produced by powder metallurgy processes. The equipments which will be used in this project are as follow:

- Grinding: US Stoneware Ball Milling Machine, Mortar Grinder, Sieve Shaker
- Compaction: Carver Autopellet Press Machine, USA
- Annealing: Tube Furnace

Graphite Powder Details

This project will use graphite powder having the following characteristics:

- Origin/Company: Alfa Aesar
- Size: -20+84 mesh (177-841μm)
- Type: Course flake graphite
- Purity: 99.9%

Physical/Mechanical Tests Details

To ensure the project is achievable in two semester's time, only two tests will be conducted. The tests and the machine/method used are:

- Green Density Measurement: Mettler Toledo AX205 Density Measurement by Archimedes' Method
- Microscopic Structure Analysis: Scanning Electron Microscope (SEM)

CHAPTER 2
LITERATURE REVIEW

2.1 Graphite

According to Olson (2008),
Graphite is one of four forms of crystalline carbon; the others are carbon nanotubes, diamonds and fullerenes. Graphite is gray to black in color, opaque and usually has a metallic luster; sometimes it exhibits a dull earthly luster. Graphite occurs naturally in metamorphic rocks. It is a soft mineral with a Mohs hardness of 1-2, and it exhibits perfect basal (one-plane) cleavage. Graphite is flexible but not elastic, has a melting point of 3,297°C, and is highly refractory. It has a low specific gravity. Graphite is the most electrically and thermally conductive of the nonmetals and is chemically inert. (p. 32.1)

Graphite is the stable form of carbon at Earth surface pressure and temperature. In fact, all of the crystalline forms of carbon (diamonds, carbon nanotubes and fullerenes) are undergoing transformation into graphite at Earth surface. However, the process is extremely slow (Graphite, 1995).

Table 2.1 shows the physical properties of ideal graphite. Such material does not exist in the real world, and most graphite will have physical properties well below the ideal data. As an example, commercially, the density of graphite is between 1.3-1.95 g/cm^3 (Pierson, 1993, p.51).

From Table 2.1, graphite can be categorized as a very soft material. Yet, graphite is widely used as an additive in high-strength composite used to build aircraft, spaceship, nuclear reactor and golf club shafts. The reason why graphite can be used to strengthen other material can be explain by its unique crystal structure.

Table 2.1: Physical Properties of Graphite (Pierson, 1993, p.51)

Properties	Value
Crystalline Form	Hexagonal
Lattice Parameter	$a_o = 0.246$ nm $c_o = 0.671$nm
Color	Black
Density at 300K, 1 atm	2.26 g/cm^3
Mohs Hardness	1-2
Atomic Volume	5.315 cm^3/mol
Sublimation Point at 1 atm (estimated)	4000K
Triple Point (estimated)	4200K
Boiling Point (estimated)	4650K

Structurally, graphite is made up of a series of stacked parallel layer planes as shown in Figure 2.1. In actuality, each carbon atoms contact its neighbors. In each layer plane, one carbon atom is bonded to three other carbon atoms. This will form a series of continuous hexagon in an infinite two-dimensional molecule (Pierson, 1993, p. 44-45).

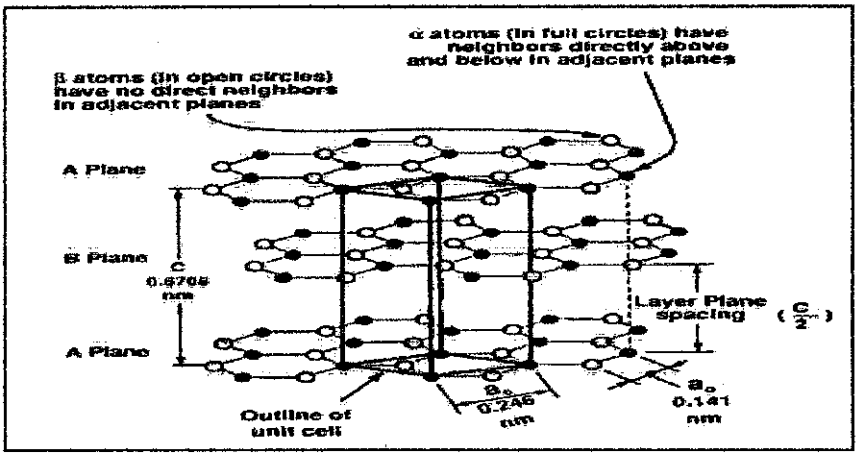

Figure 2.1: Crystal Structure of Graphite Showing ABAB Stacking Sequence and Unit Cell (Pierson, 1993, p.45)

The carbon atoms that lie within a parallel layer plane actually possess a very strong carbon-carbon bond. However, the bond between one layer plane and another layer plane at above and below is very weak. The weakly bonded layer planes tend to slide by each other to yield the slipperiness and softness, thus explaining why graphite is very soft and acts like lubricant. Despite this fact, when the layer planes are rolled up into fibers and those fibers twisted into threads, it will result in a composite having some of the highest strength-to-weight ratios of any materials. It is very advantageous as it is very light and very strong, thus explaining its wide use in aerospace industries (Graphite, 1995).

Table 2.2 summarizes the thermal properties of graphite:

Table 2.2: Theoretical Thermal Properties of Graphite (Pierson, 1993, p.54)

Properties	Value
Standard Entropy S° at 25°C	5.697-5.743 J/mol.K
Entropy ΔS_{298}	152.3 J/mol.K
Enthalpy ΔH_{298}	716.88 kJ/mol
Specific Heat at 25°C	0.690-0.719 kJ/kg.K
Thermal Conductivity at 25°C	ab directions 398 W/m.K c direction 2.2 W/m.k

According to Pierson (1993), graphite can be considered as a semi metal. It is a conductor in the basal plane and an insulator normal to the basal plane. Its electrical resistivity increases with temperature, similar to most metals (p. 61).

Pierson (1993, p. 63-64) also stated that, chemically, pure graphite is one of the most inert materials. It is resistant to most acids, alkaline, and corrosive gases. However, the presence of impurities in graphite often contributes to catalytic effect with resulting increase in chemical reactivity. Other than that, the chemical reactivity is also affected by the degree of porosity, since high porosity yield to large increase in surface area, which results in increasing reactivity. Despite the excellent chemical resistance of

graphite, it actually has poor resistance to the elements of Column VI (oxygen, sulfur, selenium and tellurium). Table 2.3 summarizes the mechanical properties of graphite :

Table 2.3 Mechanical Properties of Graphite (Graphite- Classifications, Properties and Applications of Graphite, 2002)

Properties	Value
Modulus of Elasticity	8-15 GPa
Compressive Strength	20-200 MPa
Flexural Strength	6.9-100 Mpa
Porosity	0.7-53 %

The most interesting properties of graphite is its increased strength as its temperature increases. Graphite reaches its maximum strength at around 4500°C, near to its boiling point.

2.2 Compressibility and Green Density

Compressibility is a term used to define the ability to produce "green" (unsintered) compact from the die pressing of powders. In other words, compressibility is quantified as the amount of compaction pressure needed to obtain a given green density of a specimen. Green density is merely the measured density of a green compact. A more compressible powder can be compacted to a higher density. This will allow the manufacture of a material possessing better mechanical properties (Lampman, 1998, p.302).

Compressibility of a powder can be expressed in terms of green density versus compaction pressure, or simply called compressibility curve. Figure 2.2 shows an example of a compressibility curve.

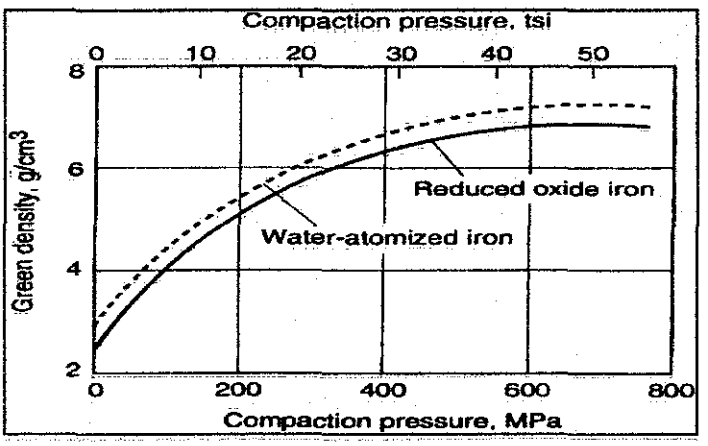

Figure 2.2: Example of a Compressibility Curve (Lampman, 1998, p.302)

According to Lampman (1998), there are several factors which influence the compressibility of a given powders (p. 302-305). They are:

- Particle size and shape
- Powder hardness and oxide
- Internal porosity
- Particle chemistry
- Lubricants
- Tool and powder temperature during compaction
- Annealing of powders

However, the factor is only related to the metal powder processing. Further research need to be carried out to investigate whether these factors apply to non-metal powders such as graphite.

2.3 Effect of Annealing Temperature on Graphite/Graphite Composites Properties

This project will focus on the study of the annealing temperature effect on the compressibility of graphite powder. Annealing temperature is one of the factors that affect the compressibility of a powder. Annealing is a heat treatment process done usually to metal powders before they are compressed in order to remove contaminant

such as oxygen, thin surface oxide films, and nitrogen that can reduce the green strength and compressibility of a powder (Lampman, 1998).

In a recent study made by Zhang et al. (2008), the team found out that the mechanical properties of silicon carbide (SiC) composites correlates with the annealing temperature imposed on the samples. The objectives of their research are: 1) to examine the effectiveness of the Al_2O_3 and La_2O_3 as additives; and 2) to investigate how the annealed temperature influences the microstructure and mechanical properties of SiC composites. With regards to the project objectives, only the second objectives of this research will be discussed.

Initially, they prepared 4 samples of SiC by hot-pressing (HP) sintering with Al_2O_3 and La_2O_3 as additives. These 4 samples were then annealed at 1650°C, 1750°C, 1850°C and 1950°C. Their specific mass was determined using Archimedes method. The microstructures of the samples were observed using XRD and TEM. The relation between flexural strength and flexural toughness with the annealing temperature was recorded, as shown in Figure 2.3.

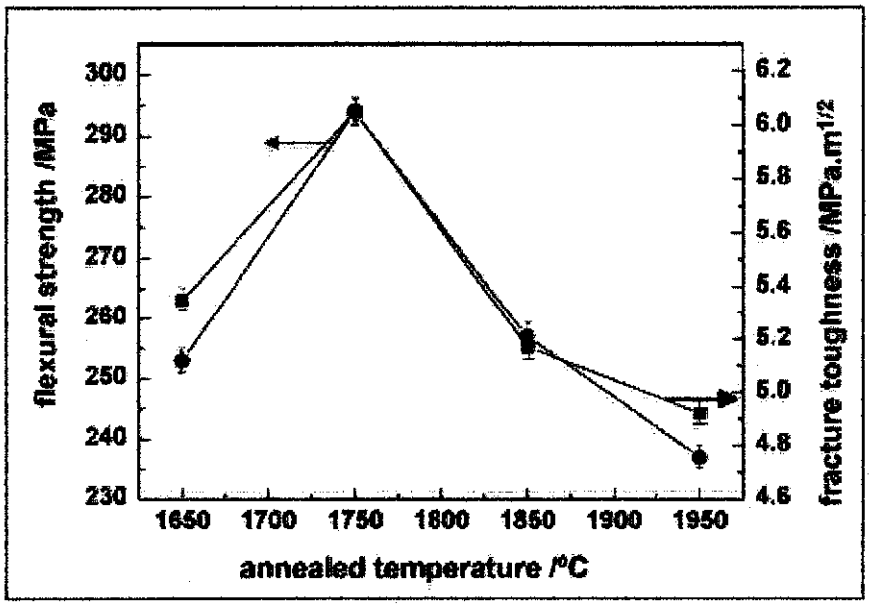

Figure 2.3: Relative Curve between Annealed Temperature and Mechanical Properties of Annealed Sample (Zhang et al., 2008, p.4)

From the data, there is a correlation between annealing temperature and mechanical properties of the SiC samples, although the correlation is not proportional. Annealing treatment had a strong influence on the fracture toughness and flexural strength of the composites. However, above the annealing temperature of 1800°C, mechanical properties reduced slightly. The reason of this decrease, as explained by Zhang et al. (2008) in their journal, is that, due to the high annealing temperature of 1800°C and 1950°C, the carbon fibers in the SiC damaged seriously, and the interface reaction is strong. These, along with the volatility of the second phase, are the main reasons behind the decrease of the mechanical properties.

Compared with other annealed samples, samples annealed at 1750°C have excellent mechanical properties. According to Zhang et al. (2008), at this temperature, the carbon fiber structure in the composite is still kept intact. The interface reaction between the matrix and the fiber is weak. The weak interface resulted in the pullout of carbon fiber, which contributes to the higher fracture toughness. On the other hand, the damage carbon fiber in sample annealed at 1950°C leads to the decrease of mechanical properties. The difference in fracture surface between sample annealed at 1750°C and 1950°C can be seen in Figure 2.4 below.

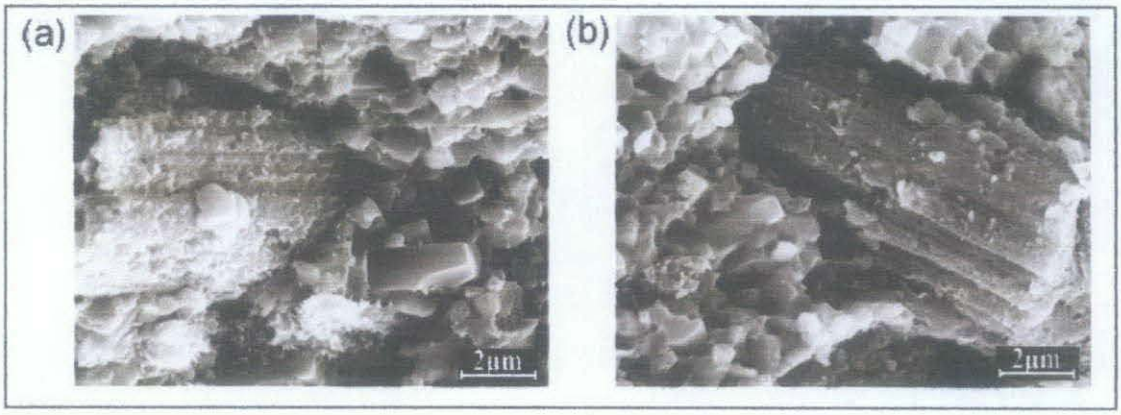

Figure 2.4: Fracture Surface of (a) 1750°C Annealed Sample and (b) 1950°C Annealed Sample (Zhang et al., 2008, p.4)

This journal, despite does not related to the project objective, provide a strong prove that annealing temperature does affect the mechanical properties of carbon-based

composite. The fracture toughness and flexural strength of the SiC increase with increasing temperature up to a certain temperature. However, the effect of annealing temperature to the compressibility of graphite powder needs to be investigated further.

Recent study conducted by Tang et al. (2007) suggested that, by annealing graphite up to a certain temperature and a certain time, recrystallization of graphite occur. The aim of their study is to investigate the structural changes of expanded graphite during milling in air atmosphere and subsequently annealing. However, for the convenience of this project objective, only the effect of the annealing to the microstructure of the graphite will be discussed.

Based on the research, it is demonstrated that annealing reorganizes the carbon structure in milled natural graphite in air atmosphere. Figure 2.5 depicts the XRD pattern of the expanded graphite milled for 16 hours and 30 hours and annealed at different times. Based on the figure, it can be seen that the intensity of (002) diffraction peaks becomes weaker and subsequently stronger again in the annealing process, which proves there are structural reorganization occur during the annealing process.

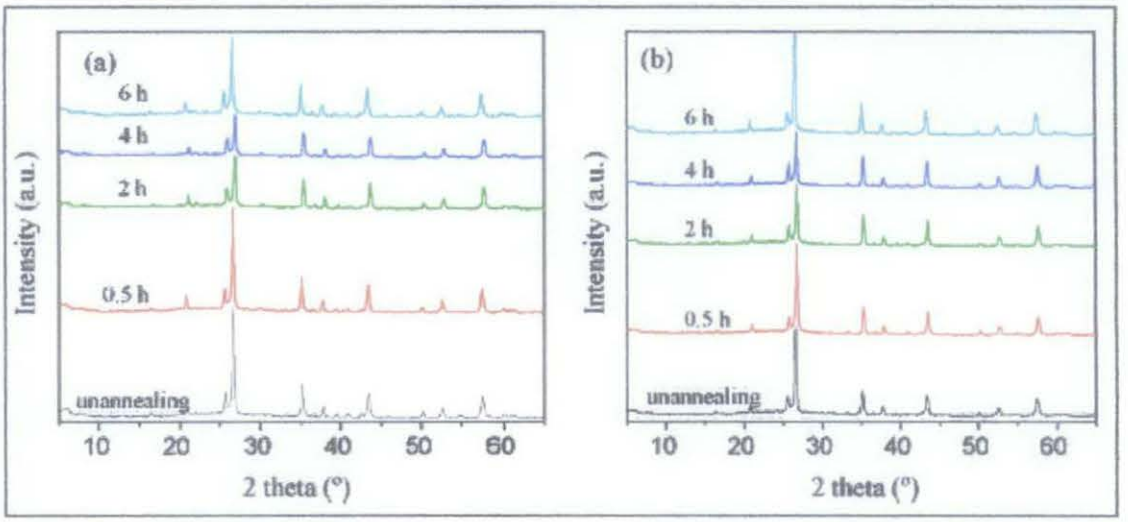

Figure 2.5: XRD patterns of expanded graphite samples milled for 16 hours (a) and 30 hours (b) as a function of annealing time at 600°C, respectively (Tang et al., 2007, p.5)

It was also found out that annealing temperature plays an important influence on the recrystallization of the expanded graphite. As an example, for annealing at 600°C, the annealing recrystallization time occur at 4 hours, while annealing at 900°C occur at 0.5 hour.

In this journal, Tang et al. (2007) conclude that, with increasing annealing time, the defects in plane accumulate, resulting in the turbulence and bending of the basal plane. The disordering of the lattice of the graphite is followed by rapid crystallization occurring in the bulk. Also, Tang et al. stated that higher annealing temperature enhance the course of recrystallization.

Again, the research made by Tang et al. (2007) does not really relate to this project objective. However, the important points from their journal are:

- The preparation of expanded graphite samples using ball milling (metallurgy) technique
- Annealing temperature and time affect the microstructure of the graphite

Since this project will use powder metallurgy process to produce graphite pellets as samples, this research provides a sound supports that graphite powder, despite being a non-metal element, can undergo such process. Other than that, the research uses annealing temperatures that is suitable to the equipment provided in the UTP Material Laboratory (600°C-900°C) that will also affect the microstructure of the graphite, as compared to research made by Zhang et al. (2008) which use higher annealing temperature (1650°C-1950°C) which may render the project unfeasible.

2.4 Powder Metallurgy Process

The experimental works on Tahir et al. (2010) for the characterization and properties of copper-silica sand nano-particles would be a good basis for this project's objective. Although their study does not relate with this project, the methodology and equipments the team used to study the mechanical and physical properties of the copper-silica sand

nano-particles composites can be a good reference for this project in producing the graphite pellets using powder metallurgy process. Their project objective is to study on the effect of nano-silica sand addition towards the hardness and tensile strength of copper-silica composites. The copper-silica composites were produced in pellet form using powder metallurgy method. The processes involved were ball milling, compaction and sintering. They conducted a number of tests too; XRD analysis, FESEM analysis and hardness testing using Rockwell B Scale. From their study, it was observed that, as the percentage of the nano-silica sand particles increased in the composites, the hardness and the tensile strength of the composites increased too.

The journal describes in detail the process and procedure involved in conducting powder metallurgy technique. The procedures can be implemented in order to ball milled, annealed, and pressed graphite powder into pellet form. Besides, they conducted hardness measurement using Rockwell B Scale and measured the green and sintered density of the composites using a Archimedes method; both of these tests can be used to measure the density and hardness of the graphite pellet.

Research made by Hyuk Lee et al. (2007) shows that his team used powder metallurgy approach to prepare nano-silica/graphite composites. The journal describes in details on the weight ratio of the nano-silica/graphite mixture (the mixture should be in 30:70 ratio), and also the size of the materials (Nano-silica, 100nm and graphite, 5μm). Some of the processes involved are ball milling, agglomeration, pelletizing and also sintering. It also specifies how long the mixture should be vacuum-dried (6 hours at 100°C) and at what temperature the composite should be sintered at (1000°C under argon atmosphere). It also goes into details on the equipments to be used (SEM, XRD, TEM analysis etc.) to observe the microstructure of the newly formed composites. Unfortunately, the journal actually studies on the electrochemical properties, reversible specific capacity, coulombic efficiency and capacity retention properties of a nano-silica/graphite composite as anode material for lithium-ion batteries. The journal does not relate with this project's objective. Nevertheless, it provides a sound approach to the preparation of graphite composites using powder metallurgy techniques.

Carreira et al. (2003) on his team's journal prepared nano-silica/graphite composites by using co-pyrolysis method. In the journal, he mentioned that carbon materials such as graphite have low mechanical strength, and by the fabrication of carbon-ceramic composites, it is possible to obtain materials with better mechanical properties. However, he also indicated that the mass production of carbon-ceramic composites is expensive, and carbon source materials, such as graphite have low sinterability properties with most refractory powders, thus making powder metallurgy approach not preferable. He suggested using co-pyrolysis as a method of production of carbon-doped materials. Through this method, a heteroatom precursor is solubilized in the carbon precursor such as coal-tar pitch, petroleum residue or synthetic pitches, and the mixtures pyrolyzed to obtain a doped carbon. The journal focuses on preparing a self-sintering silicon-doped material, in which the pyrolysis product is extracted with a solvent to give a self-sintering powder. Two different source of silicon (triphenylsilane and diphenylsilane) are used to study their effects on the properties of the carbons. This journal describe in great details on the co-pyrolysis processes, the equipments used, important details on the preparation of the composites and also the analysis of the properties of the composites.

In summary, researches on the improvement of graphite properties are plenty. Unfortunately, there are not many researches established to study the effect of annealing temperature towards the compressibility of graphite powder. It is stressed again here that the purpose of this project is to establish a study in that area. Many journals are focusing on the study of the electrochemical properties of the graphite composites, the microstructure changes of graphite after heat treatment, and also using graphite as a reinforcement material in alloys. However, the preparation method of the graphite composites using powder metallurgy methods from those journals can be taken as a guideline for conducting this project. Table 2.4 on the next page summarizes the literature review for this project:

Table 2.4: Summary of Literature Review

No	Author(s) + Title	Findings	Remarks
1	**Handbook of Carbon, Graphite, Diamond and Fullerenes: Properties, Processing and Application** (Hugh O. Pierson, 1993, Consultant and Sandia National Laboratories, Albuquerque, New Mexico)	**Materials** • Natural Graphite **Method: Non-applicable** **Processes: Non-applicable** **Results** • Physical properties of graphite • Mechanical properties of graphite • Chemical properties of graphite • Thermal properties of graphite • Electrical properties of graphite • Crystal structure of graphite	This handbook is a review of the science and technology of the element carbon and its allotropes: graphite, diamonds and fullerenes. It provides extensive coverage there are to know regarding graphite; its structure, theoretical properties and chemical reaction, to name a few. Understanding graphite is vital towards the accomplishment of this project, as it is the main element of this project.
2	**ASM Handbook, Volume 7: Powder Metal Technologies and Applications** (Steve Lampman, ASM International Handbook Committee © 1986 ASM International, 1998)	**Materials** • Metal Powders **Method: Non-applicable** **Processes: Non-applicable** **Results** • Compressibility of metal powders • Compactibility of metal powders • Factors affecting compressibility • Compressibility Testing	The ASM Handbooks, particularly in the "Compressibility and Compactibility of Metal Powders" provide a sound and extensive knowledge in understanding the basic principle behind compressibility of metal powders and the factors affecting it. The information gathered provide some basis in progressing this project using graphite powder.
3	**The effect of annealing temperature on micro-structure and mechanical properties of C/SiC composites** (Yumin Zhang, Yunlong Zhang, Jiecai Han, Yufeng Zhou, Luyang Hu, Wang Yao, Wei Qu: 2008, Harbin Institute of Technology, PR China)	**Materials** • α –Silica Carbide Powder (0.5μm, 99.5% purity) • Carbon fibers (3mm long) • Al_2O_3 (1.5μm, 99.95% purity) • La_2O_3 (0.5μm, 99.5% purity) **Method: Ball Milling, Annealing**	This journal provides a good basis to proceed with the project. The study proves that annealing temperature influence the mechanical strength of graphite composite. In this case, they study how annealing affect fracture toughness and flexural strength of the samples. The research suggests that the increase in flexural strength and fracture

		Processes • Preparation of C_f/SiC composites • Ball milling for 8 hour at 180rpm • Annealing at 1650°C, 1750°C, 1850°C and 1950°C • XRD analysis • Density measurement using Archimdes method • SEM analysis • SENB technique • TEM analysis **Equipments** • Ball Milling machine • X-ray Diffractometry (XRD) • Scanning Electron Microscope (SEM) • Densitometer by Archimedes method • Hot-pressing furnace **Results:** The addition of Al_2O_3 and La_2O_3 is effective in promoting densification of the hot-pressed materials. Annealed treatment increases the flexural strength and fracture toughness of samples up until 1950°C, where it start to show decrease.	toughness of the SiC composites is caused by the pullout of the carbon fibre due the annealing. One setback from this study is that, the annealing temperature used is much higher than the equipment in UTP lab can handle. Nevertheless, a good research regarding annealing temperature effect on carbon-based properties.
4	**Crystallization degree of change of expanded graphite by milling and annealing** (Qunwei Tang, Jihuai Wu, Hui Sun, Shi Jun Fang: 2007, Huaqiao University, Guangzhou, China)	**Materials** • Expanded graphite • H_2SO_4 Solution • HNO_3 solution • Distilled water **Method: Ball Milling, Annealing**	This journal provides more evidence that annealing can alter the microstructure of graphite, this time at a temperature suitable to be used by the equipment in UTP laboratory. The recrystallization of graphite will most likely affect its mechanical properties. Further investigation needs to be done in order to determine the effect of annealing temperature on the compressibility of graphite powder.

		Processes • Preparation of expanded graphite • Ball milling from 4 hours and up to 40 hours at 600rpm • Annealing at 600°C and 900°C • XRD analysis • Thermal Gravimetric (TGA) analysis • SEM analysis • Ultrasonic Irradiation **Equipments** • Ball Milling machine • X-ray Diffractometry (XRD) • Scanning Electron Microscope (SEM) • Ultrasonic Irradiator • Thermal Gravimetric • Tube furnace **Results:** It is found that, in the initial milling stage (less than 12 hours of milling), the crystallization degree of the expanded graphite decline gradually, but after 16 hours of milling, a recrystallization of the expanded graphite occurs. Next, at initial annealing time, recrystallization of graphite does not exist, but beyond the annealing time, recrystallization took place, changing the microstructure of the graphite.	
5	**Characterization and Properties of Copper-Silica Sand Nanoparticles Composites** (Tahir Ahmed, Othman Mamat, Bambang A. Wahjoedi: ICPER 2010, Universiti Teknologi PETRONAS, Malaysia)	**Materials** • Silica sand • Copper powder (99.7%, 66.3μm) **Method: Powder Metallurgy**	Although this journal does not relate to the project's objective, it does give a detailed approach on how to produce the graphite pellets using powder metallurgy approach. The methodology involved can serve as basic fundamental guidelines to conduct this research project

		Processes • Powder Preparation & Compaction • Sintering • Density and Hardness Measurement **Equipments** • Photon Correlation Spectroscopy (PCS) • Ball Milling • Dynamic Light Scattering (DLS) • X-ray Diffractometry (XRD) • Autopelletiser • Zetasizer Nano Analyzer • Scanning Electron Microscope (SEM) • Densitometer • Sintering Furnace **Results:** The addition of 20% silica sand nano-particles increased the hardness of copper/nano silica composite up to 70HRB and tensile strength up to 453MPa.	
6	**Spherical silicon/graphite/carbon composites as anode material for lithium-ion batteries** (Jong-Hyuk Lee, Wan-Jum Kim, Jae-Youn Kim, Sung-Hwan Lim, Sung-Man Lee: Kangwon National University, Republic of Korea)	**Materials** • Nano-silica (99.9%, 100nm) • Natural graphite powders (5μm) • Tetrahydrofuran solution (carbon yield, 76%) **Method: Powder Metallurgy** **Processes** • Powder Preparation and Compaction • Sintering • Density and Hardness Measurement **Equipments**	This journal's purpose does not relate with the project's objective and scope of study. Therefore, there is no study on the effect of annealing temperature on the compressibility of graphite. However, the method of preparation of the nano-silica/graphite composite can be used as a guideline for this project to produce graphite pellets samples. This journal implements powder metallurgy approach to prepare the nano-silica/graphite composite.

		Results: The pyrolysis products have been extracted with toluene to obtain self-sintering carbon powders. The thermochemical analysis of the self-sintering powders indicates that thermofusibility is restricted by the presence of silicon. By selecting the appropriate pyrolysis conditions, it is possible to obtain heat treated samples (2100°C) with density and mechanical properties superior to undoped ones, reaching value of 130 Mpa of bending strength and 21 Gpa of Young's Modulus.	

CHAPTER 3
METHODOLOGY

This section will discuss on the materials used in this project, the tools and equipment for conducting the processes involved, the sample preparation and experiment involved, and also the project plan, which will be shown in Gantt chart form.

3.1 Materials

The materials required for this study is natural graphite powder with the size of -20+84 mesh, as seen in Figure 3.1. The details for the materials are as follows:

Graphite Powder

- Origin/Company: Alfa Aesar
- Size: -20+84 mesh (177-841μm)
- Purity: 99.9%

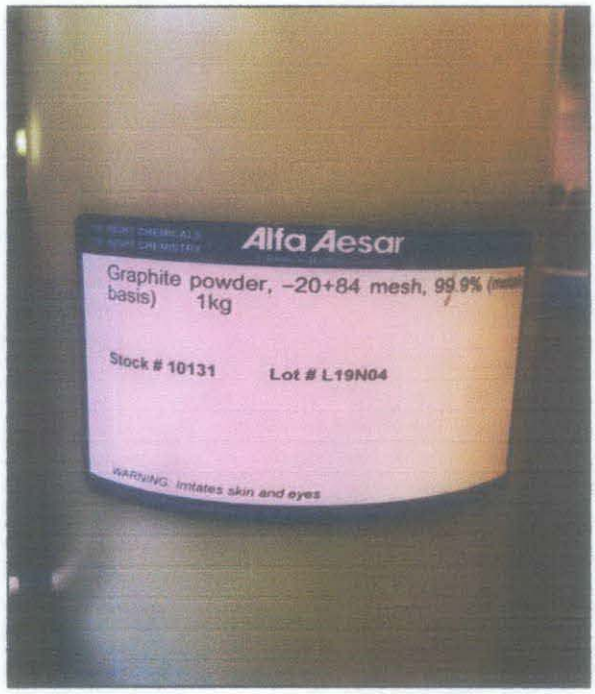

Figure 3.1: Details of Graphite Powder as Taken from the Graphite Container

3.2 Tools and Equipment

The followings are the major tools and equipment that will be used in the laboratory experiment for this study:

- Sieve Analysis Machine
- Mortar Grinder
- US Stoneware Ball Milling Machine
- Tube Furnace
- Carver Autopellet Press Machine
- Mettler Toledo AX205 Density Measurement following Archimedes' Method
- Scanning Electron Microscope (SEM)

3.3 Specific Project Activities

In this project, 40 samples of graphite pellets will be produced using powder metallurgy philosophy. These 40 samples will be grouped based on their annealing temperature. Because of the limited supply of graphite powder, only 4 temperatures will be used; room temperature (unannealed), 700°C, 800°C, and 900°C, each temperature will have 10 pellets pressed at compression pressure ranging from 14Mpa to 140Mpa.

The following samples are planned to be prepared for this project:

- 10 samples of unannealed graphite pellets
- 10 samples of graphite pellets annealed at 700°C
- 10 samples of graphite pellets annealed at 800°C
- 10 samples of graphite pellets annealed at 900°C

The specific activities which were conducted throughout this project are as follow:

- Grinding of graphite powder
- Sieve testing

- Annealing of graphite powder
- Compaction of graphite powder
- Green density measurement of graphite pellet
- Microstructure analysis using SEM

Below are the descriptions of each specific project activities mentioned above.

3.3.1 Grinding of Graphite Powder

The graphite powder received for this project is -20+84 mesh (177-841μm) in size, a considerably large particle size to be compressed to pellet form. The size of the graphite particle should be decreased to allow the compaction to pellet form occur smoothly. The graphite powder will be grounded using ball milling machine and mortar grinder, and screened using a sieve of 63μm.This is to ensure that the graphite powder used to produce the pellets has the same particle size of 63μm. Note that the 63μm sieve is the smallest sieve available in the lab.

In the previous progress of the project, the graphite powder was compressed directly without grinding it to a smaller particle size. The result of the compaction however, was a failure. The graphite powder failed to compress to a good pellet; the pellet produced was very brittle and fragile, and it crumbled when touched. Therefore, it is essential to reduce the particle size of graphite powder to a smaller size.

3.3.2 Sieve Testing

After the powder has been grounded, the powder will be screen using a sieve of 63μm (the smallest sieve available). This is to ensure that the powder used in the compaction process is homogenous and has the same particle size of 63μm.

- 10 samples of graphite pellets annealed at 800°C
- 10 samples of graphite pellets annealed at 900°C

3.3.5 Green Density Measurement of Graphite Pellets

After the graphite powder has been compacted to pellet form, the green density of the samples will be taken using Mettler Toledo AX205 Density Measurement, using Archimedes' Method. This density will then be recorded properly to be used for developing compressibility curve.

3.3.6 Microstructure Analysis using SEM

The pellets produced before will undergo SEM analysis to observe the microstructure changes after the powder has been annealed. The microstructure of the annealed samples will be compared with the microstructure of the unannealed samples.

3.4 Project Planning and Key Milestones

The project will be conducted in two semesters. The plan for this project for both semesters is shown in the next page in Gantt charts for July 2010 semester and May 2011 semester.

Table 3.1: Project Planning for July 2010 Semester

No	Activities	Week													
		1	2	3	4	5	6	Mid-Semester Break	8	9	10	11	12	13	14
1	**Selection of Project Title**														
2	**Preliminary Research Work**														
	• Literature review														
	• Preliminary report preparation														
	• Submission of preliminary report				▲										
3	**Project Work**														
	• Grinding of raw graphite powder for 6 hours using Ball Milling machine														
4	**Progress report and Seminar**														
	• Data gathering and analysis														
	• Submission of progress report								▲						
	• Seminar								▲						
5	**Project Works Continues**														
	• Grinding of raw graphite powder using mortar grinder														
	• Sieve analysis test of 63μm sieve size to the graphite powder														
6	**Interim Report**														
	• Data gathering and analysis														
	• Submission of interim report														▲
7	**Oral Presentation**	During Study Week													

Process

Key Milestone

Table 3.2: Project Planning for May 2011 Semester

No	Activities	Week													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	**Project Work Continues**														
	• Literature review	■	■	■	■	■	■	■	■	■	■	■	■		
	• Production of 40 graphite powder pellets by compaction or graphite powder using autopelletizer machine.	■	■	■	■										
	• Measurement of sintered density of the composites using Mettler Toledo AX205 Density Measurement					■	■	■							
2	**Progress Report 1**														
	• Data gathering and analysis		■	■											
	• Submission of progress report 1				▲										
3	**Project Work Continues**														
	• Microstructure observation of the composites by SEM								■	■					
4	**Progress Report 2 and Seminar**														
	• Data gathering and analysis							■							
	• Submission of progress report 2								▲						
5	**Project Work Continues**														
	• Dissertation draft preparation									■	■	■	■	■	■
6	**Poster Exhibition**														
	• Data gathering and analysis									■	■				
	• Submission of poster								▲						
7	**Dissertation Final Draft & Technical Paper**														
	• Submission of dissertation final draft													▲	
	• Submission of Technical Paper														▲
8	**Oral Presentation**	During Study Week													
9	**Hard Bound Dissertation Submission**	7 Days after Oral Presentation													

■ Process

▲ Key Milestone

3.5 Flow Chart

Figure 3.3 below shows the flow chart of the project. It describes the flow of the project's specific activities that would be conducted.

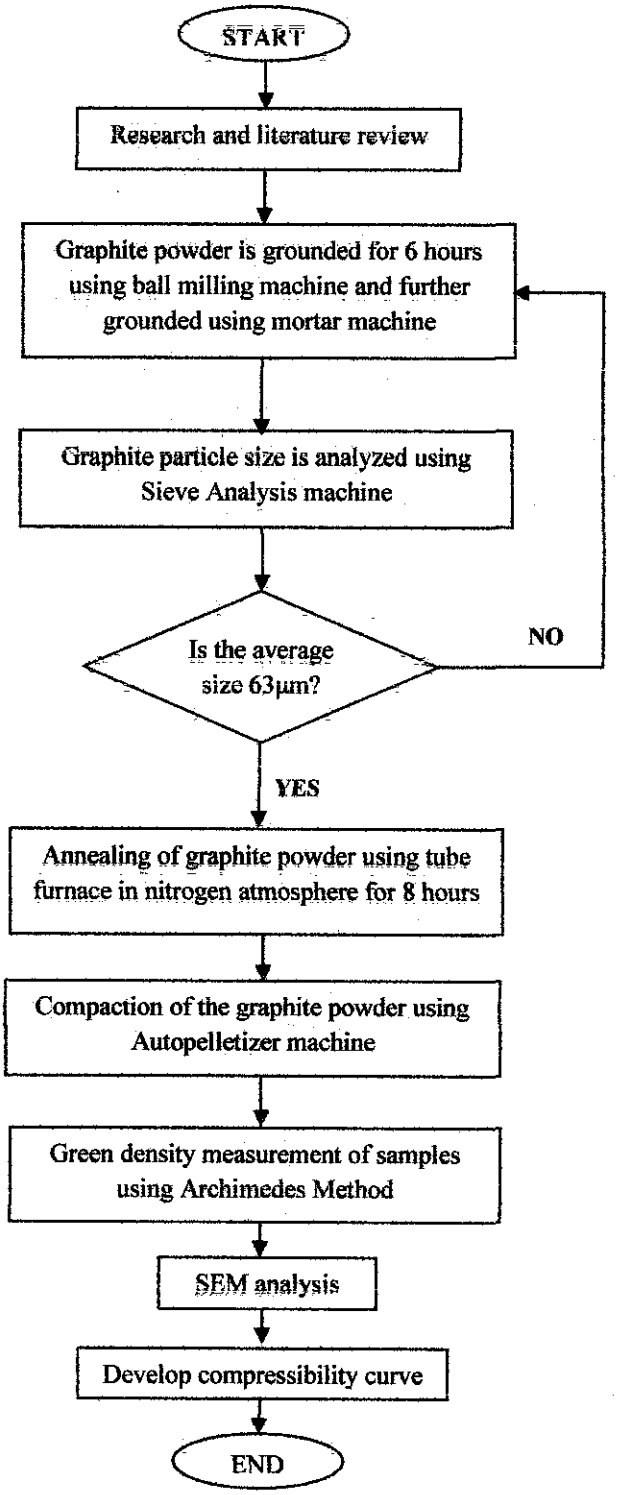

Figure 3.3: Project Flow

3.6 Work Progress

During the earliest stage of this project, the graphite powder received was assumed to have particle size that is suitable for compaction. Therefore, the powder was being compacted to pellet form straight away to begin the project quickly. However, the attempt to compact the composite powder was unsuccessful.

The cause of the problem was investigated. It was found that, through literature reviews, the compaction was unsuccessful due to the large particle size of the graphite powder (177-841μm). Most researchers used graphite powder of smaller size; some even used nano-size graphite powder.

Effort has been focused on reducing the graphite powder size used for this project to a finer size using ball milling machine and mortar grinder. It was decided to have graphite powder having 63μm particle size for this project. Sieve analysis was used to get the targeted particle size. Note that the 63μm sieve is the smallest sieve available in the lab.

The process of reducing the graphite powder size took quite a longer time than expected. The graphite powder obtained after sieve analysis was not much to begin the project. This may be due to graphite particles having high compressibility, making it difficult to reduce its size. Nevertheless, the process of reducing the powder using ball milling and mortar grinder and screened it using 63μm sieve was repeated until the amount of graphite powder needed to start the project was achieved. It is estimated that the project will need 70g of fine graphite powder for the production of 40 graphite pellets. After much hard work, the amount of fine graphite powder needed for this project was obtained.

After that, the project progressed smoothly without any major difficulties. It was again worthwhile to stress that the real challenge of this project is to reduce the graphite powder size to a much smaller size, as it takes a lot of time and effort.

The following section summarizes the work progress of the project.

3.6.1 Grinding of Raw Graphite Powder

The size of the graphite powder need to be reduced as it is too large to allow for good compactions. The reduction of graphite particle size was done by grinding the powder using ball milling machine and mortar grinder. The graphite particle size would be reduced to 63μm. Figure 3.4 shows the photo of the grinding process.

Figure 3.4: Grinding of Raw Graphite

To ensure the size reduction was effective, 250 ceramic balls were used as the grinding media to grind 70g of graphite powder. The powder was grounded for 6 hours every day. The ball mill speed was set to 100 rpm. Before the powder is screened using a 63μm sieve, the powder is grounded again using a mortar grinder. After the grinding finished, sieve analysis test was done to the graphite powder to obtain fine graphite particle of 63μm size.

3.6.2 Sieve Analysis Testing of Graphite Powder

The purpose of doing the sieve analysis testing is to obtain fine graphite powder to be used for this experiment. A sieve size of 63μm was used for this project. The 63μm sieve size was chosen because it was the smallest sieve available. Basically, the sieve will filter the graphite powder so that only particle having size of 63μm and below can pass through. Figure 3.5 shows the sieve used in this project. First, the raw graphite was placed on the sieve (see Figure 3.6) and

then the sieve would be closed and placed on a mechanical sieve shaker (see Figure 3.7). Figure 3.8 and Figure 3.9 shows the difference between raw graphite particle size and 63μm fine graphite particle.

Figure 3.5: 63μm Sieve

Figure 3.6: Raw Graphite on Sieve

Figure 3.7: Sieve Shaker

Figure 3.8: Raw Graphite Powder

Figure 3.9: Fine Graphite Powder after Sieve Analysis

One problem was encountered during this stage. The amount of fine graphite powder passing through the sieve was very small. As an example, the first attempt of conducting the sieve analysis yielded only less than 7g of fine graphite powder having size of 63μm out of 70g powder in total. This was obtained after 4 weeks of grinding the raw graphite powder and doing sieve analysis. This problem has somehow halted the progress of the project and added to its time constraint.

3.6.3 Annealing of Graphite Powder

Next, the fine graphite powder is annealed at 4 different temperatures: unannealed, 700°C, 800°C and 900°C. The powder is annealed using a tube furnace in a nitrogen atmosphere. The purpose of the nitrogen gas is to remove the oxide and water content that may contaminate the graphite powder. Figure 3.10 shows the tube furnace used for this project:

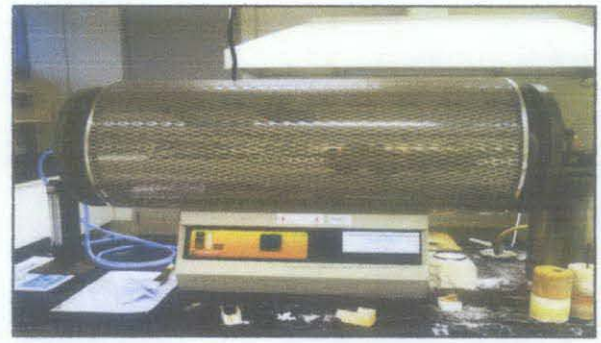

Figure 3.10: Tube Furnace

3.6.4 Compaction of Samples

After all the samples had been annealed according to their annealing temperature, the next step is to compact the samples using Autopelletizer machine. There will be 40 pellets produced in this stage; each annealing temperature will have 10 pellets that are pressed at pressure ranging from 14Mpa to 140Mpa. Each pellet will be produced from 0.5g graphite powder. Figure 3.11 shows the Autopelletizer machine used.

It is mentioned before that the project initially produced graphite pellets using the raw graphite powder having large particle size. The compaction however was a failure, and efforts had been put to reduce the graphite powder particle size since. Figure 3.12 shows the difference between a pellet produced from raw graphite powder and a pellet produced from a fine one. The pellet from raw graphite powder is very brittle and very easy to crumble, making it very hard to handle. The other pellet on the other hand is stronger and has a smooth surface, which makes it preferable to be used of this project.

Figure 3.11: Autopelletizer Machine

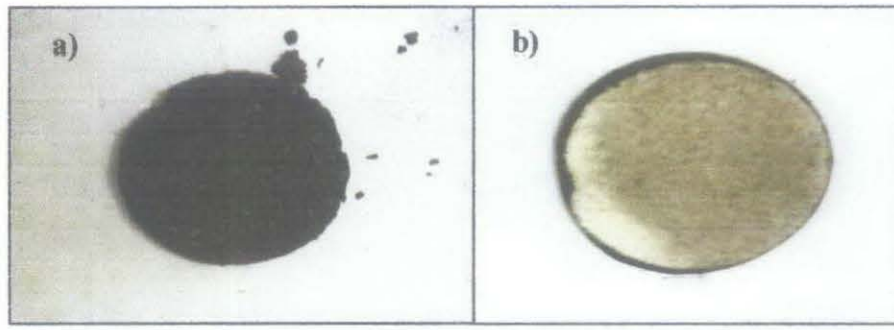

Figure 3.12: a) Pellet produced from raw graphite powder; b) Pellet produced from fine graphite powder

3.6.5 Green Density Measurement

The density of the 40 pellets produced during the compaction stage is measured by Mettler Toledo AX205 Density Measurement, using Archimedes' Method. The density is recorded and the compressibility curve of each annealing temperature is developed and compared. The density and compressibility curve of the pellets are shown in the next section. From the compressibility curve, it will be observed what effect the annealing temperature has on the compressibility of graphite powder. Figure 3.13 shows the densitometer used to measure the density of the pellets.

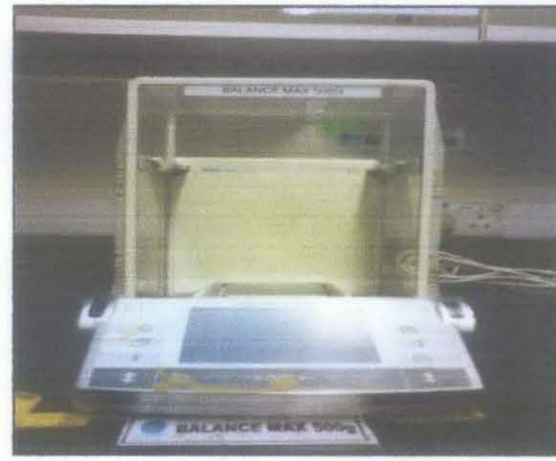

Figure 3.13: Mettler Toledo AX205 Density Measurement using Archimedes' Method

3.6.6 SEM Analysis

Pellets from 137.90 MPa compacting pressure from each annealing temperature are used in the SEM analysis. From the analysis, it will be observed how the microstructure of the pellets changes from the unannealed one, and how does annealing temperature affect them.

CHAPTER 4
RESULT AND DISCUSSION

4.1 Results and Findings

This section will show all the findings gathered from this project and the analysis of the results. Below are the results collected from the progress of the project. The first part shows the compressibility curve of the graphite powder and the second part shows the microstructure of the graphite powder by SEM analysis.

In the first part, the connection between the annealing temperature and the compressibility of the graphite powder will be discussed. In the second part, the effect of the annealing temperature to the pellets' microstructure and how the changes in microstructure affect the compressibility of the pellets will be analyzed.

4.1.1 Compressibility Curve

Table 4.1 shows the density of the graphite pellets measured using the densitometer and the corresponding compacting pressure used to compact the graphite powder into pellet form. The density of each annealing temperature (unannealed, 700°C, 800°C and 900°C) is recorded and the corresponding compressibility curves are constructed as shown in Figure 4.1.

Table 4.1: Density of unannealed, 700°C, 800°C and 900°C graphite pellet

	Density (g/cm^3)			
Compacting Pressure (MPa)	**Unannealed**	**700°C**	**800°C**	**900°C**
13.79	1.227	1.223	1.224	1.251
27.58	1.222	1.234	1.247	1.278
41.37	1.243	1.246	1.264	1.286
55.16	1.236	1.276	1.249	1.291
68.95	1.252	1.323	1.315	1.314
82.74	1.274	1.343	1.341	1.328
96.53	1.343	1.353	1.361	1.351
110.32	1.298	1.356	1.348	1.367
124.11	1.343	1.346	1.354	1.357
137.90	1.229	1.340	1.352	1.341

From Table 4.1, it is observed that the increasing compacting pressure results in increasing the green density of the graphite pellets. This implies that the porosity of the pellets also decrease, thus improving the density of the pellets. It can be also observed that the density of the pellets of the same compacting pressure increase as the annealing temperature increases.

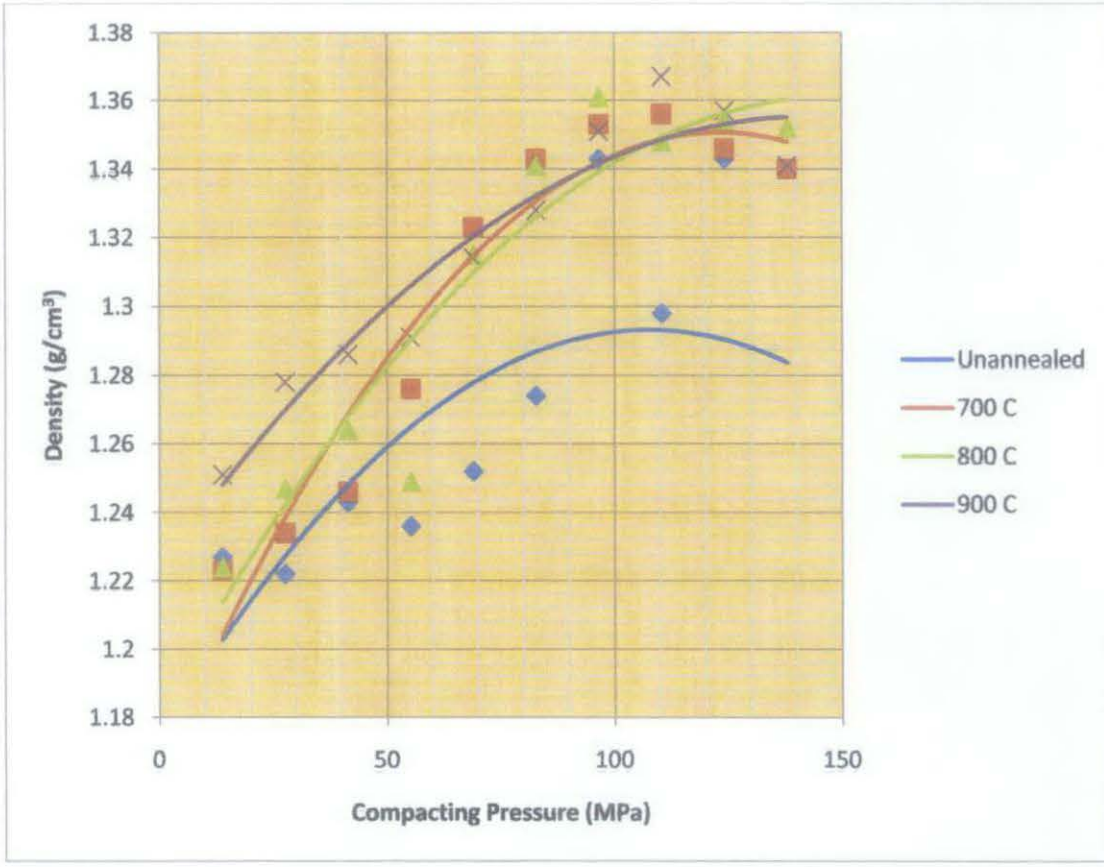

Figure 4.1: Compressibility Curve of unannealed, 700°C, 800°C and 900°C graphite pellets

The compressibility curves of pure graphite as shown in Figure 4.1 above are constructed based on the data collected in Table 4.1. As shown in the figure, it is seen that, increasing the annealing temperature will increase the compressibility of the pellets. Pellets annealed at 900°C have higher density (thus higher compressibility) than the other annealing temperature.

4.1.2 SEM Analysis

The microstructure of the graphite pellets have also been recorded using SEM. Figure 4.2 shows the microstructure of the pellets at their corresponding annealing temperature. For the SEM analysis, all the annealing temperature use pellets compacted at 137.90MPa. The images were captured at 1000x magnification.

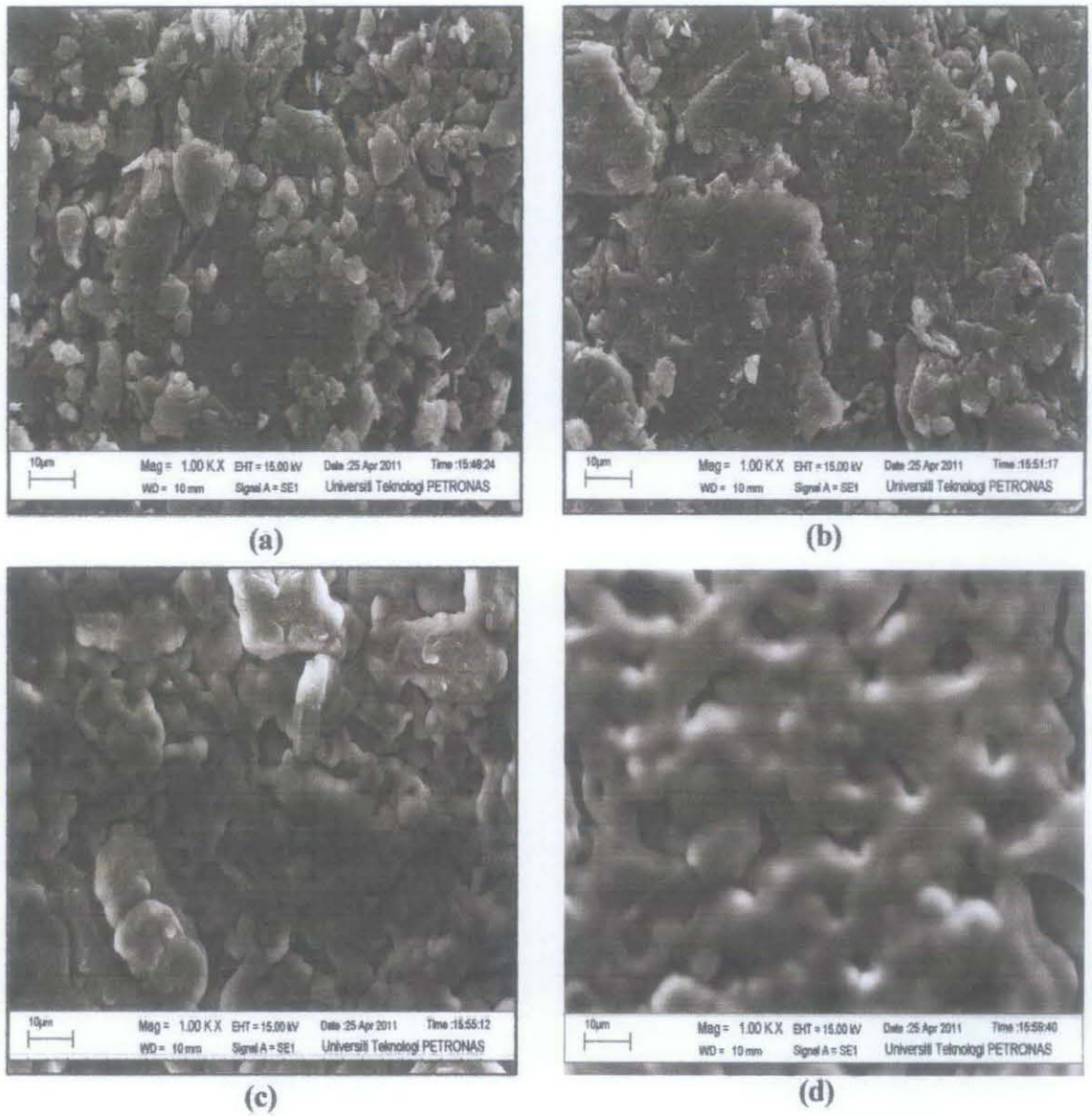

Figure 4.2: The microstructure of graphite pellets at 137.90MPa compaction pressure for (a) unannealed, (b) 700°C, (c) 800°C and (d) 900°C annealing temperature

From the microstructure, it can be observed that with increasing annealing temperature, the diffusion between graphite particles take place, thus reducing porosity. Necking and bridging structure can be seen clearly in Figure 4.2 (d) as compared to Figure 4.2 (a).

4.2 Discussion

Graphite powders, and generally any metal powders will contain residual amount of carbon, oxygen and nitrogen (also called residual stress) that reduce its green strength and compressibility. Thus, "annealing", or the stress relieving and recrystallizing of a cold worked material can increase the ductility of the material, consequently improving the green strength and compressibility (Lampman, 1998).

With regards to the project, as the annealing temperature increases, graphite particles will come closer to each other. This is because, when the powders are subjected to annealing, the residual stress will be released. With the absence of residual stress, during the compression of powders, graphite particle will be bonding with each other, making a homogenous structure and improving the density of graphite.

With respect to the microstructure of graphite pellets, increasing the annealing temperature results in the development of grain boundaries. Necking and bridging of particles take place as seen in Figure 4.2. As the compression force increase, the interstice places between the particles decreases and it causes improvement of the strength and density of graphite by reducing the porosity of graphite.

Therefore, based on the analysis, it can be concluded that annealing the graphite powder will increase the compressibility of graphite. Annealing remove residual stress between graphite particles, thus decreasing the number of voids or interstice places after the powder is compressed into pellets form. Compressing the powders after annealing will increase the pellets density as compared to compressing the unannealed powder. Annealing will also increase the development of grain boundaries, thus making the bond between graphite particles stronger, increasing the green strength as well during the process.

CHAPTER 5
CONCLUSION AND RECOMMENDATIONS

The study on the compressibility and microstructure changes of graphite is an important step towards improving the physical and mechanical properties of graphite. Graphite is widely used in numerous industries; some of them are highly risky and dangerous industries. Graphite is known to be used as a material in manufacturing components in nuclear reactor as well as rocket nozzle. This is due to graphite having very good thermal properties. However, despite having good thermal properties, graphite has low physical and mechanical properties, and this is seen as a liabilities and weaknesses to graphite. The objective of this research is to study on the effect of annealing temperature on the compressibility and microstructure changes of graphite, in hope that compressibility, one of the mechanical properties of graphite, can be improved.

5.1 Conclusion

The first part of this project is to study whether by annealing the graphite powder, the compressibility can be improved. 4 sets of graphite powder were annealed at 4 different temperatures (unannealed, 700°C, 800°C and 900°C). The powders then were compressed at compaction pressure of between 14MPa-140MPa. Each annealing temperature will have 10 pellets. Next, the pellets' densities were measured and a compressibility curve was developed. The compressibility curve would show clearly whether annealing can improve graphite compressibility. Then, SEM analysis was conducted to pellets at compaction pressure of 137.90 MPa for every annealing temperature.

From the compressibility curve, it was found that, with increasing annealing temperature, the compressibility of graphite increases too. It is evident from the trend of the compressibility curve for each of the annealing temperature. The unannealed samples, which act as the datum for the research, has the lowest compressibility,

compare to the other annealing temperatures. With increasing annealing temperatures, it is observed that the compressibility curves for the rest of annealing temperatures exhibit increasing trend, with the annealing temperature of 900°C having the highest compressibility.

SEM analysis was conducted to pellets compressed at 137.90 MPa for all annealing temperatures. From the microstructures of all 4 pellets, it is observed that, annealing changes the microstructures of the graphite pellet. Necking and bridging structures occur on pellet annealed at 900°C as compared to the unannealed pellet.

Annealing remove residual stress between graphite particles, thus increasing the number of voids or interstice places after the powder is compressed into pellets form. Compressing the powders after annealing will increase the pellets density as compared to compressing the unannealed powder. This leads to the increasing trend of the compressibility curve, where higher annealing temperature will exhibit higher compressibility as compared to the lower temperature. Annealing will also increase the development of grain boundaries, thus making the bond between graphite particles stronger, increasing the green strength as well during the process.

5.2 Recommendations

There are rooms of improvements for the continuity of this project. For further studies on this topic, a study on the optimum annealing temperature which will produce the best compressibility can be conducted. Other than that, studies can also be made on the improvement of other mechanical properties, such as hardness and wear resistance of graphite.

CHAPTER 6
REFERENCES

Azom.com. *Graphite (C) – Classifications, Properties and Applications of Graphite.* September 10, 2002. Retrieved May 24, 2011, 8.10 PM from: http://www.azom.com/article.aspx?articleid=1630#_Background

Donald W. Olson. 2008, *2008 Minerals Year Book: Graphite [Advance Release]*, U.S. Department of Interior & U.S. Geological Survey.

Galleries.com. *Graphite*. 1995. Retrieved May 25, 2011, 9.30 PM from: http://www.galleries.com/minerals/elements/graphite/graphite.htm

Geology.com. *Graphite - Mineral Properties and Uses*. 2005. Retrieved May 24, 2011, 3.00 PM from: http://geology.com/minerals/graphite.shtml

Hugh O. Pierson. 1993. *Handbook of Carbon, Graphite, Diamond and Fullerenes: Properties, Processing and Application.* New Jersey. Noyes Publications.

Jong-Hyuk Lee, Wan-Jum Kim, Jae-Youn Kim, Sung-Hwan Lim and Sung-Man Lee. 2007, "Spherical silicon/graphite/carbon composites as anode material for lithium-ion batteries", *Journal of Power Sources*, 176 (2008) 353-358.

P. Carreira, J. Sánchez-Coronado, J.Narciso, M. Martínez-Escandell, F. Rodríguez-Reinoso. 2008, "Preparation of graphite/nano-SiC composites by co-pyrolysis of a petroleum residue with phenylsilanes", *Journal of Analytical and Applied Pyrolysis*, 83 (2008) 137-144.

Qunwei Tang, Jihuai Wu, Hui Sun, Shi Jun Fang. 2007. "Crystallization degree change of expanded graphite by milling and annealing". *Journal of Alloys and Compounds.* 475(2009) 429-433.

Sontea Sever and Raducanu Liviu. 2002, "Introduction to Powder Metallurgy", *University's Day 8th International Conference*, Constantin Brâncusi University, Romania.

Steve Lampman, ASM International Handbook Committee © 1986 ASM International, 1998, *ASM Handbook, Volume 7: Powder Metal Technologies and Applications*

Tahir Ahmed, Othman Mamat and Bambang A. Wahjoedi. 2010, *Characterization and Properties of Copper-Silica Sand Nanoparticles Composites*, Ph.D. Thesis, ICPER 2010, Universiti Teknologi PETRONAS, Malaysia

Yumin Zhang, Yunlong Zhang, Jiecai Han, Yufeng Zhou, Luyang Hu, Wang Yao, Wei Qu. 2008. "The effect of annealing temperature on micro-structure and mechanical properties of C/SiC composites". *Material Science and Engineering A*. 497(2008) 383-387