Sintering Carbon Nanotubes Reinforced Copper Powder Using Different Particle

By

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

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
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Approved by,

(Associate Professor Dr. Faiz Ahmad)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

May 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. Also some data in the project was taken with permission by the person involved.

(ARIFF EZWAN BIN ZAKARIA)

ABSTRACT

This research is about developing a carbon nanotubes reinforced copper matrix composite by using the powder metallurgy process and the result is being analyzed. The copper composite was prepared on the volume fraction of the carbon nanotubes of 2%. The test sample of pure copper also developed to compare the effect of reinforcing carbon nanotubes to the copper. The powder metallurgy method that was used in this experiment to develop the samples has three main stages that are mixing, compacting and sintering. Sintering is the most crucial part to be taken care of including the temperature and also the environment of the sintering process. In this experiment, the sintering temperature used is 850°C and the sintering process is done in and inert gas environment which is Argon. The usage of carbon nanotubes reinforcement is become a new invention in advance material technology and expected to be wider and wider globally because of the enhanced properties of the composite. By increasing the volume fraction of the carbon nanotubes to 2% some of the properties value such as thermal conductivity is significantly increased which prove that the carbon nanotubes give a good benefits as the reinforce material. And as the outcome of this research, the usage of composite will be widened in the advance material technologies.

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CHAPTER 1
INTRODUCTION

1.1. BACKGROUND

Composites materials have been utilized to solve technological problems for a long time but only in the 1960s did these materials start capturing the attention of industries with the introduction of polymeric-based composites [1]. Since then, composites materials have become common engineering materials and are designed and manufactured from various applications including automotive components, sporting goods, aerospace craft, consumer goods and in the marine and oil industries. The growth in composites usage also came about because of increased awareness regarding product performance and increased competition in the global market for lightweight components.

Today, the most common man-made composites can be divided into three main groups which are Metal Matrix Composite (MMC), Polymeric Matrix Composite (PMC) and Ceramic Matrix Composite (CMC). The PMC use the metal based as the matrix field and the fibrous materials as the reinforcement. CNT reinforced copper composite is one example of MMC.

CNT are molecular-scale tubes of graphitic carbon that have outstanding properties. They are among the stiffest and strongest reinforcement material known and have remarkable electronic properties, mechanical properties and many other unique characteristics. It is grown now by several techniques in the laboratory and is just a few nanometers in diameter and several microns long. CNT can be metallic or semiconducting and offers amazing possibilities to create future nanoelectronics devices, circuits, and computers. CNT exhibits extraordinary mechanical properties: the Young's modulus is over 1 Tera Pascal. It is stiff as diamond. The estimated tensile strength is 200 Giga Pascal. These properties are ideal for reinforced composites, nanoelectromechanical systems (NEMS).

CNT have a good characteristic in acting as a reinforcement material in composite because of the outstanding properties that it has. In this project, the CNT will be used as a reinforcement material in copper matrix. By reinforcing the CNT into the copper, the properties of the composite will be improved.

1.2 PROBLEM STATEMENT

The behaviour of Carbon Nanotube reinforced Copper powder may vary with the particle shape of the composite. Study will be focuse ' to investigate the effects of particle shape on sintering of copper matrix composites. Also the different of the density value for the different matrix used. The potential application may as heat sink in electronic industry as the composite has a high thermal conductivity value.

1.3 OBJECTIVE AND SCOPE OF STUDY

The objective of this research is to develop copper matrix composites reinforced with CNT using powder metallurgy route. Study bonding between CNT and copper powder, measure sintered density, green density and work out theoretical density.

This study is about the development and characterization of CNT reinforced copper matrix composite using spherical shape of copper which have different size. This project will also cover the properties of the composite in term of mechanical properties including thermal conductivity.

CHAPTER 2

LITERATURE REVIEW AND THEORY

2.1 Properties Of Composite

The properties of the composites, usually uensity can be predicted by the 'Rule of Mixtures' can be denoted as weight fraction or volume fractions [6]. The rule is mathematical expressions which give some properties of the composites, quantity and arrangement of its constituent. The volume fractions and weight fractions are given by the equation below [2].

$$v_c = v_f + v_m \qquad (2.1a)$$

$$V_f = \frac{v_f}{v_c}, \quad V = \frac{v_m}{v_c} \qquad (2.1b)$$

And

$$w_c = w_f + w_m \qquad (2.1c)$$

$$W_f = \frac{w_f}{w_c}, \qquad W_m = \frac{w_m}{w_c} \qquad (2.1d)$$

where:

v_c : volume of composite

v_f : volume of reinforcement material

v_m : volume of matrix material

V_f : volume fraction of reinforce material

V_m : volume fraction of matrix material

w_c : weight of the composite

w_f : weight of the reinforce material

w_m : weight of the matrix material

W_f : weight fraction of the reinforce material

W_m : weight fraction of the matrix material

The density (ρ) of the composites material must be obtained in order to establish conversion relations between the weight (m) fractions and the volume (v) fractions. From the basic equation of density, the mass divided by the volume.

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

The weight in equation Eq. (2.1c) can be replaced by the density and volume and equation written as:

$$\rho_c = \rho_f v_f + \rho_m v_m \tag{2.2}$$

Dividing both sides in Eq. (2.2) by v_c and substituting Eq. (2.1b), the Eq. (2.2) can be rewritten as:

$$\rho_c = \rho_f V_f + \rho_m V_m \tag{2.3}$$

For the case of fiber-matrix composites, the equation Eq. (2.3) can be written as:

$$\begin{aligned} \rho_c &= \rho_f V_f + \rho_m V_m \\ &= \rho_f V_f + \rho_m (1 - V_f) \\ &= V_f (\rho_f - \rho_m) + \rho_m \end{aligned} \tag{2.4}$$

Since $V_f + V_m = 1$

Thermal conductivity prediction using Rule of Mixture

$$k_c = k_f V_f + k_m V_m \tag{2.5}$$

where

k_c: thermal conductivity of composite

k_f: thermal conductivity of reinforced material

k_m: thermal conductivity of matrix material

2.2 Carbon Nanotubes

2.3.1 Types of Carbon Nano Tubes (CNT)

There are several types of CNT that are used in today's industries that are [6]

- Single-walled
- Multi-walled
- Fullerite
- Torus

For this project, the type of CNT that will be used is the single-walled CNT.

2.2.2 Properties of CNT

Carbon nanotubes have received considerable attention because of their excellent in electrical and mechanical properties. [9] The strength of the sp^2 carbon-carbon bonds gives carbon nanotubes amazing mechanical properties. The stiffness of a material is measured in terms of its Young's modulus, the rate of change of stress with applied strain. The Young's modulus of the best nanotubes can be as high as 5 TPa which is approximately 5 times stiffer than iridium. [3] The tensile strength, or breaking strain of nanotubes can be up to 63 GPa, around 50x higher than steel. [6] These properties, coupled with the lightness of carbon nanotubes, give them great potential in applications such as aerospace. It has even been suggested that nanotubes could be used in the "space elevator", an Earth-to-space cable first proposed by Arthur C. Clarke. The electronic properties of carbon nanotubes are also extraordinary. Especially notable is the fact that nanotubes can be metallic or semiconducting depending on their structure. There is great interest in the possibility of constructing nanoscale electronic devices from nanotubes, and some progress is being made in this area. However, in order to construct a useful device we would need to arrange many thousands of nanotubes in a defined pattern, and we do not yet have the degree of control necessary to achieve this. There are several areas of technology where carbon nanotubes are already being used. These include flat-panel displays, scanning probe microscopes and sensing devices. The unique properties of carbon nanotubes will undoubtedly lead to many more applications. For thermal conductivity, CNT is the best thermal conducting materials and for single-multi walled CNT reached as much as 3000 W/mK at room temperature. [10]

2.3 Properties of Copper

Copper powder particles can be made by several methods such as atomization using gas or water, electrolysis, hydrometallurgy and solid state reduction. The copper powder properties like the shape and particle size, density etc depending on how they are produced. For example in atomization, first the copper will melted down and the liquid metal flows through the orifice where it struck by a high velocity stream of gas or liquid, usually water to broke the molten metal into particle which solidify rapidly. Particle size and shape are influenced particularly by the atomizing medium, the pressure and the flow rate. Controlled small additions of deoxidizing elements, such as phosphorus, also influence the particle size and shape. After atomization and annealing in a reducing atmosphere to decrease any surface oxide formed during atomization, the product is milled, classified and blended to achieve the particle size distribution required [2].

Copper has density of 8.93 g/cm^3 with 1084.87^{o}C of melting point temperature. Copper is very good in thermal conductivities, denoted by *k*, about 401 (W/m.K) at room temperature [6]. Copper powder can vary in shapes depending on how the production takes place, whether atomization, chemical or electrolysis. To determine the shape of copper powder, Scanning Electron Test can be carried out. Once we know the shape, we know how the copper powder was produced.

2.4 Powder Metallurgy

Powder metallurgy, or PM, is a process for forming metal parts by heating compacted metal powders to just below their melting points. Although the process has existed for more than 100 years, over the past quarter century it has become widely recognized as a superior way of producing high-quality parts for a variety of important applications. This success is due to the advantages the process offers over other metal forming technologies such as forging and metal casting, advantages in material utilization, shape complexity, near-net-shape dimensional control, among others. These, in turn, yield benefits in lower costs and greater versatility. [5] The properties which the components exhibit makes them are allowed to be used in most application without further processing. It offers a means conserving materials, reducing machining and securing a uniform product at a reasonable cost. By proper selection of material, powder metallurgy specialist can controlled the density of product and secure mechanical and physical properties. A wide range or products have been produced using powder metallurgy such as gears, surgical scissors and connecting rod etc.

Powder metallurgy involves several basic processes starting from powder particles or raw materials. Basically this powder will mix with other materials plus some additive or binder acts as a lubricant. We call it mixing process. Forming process follow up after that which can be divided into three types which are:

1. Hot compaction.
2. Warm compaction.
3. Cold compaction.

After compaction, the compacted material will be sintered in sintering process which occurs in the normal atmosphere, vacuum or inert gas atmosphere at specific temperature of the material. Then the sintered materials will go on to the optional operations include sizing, coining, machining, heat treating and etc. In basic there are three processes involved which are:

1. Mixing
2. Compaction
3. Sintering

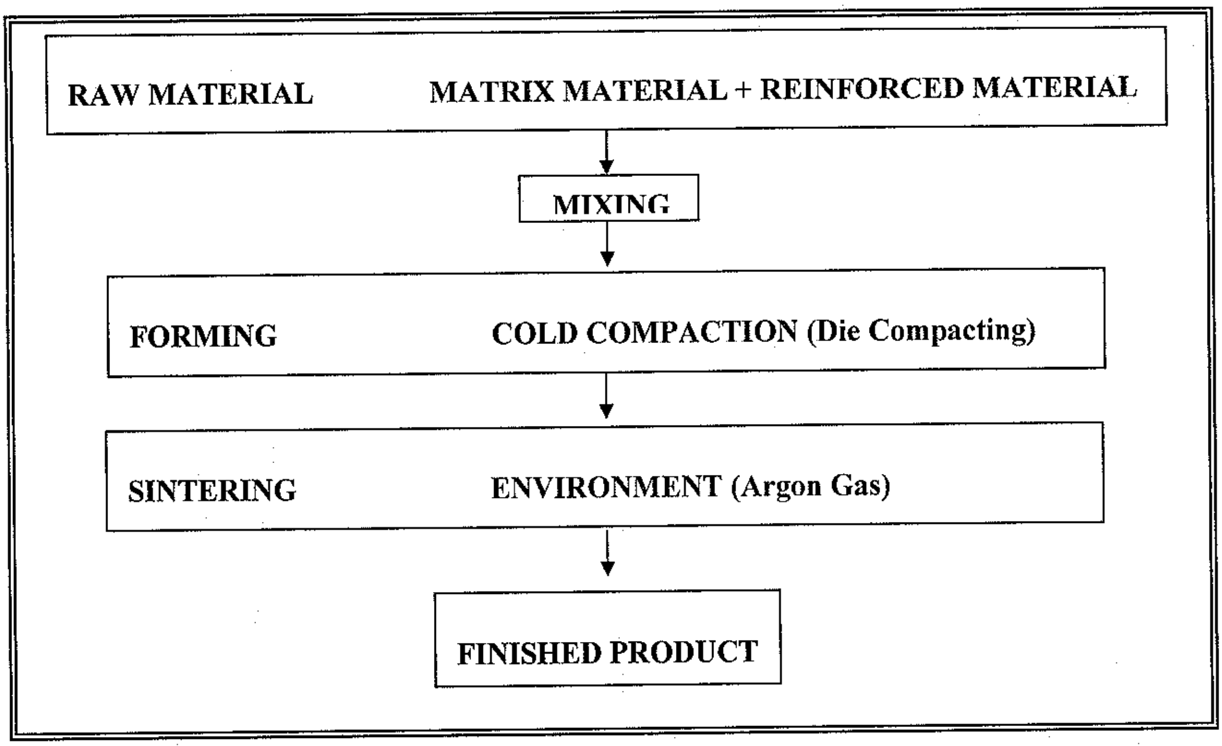


Figure 2.1: Flow-chart of Powder Metallurgy involved

CHAPTER 3

EXPERIMENTAL PROCEDURE

3.1 Scanning the shape of the Copper powder

For the first stage of the project work, the shape of the copper powder was scanned using the Scanning Electron Microscope (SEM). Scanning electron microscope (SEM) is used in copper powder analysis to know the characterization of the powder sample. Using SEM we can know the shape of the particle size and shape. To determine the size is quite difficult as the size of the particles are not constant.

The process was conducted by the technician who is responsible in handling the machine. A fraction of the copper powder was taken and stick to the material holder specifically designed for the SEM. The sample is then magnified, ranging from 100X to 5000X magnifying power. From the SEM, the particle shape and average size can be determined. Two samples of Copper is tested with SEM.

3.2 X-Ray Diffraction

X-Ray diffraction (XRD) used to determine whether the copper powder purity or in other words, the copper powder is not been oxidized. In this process, the copper is put into the XRD chamber then vacuumed. This process take about two (2) and half (1/2) hours to complete. This experiment was conducted by the respective technicians of the machine. The results are display in graph of spectrum of each sample, copper powder (database), copper powder (sample) and copper oxide.

3.3 Powder Metallurgy Process

3.3.1 Mixing Process

Controlled amount of copper powder and carbon were mixed up and blend homogenously before pour into die compacting mould. This process is manual, no machine involved. The first step is copper powder is weighted and put into a beaker, then followed by the carbon nanotubes. Second step is carbon nanotubes is mixed with the copper powder. It is to ensure that the mixture is homogenous.

3.3.2 Compaction Process

In this process, control amount copper powder is mixed with chopped carbon nanotubes. We used volume fraction of 1% 2% carbon nanotubes and copper powders in term of fiber volume fraction by using the rule of mixture formula. After calculating respective weight of both carbon nanotubes and copper powder, the material is mixed up. Then this mixture is stir to make it homogeneous. After several minute of stirring, the mixed material is put into the precision die and the pre-compress using bare hand. Then the mixture is put on the die compacter plate and compressed approximately 17 ton or approximately 9MPa of load. After that, green product is ejected out from the die.

3.3.3 Sintering Process

Sintering process is heating up products below melting point of the material approximately 80% to 85% of the melting point temperature of material in the argon gas atmosphere. For these purposes the green products from compaction is put in the furnace, and heated up for one and half hour under the argon gas atmosphere. Argon is purpose to prevent the products from oxidation. The temperature is set at 900°C. These process take about 3 hours, one and half hour for the furnace to reach up the desired temperature and one and half hour for heating at the desired constant temperature. First, the furnace was setting up to required parameters. Sintering temperature is set at 900°C and the sintering time is one (1) and half (1/2) hours. Secondly, the green sample is put into the furnace chamber and the door is closed and all the respective screws were tightened. Thirdly, the argon was flown into the furnace and the process start. After the heating process was finished, then the product was cooled down under standard room condition.

3.3.4 Sintering Environment

Sintering environment is the most important part in sintering process. It helps to prevent the product from oxidation that influent the properties of the composites at the end. Normally people use inert gas such as hydrogen or argon to control sintering environment. Before the heating process takes place, the argon gas is flown into the furnace to remove air. Then the heating process will start as the argon gas is continuously flow until the heating process finish.

CHAPTER 4

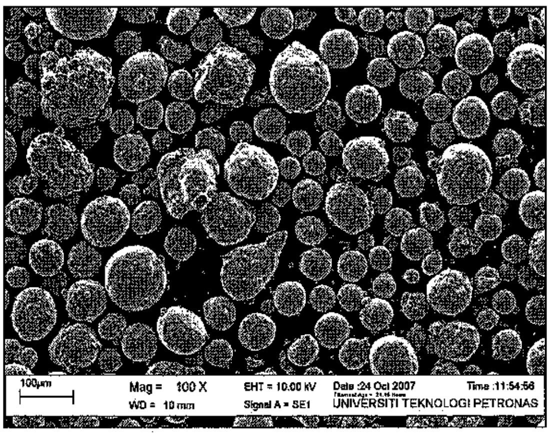
RESULTS AND DISCUSSION

4.1 Copper Powder Shape and Characterization

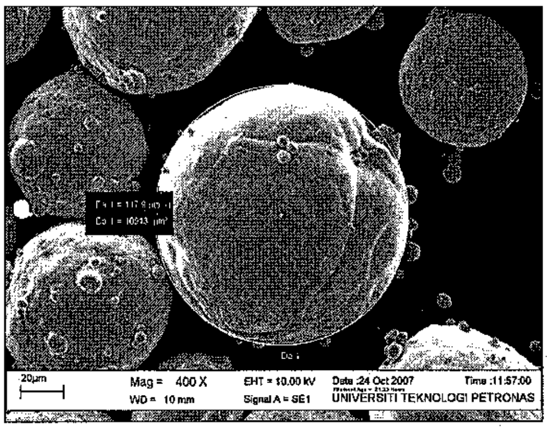
4.1.1 Copper Powder (Sample 1)

The shape of the powder particles is shown in the Figure 4.1 and also the particle size distribution. SEM image showed that the shape of powder particle is spherical. This gives a proof that the manufacturing of the copper powder is from the gas atomization process. In gas atomization, the molten stream of metal is struck by high pressure gas. This breaks up the metal stream [1].

While the Figure 4.4 shows the particle size distribution at a mean of 117.9 μm magnifying at 400X.



Copper particle size distribution at 100 X



Copper particle size distribution at 400 X

Figure 4.1: SEM image show size of the copper particle

Figures below shows the X-ray diffraction (XRD) result onto powder particle. The purpose is to define the existence of oxidation layer on the surface of powder particle. The red wave line is the spectrum of copper powder sample while the black wave line is pure copper which is in the database. Then the lines are matching up with each other. From this graph, the sample indicate that there is no oxide layer since the match is perfect but for confirmation, these wave line or spectrum are match up with the copper oxide spectrum.

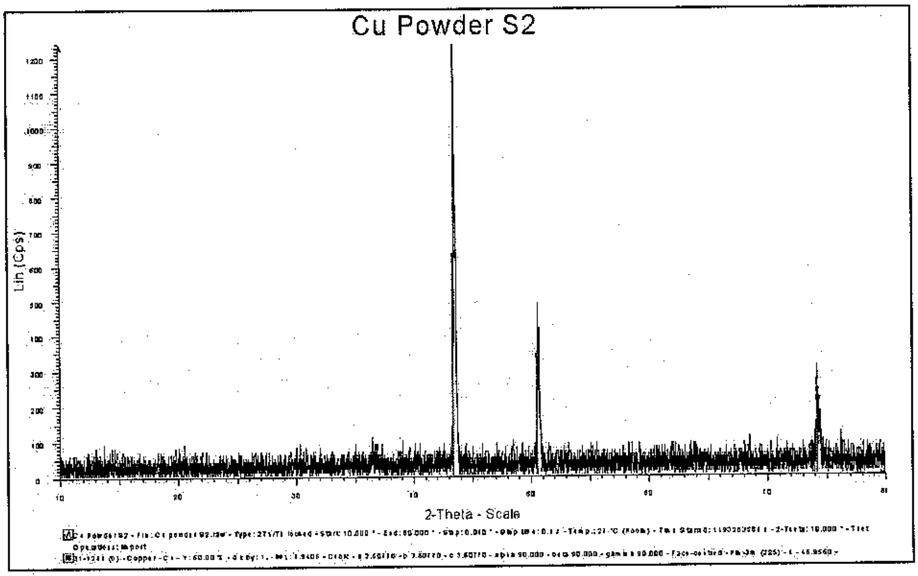


Figure 4.2: spectrum of copper powder

4.2 Density Measurement

4.2.1 Pure Copper Sample

Firstly the sample of 100% of copper is prepared. The sample is prepared using the Autopellet Press Machine. The sample is varied on their thickness with the same diameter that is 13mm of diameter. The thicknesses taken were 2mm.

By using the density formula, the mass of the copper is calculated and used. Taking the density of the copper is 8.96 g/cm^3. The theoretical volume of the sample can be calculated from the value of thickness and diameter of the pellet. The density value of the sample is in the table below:

Table 4.1: Properties of pure copper

For 2 mm of thickness				
Sample No.	Thickness Cm	Volume cm^3	Mass g	Density (ρ) g/cm^3
1	0.266	0.353068	2.324	6.5823035
2	0.236	0.313248	2.309	7.3711516
3	0.23	0.305284	2.189	7.1703663
4	0.232	0.307939	2.251	7.3098914
5	0.236	0.313248	2.315	7.3903058

Average volume	0.24	cm^3
Average mass	2.2776	g
Average density	7.164804	g/cm^3

4.2.2 CNT reinforced sample

After the pure copper samples have been prepared, the composite samples were the prepared. The composite prepared was 2 mm in diameter consist of both single walled nanotubes and also multi walled nanotubes. Using the rule of mixture, theoretical density of composites calculated.

Table 4.2: Composite density

1% MWNT			Theoretical Density (g/cm^3)
Sample	Mass (g)	Density (g/cm^3)	
1	2.325	8.757	8.215
2	2.253	8.486	
3	2.512	9.461	
4	2.274	8.565	

2% SWNT			Theoretical Density (g/cm^3)
Sample	Mass (g)	Density (g/cm^3)	
1	2.284	8.603	7.5
2	2.287	8.614	
3	2.063	7.770	
4	2.273	8.561	

1% SWNT			Theoretical Density (g/cm^3)
Sample	Mass (g)	Density (g/cm^3)	
1	2.325	8.757	8.215
2	2.253	8.486	
3	2.512	9.461	
4	2.274	8.565	

4.3 Sintered Sample

The sintering process is done in the Argon gas environment at the 850°C of temperature. In this process, three different times to sinter is being selected in order to identify the best time for sintering process to be done. The times selected are 45 minutes, 60 minutes and 90 minutes. After the sintering process completed, the sintered density of the composite is calculated.

Table 4.3: Sintering Result

45 minutes	
1% SWNT	
Mass (g)	2.304
Density (g/cm^3)	8.678

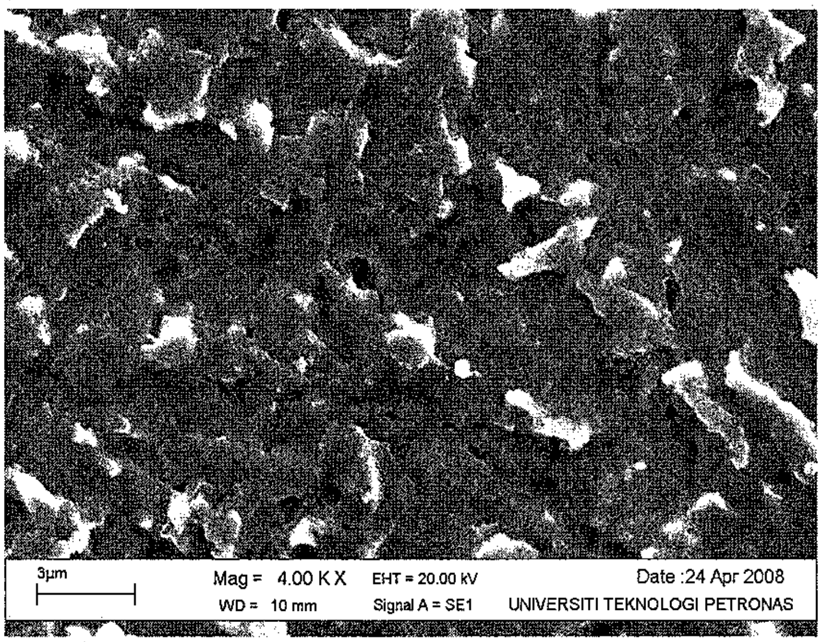
60 minutes		
	1% SWNT	2% SWNT
Mass (g)	2.26	2.216
Density (g/cm^3)	8.51	8.347

90 minutes		
	1% SWNT	2% SWNT
Mass (g)	2.221	2.24
Density (g/cm^3)	8.365	8.437

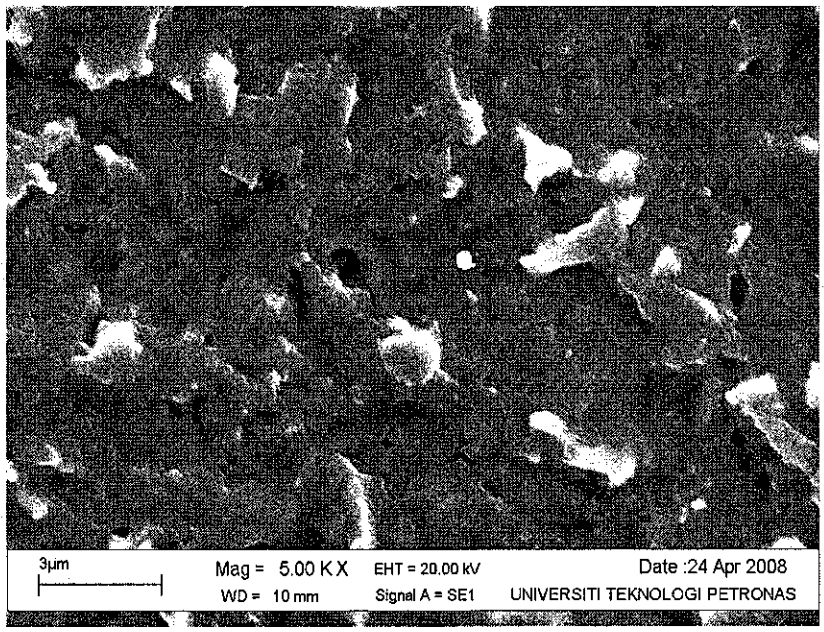
After the sintering process, to determine the best time for sintering, the samples microstructure is looked under the SEM.

Figure 4.3: Composite Microstructure

2% SWNT sintered for 45 minutes



4000 X magnification



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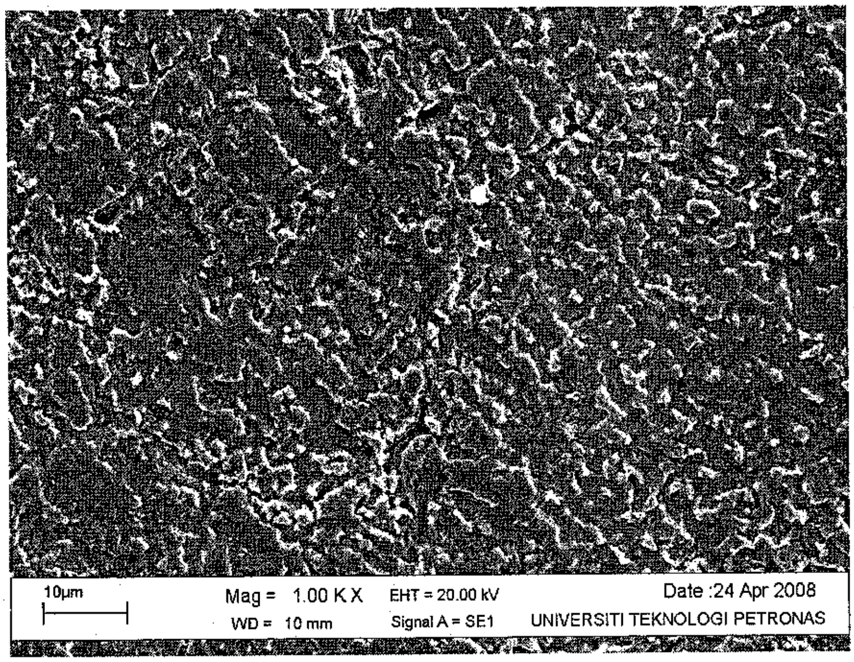
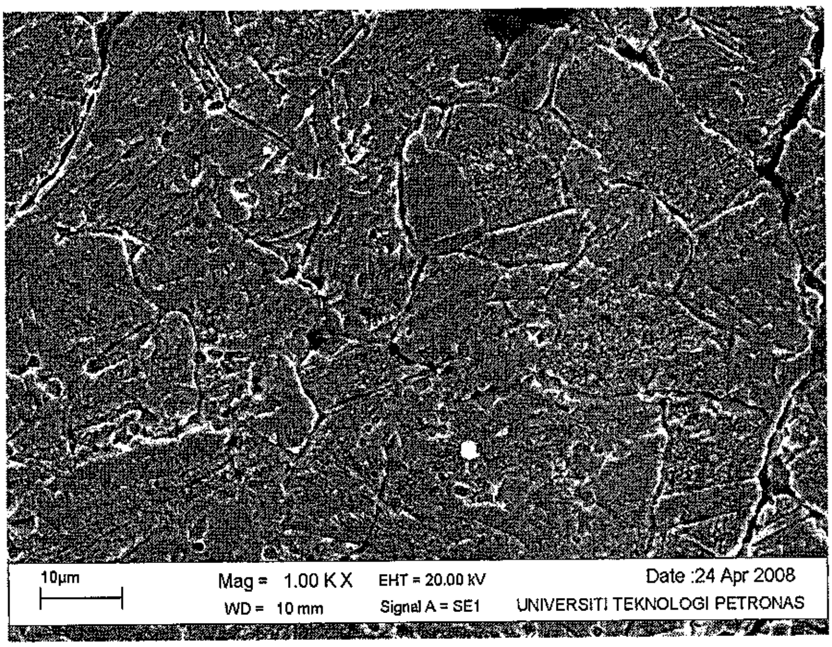
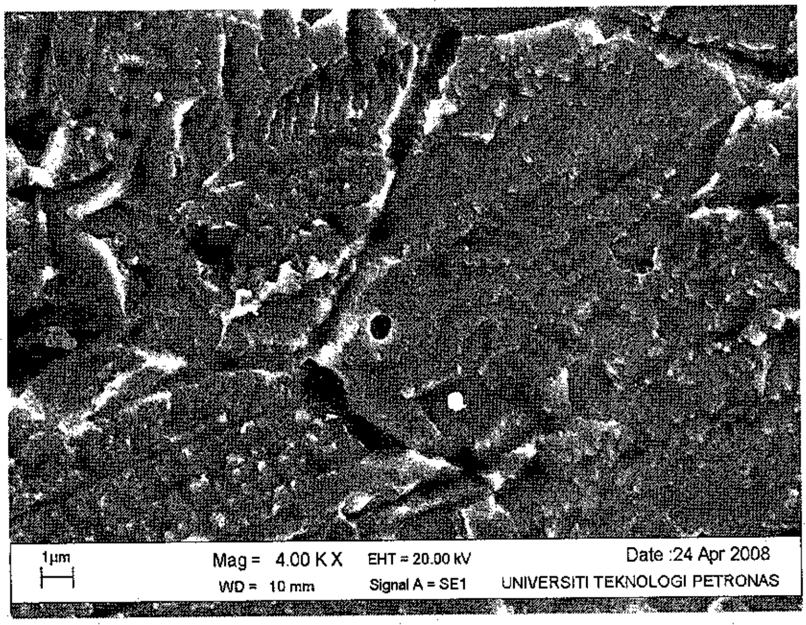


2% SWNT sintered 45 minutes under 1000 X magnification

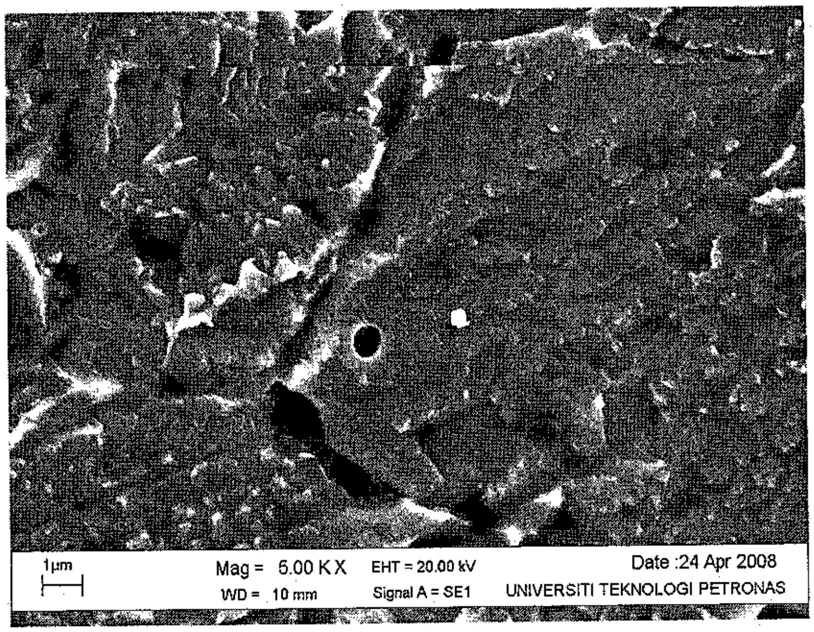
2% SWNT sintered for 60 minutes



1000 X magnification

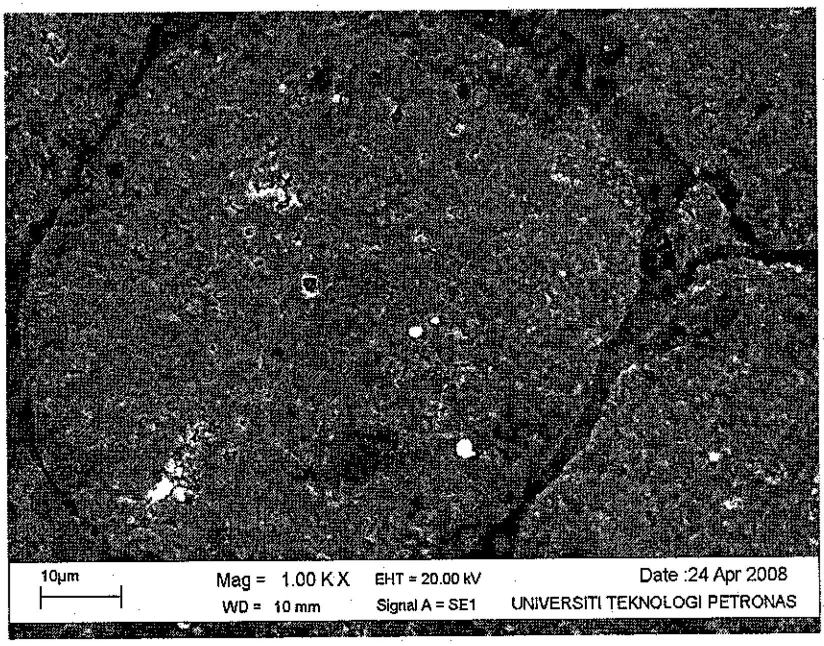


2% SWNT sintered 60 minutes under 4000 X magnification

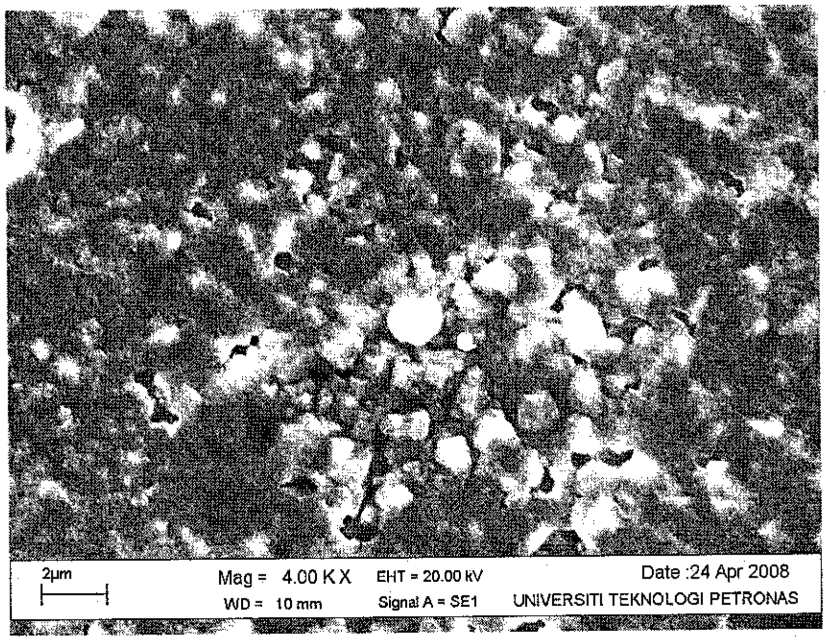


5000 X magnification

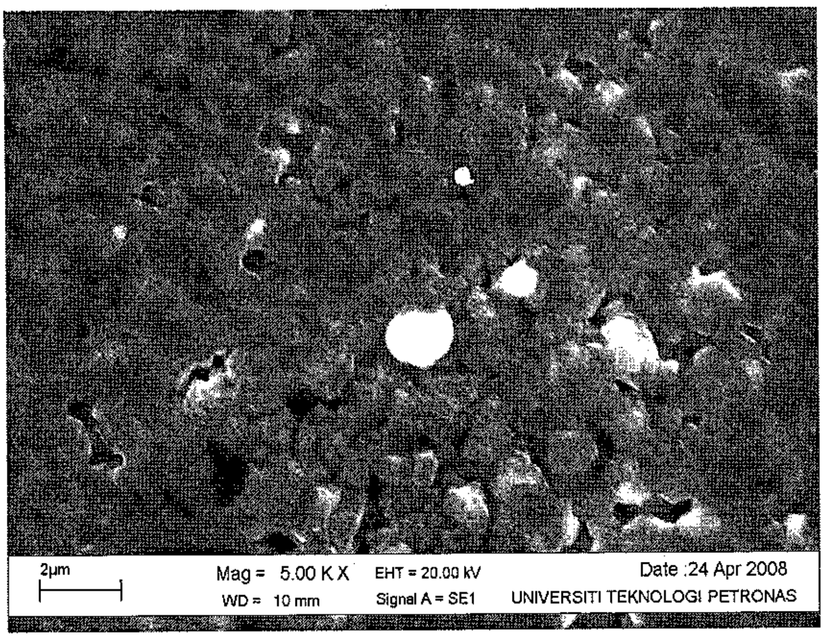
2% SWNT sintered for 90 minutes



1000 X magnification



4000 X magnification



2% SWNT sintered 90 minutes under 5000 X magnification

From the SEM result, the best time to sinter the CNT reinforced copper composite is 90 minutes.

There are slight different of the density between the theoretical and the sintered products because of some reasons:

a. Theoretical density is base on the exact density of copper.
b. The actual density of copper might be difference due to the production route of the copper. Manufacturers usually come with their own material data sheet. Same goes the properties of copper powder.

4.4 Thermal Conductivity

Theoretical calculations of thermal conductivity of the composites are based on the Eq 2.5 which is:

$$k_c = k_f V_f + k_m V_m$$

The result of increasing fiber volume fraction to the thermal conductivity value can be tabulated in table below.

Table 4.4: Theoretical Thermal Conductivity

Fiber Volume Fraction, V_f	Matrix Volume Fraction V_m	Thermal conductity of fiber, K_f (W/m.K)	Thermal Conductivity of Copper, K_m(W/m.K)	Thermal conductivity of Composite, K_c
1	99	3000	401	426.99
2	98	3000	401	452.98
3	97	3000	401	478.97
4	96	3000	401	504.96
5	95	3000	401	530.95
6	94	3000	401	556.94
7	93	3000	401	582.93
8	92	3000	401	608.92
9	91	3000	401	634.91
10	90	3000	401	660.9

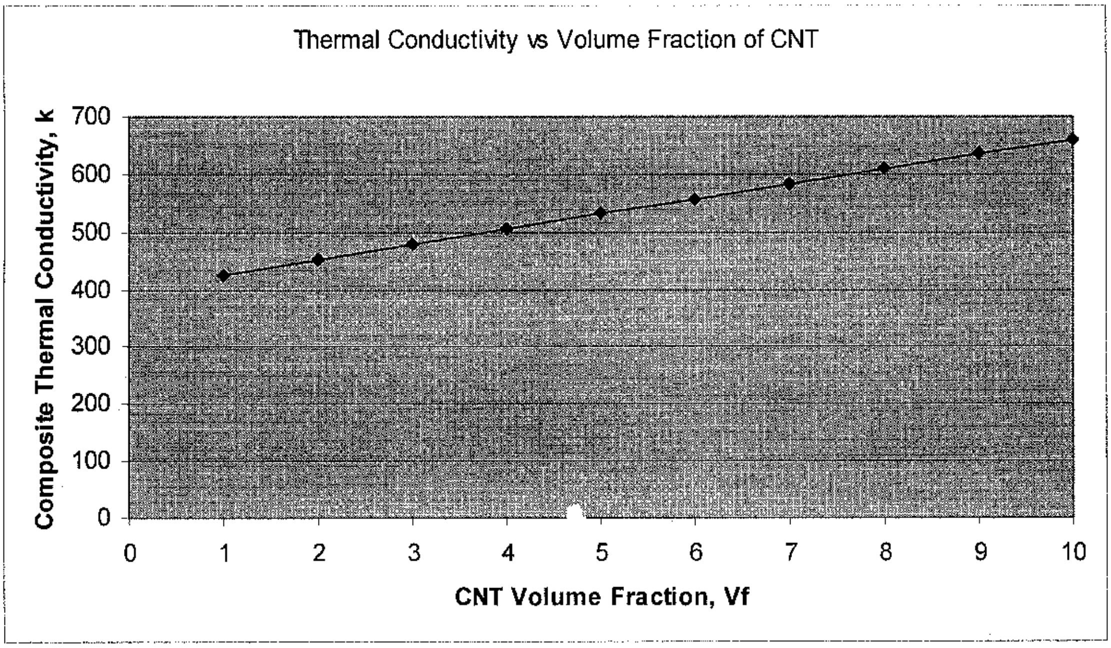


Figure 4.6: Graph for thermal conductivity versus CNT volume fraction

From the result calculated, it shows that the thermal conductivity of the carbon nanotubes reinforced copper composite is increase as the increase of the volume fraction of the reinforce material. The increment of thermal conductivity of the composite was approximately 60% as we compared the thermal conductivity of pure copper and the composite with 10% reinforced material.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATION

5.1 Conclusion

Carbon nanotubes reinforced copper matrix composites had been successfully developed through powder metallurgy and characterized their microstructure properties. With about 17 ton load, sufficient enough to produce near full density copper-fiber composite. Sintering temperature at 850°C in the argon gas atmosphere is sufficient to produce good sintered product at the 90 minutes sintered time. The powder particle diffusion bonding can be seen clearly in SEM images and also the carbon nanotubes imbedded in the powder particle bonding. There is a slight change of density between theoretical and sintered density, about 1% decrement due to some reason mentioned before. Furthermore, the reinforcement of the carbon nanotubes proves to enhance the properties of the copper composite such as density and thermal conductivity.

5.2 Recommendation

This project is to develop metal matrix composites that will have enhanced properties through powder metallurgy process. Some considerations need to be taken into account for further improvement of the project. For a better result in this project, a wider aspect of the experiment can be implemented. For example:

1. The usage of different types of CNT can be implemented
2. Other testing related to the composite can be done.
3. More specimens are prepared to get better results.

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