

CERTIFICATION OF APPROVAL

EFFECT OF SINTERING TIME ON PHYSICAL AND MECHANICAL PROPERTIES OF CARBON NANO-TUBES REINFORCED COPPER COMPOSITE

by

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A project dissertation submitted to the
Mechanical Engineering Programme
Universiti Teknologi PETRONAS
in partial fulfilment of the requirement for the
BACHELOR OF ENGINEERING (Hons)
(MECHANICAL ENGINEERING)

Approved by,

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UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

July 2010

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.

Zikri Fahmi Bin Zainon

ABSTRACT

Nowadays, the rapid growth of advance electronics device in electronics industry has its limitation in overheating problem. The solution for this constraint is by introducing the heat sinking material which has excellent properties such as higher strength, thermal conductivity and hardness. This report presents research of final year project, the Effect of Sintering Time on Physical and Mechanical Properties of Carbon Nanotubes reinforced Copper Composite. Carbon nanotubes were reinforced in 1% of volume fraction. The main objective of the project is to develop the samples of composites using a powder metallurgy (PM) process. One mixture of copper and carbon nanotubes was blended and compacted into metallic die under pressure of approximately 530MPa. The compacted samples were sintered at 850°C in argon environment for 1, 1 ½, 2, 2 ½ hour sintering time. This research focuses on the microstructures of sintered samples to determine the hardness and bonding between carbon nanotubes-copper. The results show that density of the composites is decreased after sintering process and the composites experience volume expansion due to grain growth which can be seen in the microstructure images result from optical microscopy and Scanning Electron Microscope. Hardness of the carbon nanotubes reinforced copper matrix is improved from the range of 62.01HV up 70.97HV with the increasing of sintering time.

ACKNOWLEDGEMENTS

In such a great of opportunity, I would like to express my acknowledgements and appreciations to those who had contributed and assisted in completing my Final Year Project (FYP).

A special acknowledgement is reserved for the project Supervisor, **Associate Professor Dr. Faiz Ahmad** for the coaching and guidance throughout the project completion. My greatest gratitude also goes to Mr. Muhammad Rafi Raza, a Post Graduate student for his cooperation in sharing the data and knowledge for the project.

My appreciation is extended to mechanical technicians especially Mr. Anuar, Mr. Faisal, Mr. Irwan and Mr. Shahrul for their assistance and help in completing the experimental work.

Last but not least, thanks to my beloved family and friends for their support and help through out this project.

The efforts of those involved in preparing this report are gratefully acknowledged.

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LIST OF ABBREVIATIONS

CF	Carbon Fiber
CNTs	Carbon Nanotubes
Cu	Copper
FESEM	Field Emission Scanning Electron Microscopy
HIP	Hot Isostatic Pressing
MIM	Metal Injection Molding
MMC	Metal Matrix Composite
MWCNT	Multi Walled Carbon Nanotubes
PF	Powder Forging
PM	Powder Metallurgy
ROM	Rule of Mixture
SEM	Scanning Electron Microscope
SWCNT	Single Walled Carbon Nanotubes
TEM	Transmission Electron Microscope

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Since from past few years, many new devices and technologies are developing through the innovation of new engineering materials or improvement of the existing materials.

One of the material technologies which is Metal Matrix Composites (MMCs), is results from the rapid growth in engineering material. Even though, MMCs are more expensive than the conventional materials processing they are replacing, they are found this process can improve properties and performance which bring a worthy value for the added cost. Today these applications are found most often in space systems and high-end, aircraft components, sports equipment or electronic devices.

The most popular method for fabrication MMCs is the infiltration of a preform by a liquid metal under pressure [1]. Since the project used powder form material, the solid state method of manufacturing MMC which is the Powder Metallurgy process is more suitable to produce the sample.

The process of P/M begins with homogeneous mixing of reinforcement with the powder matrix. The mixing is followed by compaction and then the compacted green samples are sintered in selected environments for densification of samples [2].

Generally, Powder Metallurgy techniques have an advantage with respect to microstructure control, material use, product homogeneity and mass production [3].

1.2 PROBLEM STATEMENT

High demand on light weight materials which have good properties especially in electronics industry required a detail review and analysis into the ways to improve the hardness properties of Copper by reinforcing Carbon Nanotubes (CNTs). The process involves mixing of CNTs with copper in a suitable proportion followed by compacting and sintering at high temperature. Copper is highly conductive material and by the reinforcing CNTs in copper, its hardness properties can be further enhanced since CNTs has an excellent thermal conductivity which is up to 3000W/m.K. Therefore these composite is suitable in electronics industry for heating removal purpose.

1.3 OBJECTIVES

The main objectives of this project are to develop the samples of Carbon Nanotubes (CNTs) reinforced Copper matrix by Powder Metallurgy technique and to study the microstructural of the sintered composites which had sintered at different time. The hardness properties of the composites will also be covered in this study.

1.4 SCOPE OF STUDY

The 1% Carbon Nanotubes reinforced Copper matrix and Pure Copper (for comparison purpose) samples will be developed using Powder Metallurgy process. Samples will sintered at different time which are at 1, 1 ½, 2, 2 ½ hour

and at constant temperature and atmosphere surrounding which are at 850⁰C and under Argon atmosphere. The effect of sintering time in the powder metallurgy process will be studied in the hardness properties of composites using microhardness testing. While the microstructural examination will be carried out using optical microscopy and scanning electron microscopy (SEM) to investigate the dispersion of Carbon Nanotubes (CNTs) in the copper matrix and the bonding between matrix and reinforced materials.

CHAPTER 2

LITERATURE REVIEW

2.1 CARBON NANOTUBES (CNTs)

Carbon nanotubes, long, thin cylinders of carbon, were discovered in 1991 by S. Iijima. These are large macromolecules that are unique for their size, shape, and remarkable physical properties. They can be thought of as a sheet of graphite (a hexagonal lattice of carbon) rolled into a cylinder. These intriguing structures have sparked much excitement in the recent years and a large amount of research has been dedicated to their understanding. Currently, the physical properties are still being discovered and disputed. What makes it so difficult is that nanotubes have a very broad range of electronic, thermal, and structural properties that change depending on the different kinds of nanotube (defined by its diameter, length, and chirality, or twist). To make things more interesting, besides having a single cylindrical wall (SWNTs), nanotubes can have multiple walls (MWNTs)--cylinders inside the other cylinders [4].

Carbon nanotube is the one of the strongest materials in nature. Carbon nanotubes (CNTs) are basically long hollow cylinders of graphite sheets. Although a graphite sheet has a 2D symmetry, carbon nanotubes by geometry

have different properties in axial and radial directions. It has been shown that CNTs are very strong in the axial direction. Young's modulus on the order of 270-950 GPa and tensile strength of 11-63 GPa were obtained [5].

The ratio of length to diameter of nanotubes can reach 100–1000 and their diameter is at about nanometer level. Therefore carbon nanotubes are considered excellent candidate reinforcing fibers for a variety of composite materials [6].

Carbon nanotubes and nanofibers have excellent mechanical characteristics such as high tensile strength and high elastic modulus, and also have high thermal and electric conductivity. Research into practical applications of carbon nanotubes and nanofibers has been actively pursued, and metal composites of such nanosized materials are promising new materials offering innovative functions. Powder carbon nanofiber–metal composites represent promising raw materials for powder metallurgy, such as powder rolling, powder flame spraying, and powder forging [7].

2.2 COPPER (Cu)

Copper is typically produced by a pyrometallurgical (high temperature) process. Other methods for copper extraction include leaching copper from low sulfur ores with a weak acid, then electrolytically extracting the copper from solution [8].

Copper was one of the first metals to be worked into implements and the first metal to be smelted from ores. Its excellent ductility and high conductivity assure its use in modern society.

Like gold and silver, copper is malleable. It can be bent and shaped without cracking, when either hot or cold. It can be rolled into sheets as thin as 1/500 of an inch. Copper also is ductile, it can be drawn out into thin wire. [9]

Industry valued copper for these properties. Copper is second material after silver in its ability to conduct electricity, but silver is too expensive for this sort of use. Bronze and brass, however, do not conduct electricity as well as pure copper [10].

Besides electricity, copper also is an excellent conductor of heat, making it an important metal in cookware, refrigerators, and radiators. Copper is resistant to corrosion, that is, it will not rust. If the air around it often is damp, it will change from its usual reddish orange color to reddish-brown [9].

2.3 METAL-MATRIX COMPOSITES (MMC)

Metal–matrix composites (MMCs) are metals that are reinforced with fibers or particles to improve or tailor properties such as stiffness, strength, thermal conductivity, thermal expansion, friction, and wear resistance [11].

MMC can be classified according to whether the reinforcement is continuous (monofilament or multifilament) or discontinuous (particle, whisker, short fibre or other). MMCs with discontinuous reinforcements are usually less expensive to produce than continuous fibre reinforced MMCs, although this benefit is normally offset by their inferior mechanical properties. Consequently, continuous fibre reinforced MMCs are generally accepted as offering the ultimate in terms of mechanical properties and commercial potential [12].

The wide variety of matrix and strengthening materials allows the designer of the product to choose an optimum combination. The matrix material can be introduced to the reinforcement before or after the reinforcement material is

placed into the mold cavity or onto the mold surface. The matrix material experiences a melding event, after which the part shape is essentially set. Depending upon the nature of the matrix material, this melding event can occur in various ways such as chemical polymerization or solidification from the melted state.

MMC manufacturing can be broken into three types: solid, liquid, and vapor [13].

Solid state methods

- Powder blending and consolidation (powder metallurgy): Powdered metal and discontinuous reinforcement are mixed and then bonded through a process of compaction, degassing, and thermo-mechanical treatment (possibly via hot isostatic pressing (HIP) or extrusion).
- Foil diffusion bonding: Layers of metal foil are sandwiched with long fibers, and then pressed through to form a matrix.

2.4 POWDER METALLURGY (PM)

There were three processing methods have been primarily used to develop MMCs: high-pressure diffusion bonding, casting, and powder-metallurgy techniques. More specifically, the diffusion-bonding and casting methods have been used for continuous- fiber reinforced MMCs. Discontinuously reinforced MMCs have been produced by powder metallurgy and pressure-assist casting processes.

Powder metallurgy is a forming and fabrication technique consisting of three major processing stages. First, the primary material is physically [powdered](#), divided into many small individual particles. Next, the powder is injected into a [mold](#) or passed through a [die](#) to produce a weakly [cohesive](#) structure (via [cold welding](#)) very near the dimensions of the object ultimately to be manufactured.

Finally, the end part is formed by applying pressure, high temperature, long setting times (during which self-welding occurs), or any combination thereof [14].

Under high pressure, nanotubes can merge together, trading some sp^2 bonds for sp^3 bonds, giving the possibility of producing strong, unlimited-length wires through high-pressure nanotube linking [5].

Sintering is the bonding of particles in a mass of powder by atomic or molecular attraction in the solid state through the application of heat. Powders differ from massive metals in having a much greater ratio of surface area to volume. Consequently, the surface energy is greater. During sintering, changes in the shapes of the pores and a reduction in their volume reduce the surface energy [15]

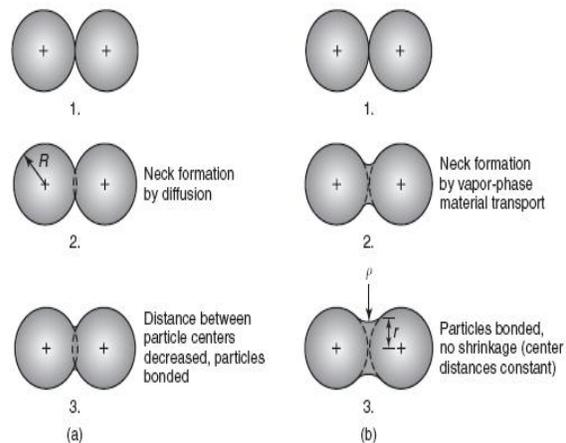


Figure 2.1: Sintering Process [16]

Sintering can be considered to proceed in three stages. During the first, neck growth proceeds rapidly but powder particles remain discrete. During the second, most densification occurs, the structure recrystallizes and particles diffuse into each other. During the third, isolated pores tend to become spheroidal and densification continues at a much lower rate. **Figure 2.2** illustrates the progress of densification of compacted copper powder as a function of time and temperature [15].

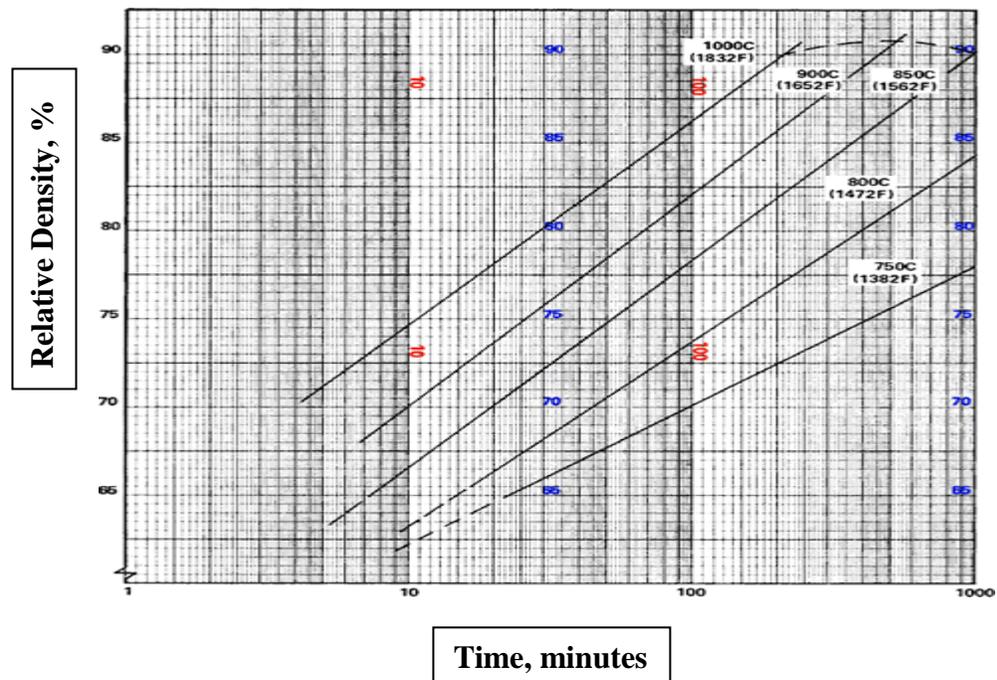


Figure 2.2 Effect of Sintering Temperature and Time on Densification of Copper Powder Compacts

2.5 RULE OF MIXTURE (ROM)

'Rules of Mixtures' are mathematical expressions which give some property of the composite in terms of the properties, quantity and arrangement of its constituents [17].

The properties of the composites, usually density can be predicted by the 'Rule of Mixtures' can be denoted as weight fraction or volume fractions. These

expressions are derived for a two-phase material and then generalized to a multiphase material.

The volume fractions and weight fractions are given by the equation below.

$$v_c = v_f + v_m \quad (2.5.1a)$$

$$V_f = \frac{v_f}{v_c}, \quad V_m = \frac{v_m}{v_c} \quad (2.5.1b)$$

and

$$w_c = w_f + w_m \quad (2.5.1c)$$

$$W_f = \frac{w_f}{w_c}, \quad W_m = \frac{w_m}{w_c} \quad (2.5.1d)$$

The density of the composites material can be obtained in order to establish conversion relations between the weight fractions and the volume fractions. From the basic equation of density, the mass divided by the volume.

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

The weight in equation Eq. (2.5.1c) can be replaced by the density and volume and equation written as:

$$\rho_c v_c = \rho_f v_f + \rho_m v_m \quad (2.5.2)$$

Dividing both sides in Eq. (2.5.2) by V_c and substituting Eq. (2.5.1b), the Eq. (2.5.2) can be rewritten as:

$$\rho_c = \rho_f V_f + \rho_m V_m \quad (2.5.3)$$

For the case of fiber-matrix composites, the equation Eq. (2.5.3) can be written as:

$$\begin{aligned} \rho_c &= \rho_f V_f + \rho_m V_m \\ &= \rho_f V_f + \rho_m (1 - V_f) \\ &= V_f (\rho_f - \rho_m) + \rho_m \end{aligned} \quad (2.5.4)$$

Since $V_f + V_m = 1$

where v_c, v_f, v_m represent the volume of composite, fiber and matrix material.

V_f And V_m represent volume fraction of fiber and matrix material.

w_c, w_f, w_m Denoted as weight of composite, fiber and matrix material.

W_f, W_m represent weight fraction of fiber and matrix material.

2.6 CARBON NANO-TUBES REINFORCED COPPER COMPOSITE

The relative density of CNT/Cu nanocomposites was calculated by the ratio of the measured Archimedes density relative to the calculated densities of the nanocomposite by the rule of mixtures. It was observed that the relative densities of the prepared CNT/Cu nanocomposites were decreased by increasing the CNTs volume fraction. The relative densities were 99.7 in case of 5 vol.% CNT/Cu, 99.1 in case of 10 vol.% CNT/Cu, 97.1 in case of 15 vol.% and 96.51 in case of 20 vol.% CNT/Cu. The relative density of CNT/Cu was lower than pure Cu due to agglomerations of CNTs at grain boundaries of the copper matrix which is the origin of the pores formation. These agglomerations increased by increase the CNTs volume fraction [18].

The thermal conductivity of the carbon nanotubes (CNTs) ranges between 1200-3000 W/m.K are the best candidate for the development of heat sink material. This research investigates the effects of reinforcing CNTs were prepared and pressed under compacting pressure of 580 MPa. The compacted samples were sintered in the inert atmosphere at 900°C. Sintered density of copper composites was measured and compared with theoretical density calculated using Rule of Mixtures (ROM). The results showed 96-98% sintered density was achieved. The sintered compacts showed good grain growth which resulted in approximately 1% increase in dimension of specimen. However, in few samples, porosity was also noted. Field emission scanning electron microscopic (FESEM) study showed well dispersion of CNTs in copper matrix and interfacial bonding between copper particle and CNTs. In this experiment, the addition of 2 V/o of CNTs in copper resulted in 9% increase in the thermal conductivity of sintered copper [2].

Compacted samples were sintered for densification of powder particles at 850°C to 900°C and sintering time was varied between 45-90 minutes. All the samples were sintered in the argon gas to avoid the surface contamination [2].

CHAPTER 3

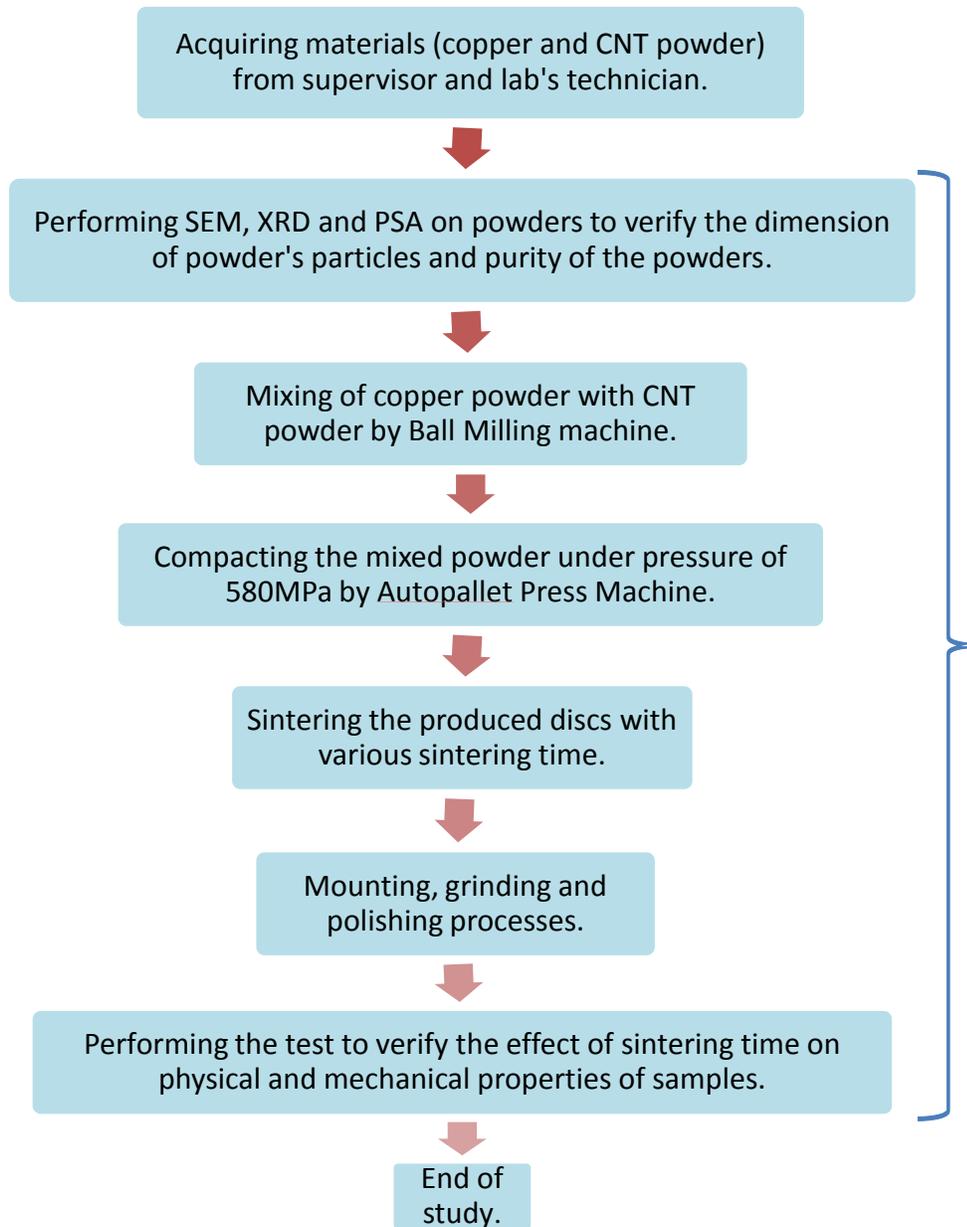
METHODOLOGY

3.1 PROJECT FLOW

The project begins with gathering all information by researching and understanding on the fundamentals of metal matrix composites, powder metallurgy process and sample preparation for testing. All information about the materials, equipment and experimental procedure were gathered in the first semester of the final year project. The flow of the research processes is simplified in **Figure 3.1**.

Next step of this project is the preparation of samples by powder metallurgy technique. Then the analysis of the microstructure will be done on samples to characterize the bonding of composites. The last part was the hardness testing of the samples using micro hardness indentation test.

All the experimental work is done in Block 17 of Mechanical Engineering Building with the assistance of mechanical technicians.



Experimental

Work

Figure 3.1 Flow chart of Project.

3.2 SAMPLE PREPARATION

3.2.1 Materials and preparation of CNTs and copper powder.

Matrix and reinforcement material are prepared which the samples volume fraction of CNTs is 1%. The mass and theoretical density of the composites samples were calculated using Rule of Mixture (ROM). Refer **Appendix II** for detail calculation.

3.2.2 Mixing

The accurately weighted matrix and reinforced material is mixed uniformly. This process was done manually by using 400ml beaker and Ball Mill mixing for one (1) hour and above to get smooth mixed powders.

3.2.3 Compaction of mixture

The mixture of the CNTs and copper powders are compacted using auto pellet press machine at ambient temperature in a circular die to produce a disc shape of solid green sample with average about 3mm thickness and 13mm diameter. The samples were pressed under compacted load of 7000kg or approximately about 530MPa pressure.

The dimension of the compacted samples are measured by using vernier caliper and recorded to analyze the sample size and volume before and after sintering process.



Figure 3.2: Auto pellet Press Machine used for compaction

3.2.4 Sintering

The compacted samples or known as 'green samples' are sintered in furnace tube for densification of powder particle below the matrix's melting point (70-80% melting temperature) which is 850°C for the composites. This process took half an hour for furnace to heat from room temperature up to 850°C with heating rate of 27°C per minute. Then the samples were heated at constant 850°C temperature for different sintering time which are at 1, 1 ½, 2, 2 ½ hour for each every different samples. The argon gas in the furnace is control by flow rate of 1100cm³ per minutes.

After the heating process is completed, the samples were cooled down under standard room temperature. All samples were sintered in the argon gas environment to avoid the surface contamination [2].

Then the dimension of the sample are measured again and recorded to analyze the sample size and volume before and after sintering process.

Note: The furnace need to be purge for 15 minutes before start the heating process.

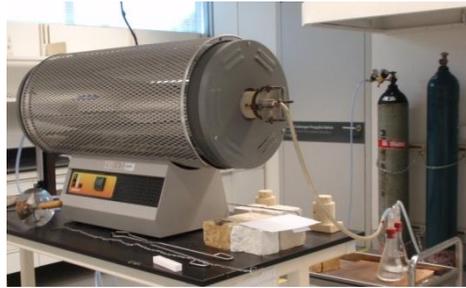


Figure 3.3: Tube Furnace used for sintering

3.2.5 Hot Mounting

After the samples were sintered, the samples were then mounted by using Buehler, Simpliment 1000 mounting machine. Thermosetting polymeric powder used is phenolic powder. The phenolic powder is placed in the mould with samples then was heated for 2min and cooled for 5min under the pressure of 3500psi.



Figure 3.4: Auto Mounting Pressing Machine used for mounting

3.2.6 Polishing and Grinding

Grinding and polishing process were performed by using Grinder and Polisher machine model Metaserv 2000. The samples were ground with SiC paper and running water. The SiC paper used ranging from 320 grits to 2400 grits.

The samples are polished with the rough polish started with 6micron and then with the 1 micron diamond compound. The speed for both processes is 150 to 200 rpm.



Figure 3.5: Grinder and Polisher Machine used for grinding and polishing samples

3.3 SAMPLE TESTING

3.3.1 Microscopic Examination

The etching process was done first on samples before microscopic examination. Then the microstructures of composites were analyzed by using metallurgy optical microscope and scanning electron microscope (SEM).

The model for metallurgy optical microscope is Zeiss and the magnification employed in this experiment is 20X. For scanning electron microscope the magnification employed is 500X.



Figure 3.6: SEM used to get the microscope image of the samples surface

3.3.2 Hardness Test

Then hardness of the composites was measured by conducted Micro hardness testing using Leco LM 247 AT, microhardness machine. The unit of hardness

used was Vickers Pyramid Number. The load used was 100gf with the magnification of 50X.

The hardness reading for each sample was taken at ten (10) different locations of the sample's surface to get the most accurate average hardness of the composites. These hardness values provided the data to estimate the yield strength properties of the nanocomposite.



Figure 3.7: Microhardness Testing Machine used for indentation test

CHAPTER 4

RESULTS AND DISCUSSION

4.1 PARTICLE SHAPE AND SIZE DISTRIBUTION

Copper and CNTs powders were characterized by using Scanning Electron Microscope to determine their particle shape and size distribution of the used elements.

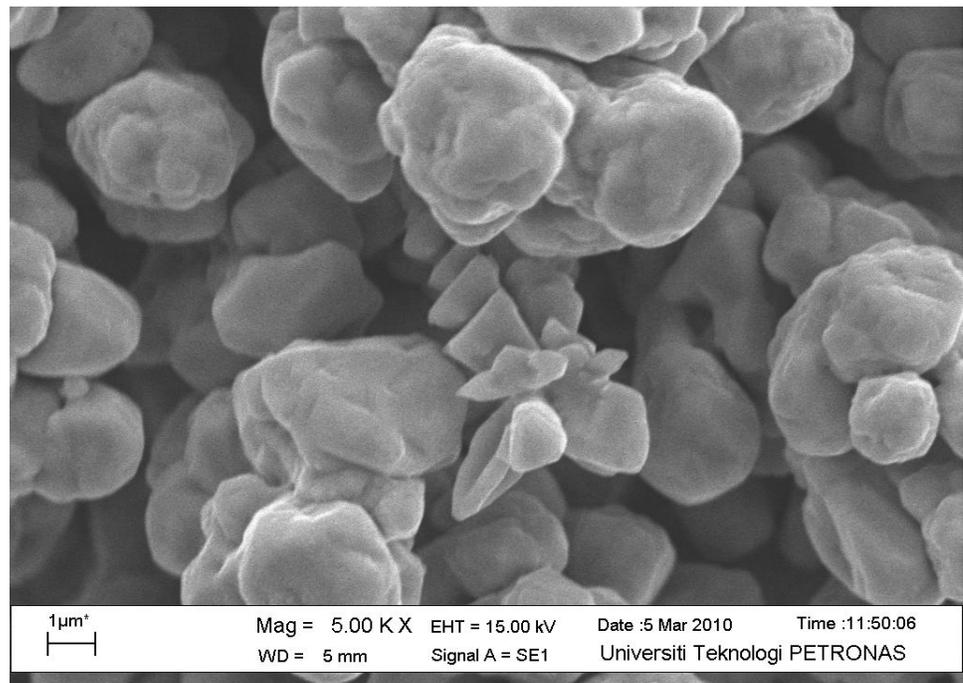


Figure 4.1: Particle shape of pure copper powder

Figure 4.1 show a scanning electron micrograph image of spherical shape copper powder used for this experimental research. This image is 100x magnifications. The particles size ranges approximately from 10 μm to 120 μm. From the shape this shows that this copper powder was produced by gas atomization process. However, there are some of the particles are not in spherical shape with rough and irregular surface. Particle size and shape are the important criteria in producing green product with better packing and improved density during final compaction [19].

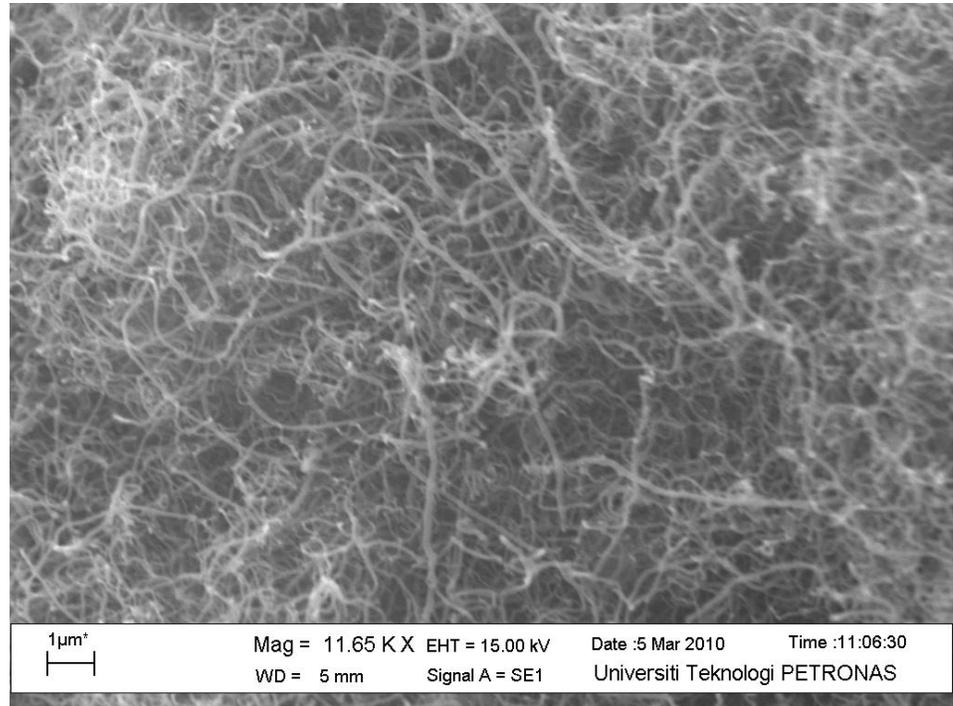


Figure 4.2: Particle shape of CNTs powder

Figure above shows the SEM image of long hollow cylinders shape of CNTs powder which has been use in this project. Its typical ratio of length to diameter of nanotubes can reach 100–1000 and their diameter is at about nanometer level. Therefore carbon nanotubes are considered excellent candidate reinforcing fibers for a variety of composite materials [6].

4.2 PHYSICAL CHARECTERIZATION

4.2.1 Dimensional changes

After the sintering process, the dimension of the samples were measured and compared with the dimension of green density samples. As the result, the dimensions of the samples differ between green sample and sintered sample. Diameter and thickness of the disc shape samples increased or expand after sintering process. During the sintering process, the mass flow of the copper molecule moves to fill in the porosity and defect in the material and resulted the densification occurred. However, the increasing of the samples dimension happened because due to the grain growth of the composites.

From the sample observation, some cracks appear at the wall surface of the disc shape sample. This crack formed during the sintering process due to the solid carbon which inside the pores of the parts precipitates from the carbon monoxide in the endogas. This process happened due to inhomogeneous of the composites material between Copper and reinforcement which is CNTs.

The existence of the cracks on the sintered samples is due to the thermal different coefficient between the copper and reinforced materials.

Refer to appendices for the value of the samples' dimensions.

4.2.2 Density

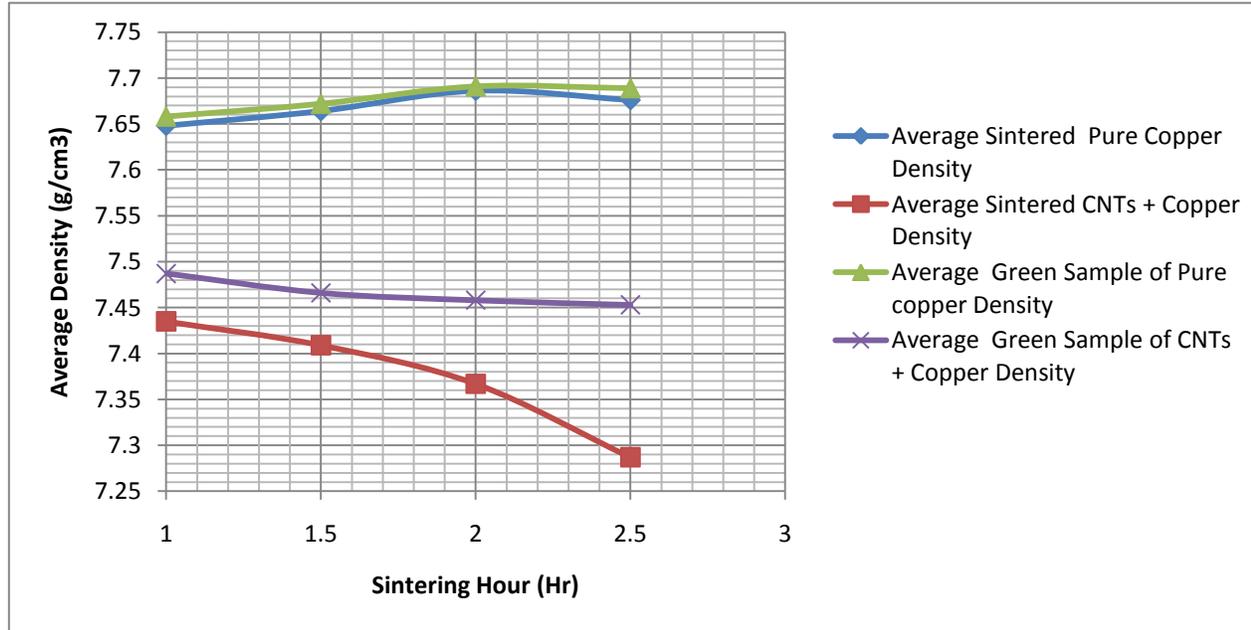


Figure 4.3: Density of green and sintered of pure copper and Cu-CNTs composites for various sintering time

Above figure (**Figure 4.3**), shows that density of the composites decreased by increasing the sintering time and there are slightly differences between green and sintered density of composites and pure copper samples. The reduction of composites' density with the increasing of sintering time is due to the rapid growth of grain boundaries of the material. If the longer sintering process took place, more grain boundaries will develop and as a result the density will be reduced.

Theoretical density is calculated using rule of mixture base on the exact density of copper and reinforcement (CNTs) materials properties (refer appendix for the calculation). Therefore, the result was found that the green density is lower than

the theoretical density. This is also because the production route of the materials and sample preparation were affects the actual density.

The green density of Cu-CNTs was found in the range of 7.453 g/cm³ to 7.487 g/cm³ while for sintered density of Cu-CNTs is 7.287 g/cm³ to 7.435 g/cm³. The sintered density was lesser than the green density of the composites because of the dimensional increase of samples after the sintering process.

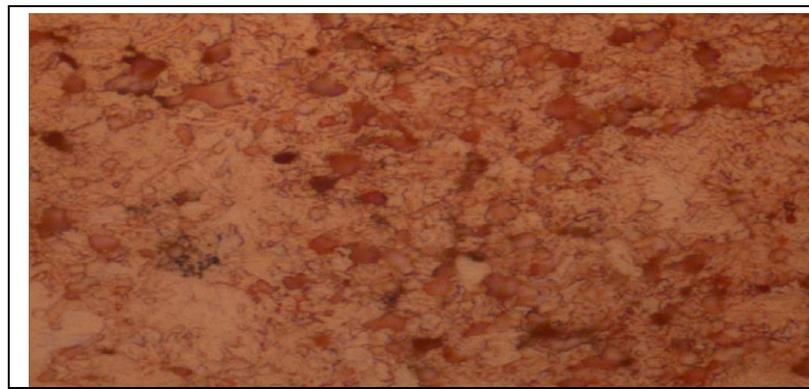
The green and sintered density is calculated from the mass of samples divide by measured volume of the composites samples.

$$\rho = \frac{m}{v} \quad \text{where} \quad \begin{array}{l} \rho = \text{density} \\ m = \text{mass} \\ v = \text{volume} \end{array}$$

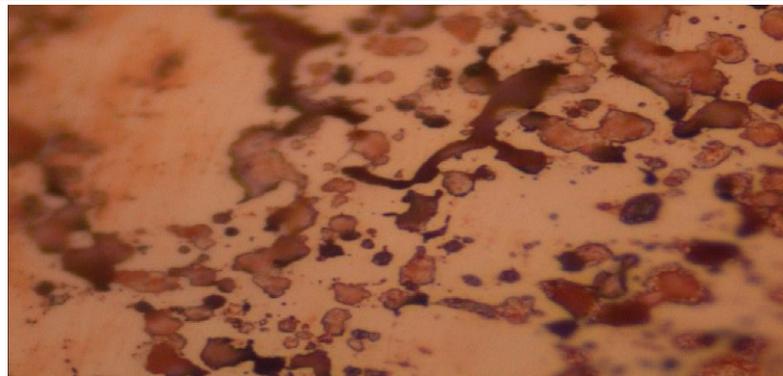
Since volume of the samples increase, the density decreased.

4.3 MICROSTRUCTURE OF SINTERED SAMPLES

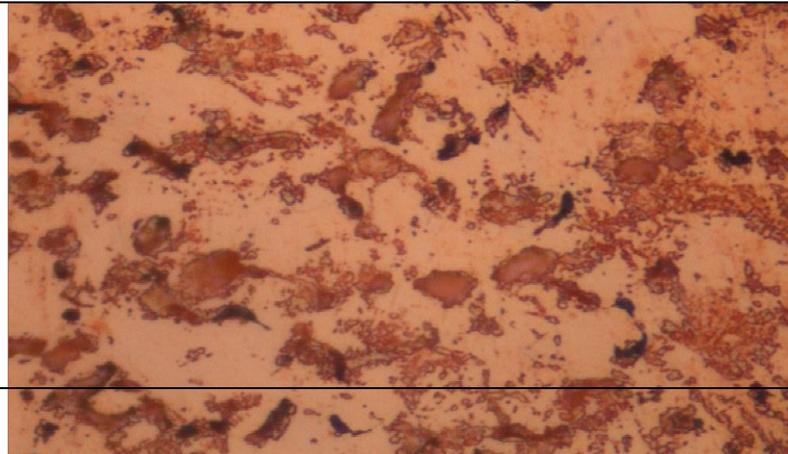
4.3.1 Optical Microscopy



Pure Copper 2 Hour Sintering Time



Cu-CNTs 1 hour Sintering Time



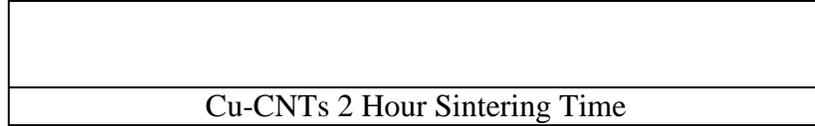


Figure 4.4: Optical micrograph of samples composites which sintered at 850°C under Argon atmosphere : 150 X

An optical micrograph surface of the composites samples with 150 magnifications are shown above in **Figure 4.4**. The micrograph specifies the

grain growth and diffusion of copper particle with the reinforcement material as a result from the sintering process. The twin boundaries occur between crystallites. This indicates that recrystallization also happen during the sintering process. However, there are some pores lying near the boundary. This behavior will lead to ductility properties of the material.

4.3.2 Scanning Electron Microscopy

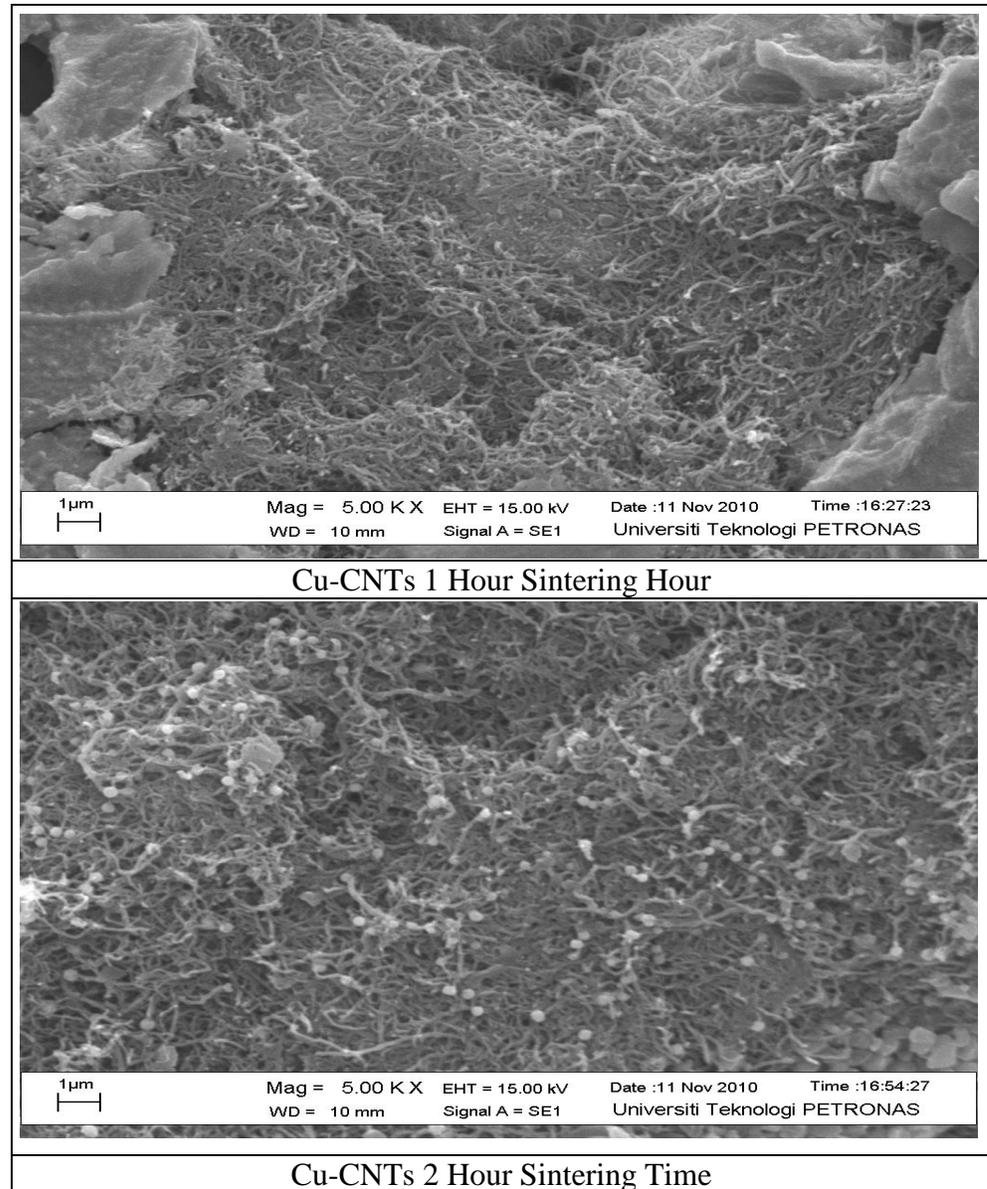


Figure 4.5: SEM image of sintered Cu-CNTs which sintered at 850⁰C under Argon atmosphere : 5K X

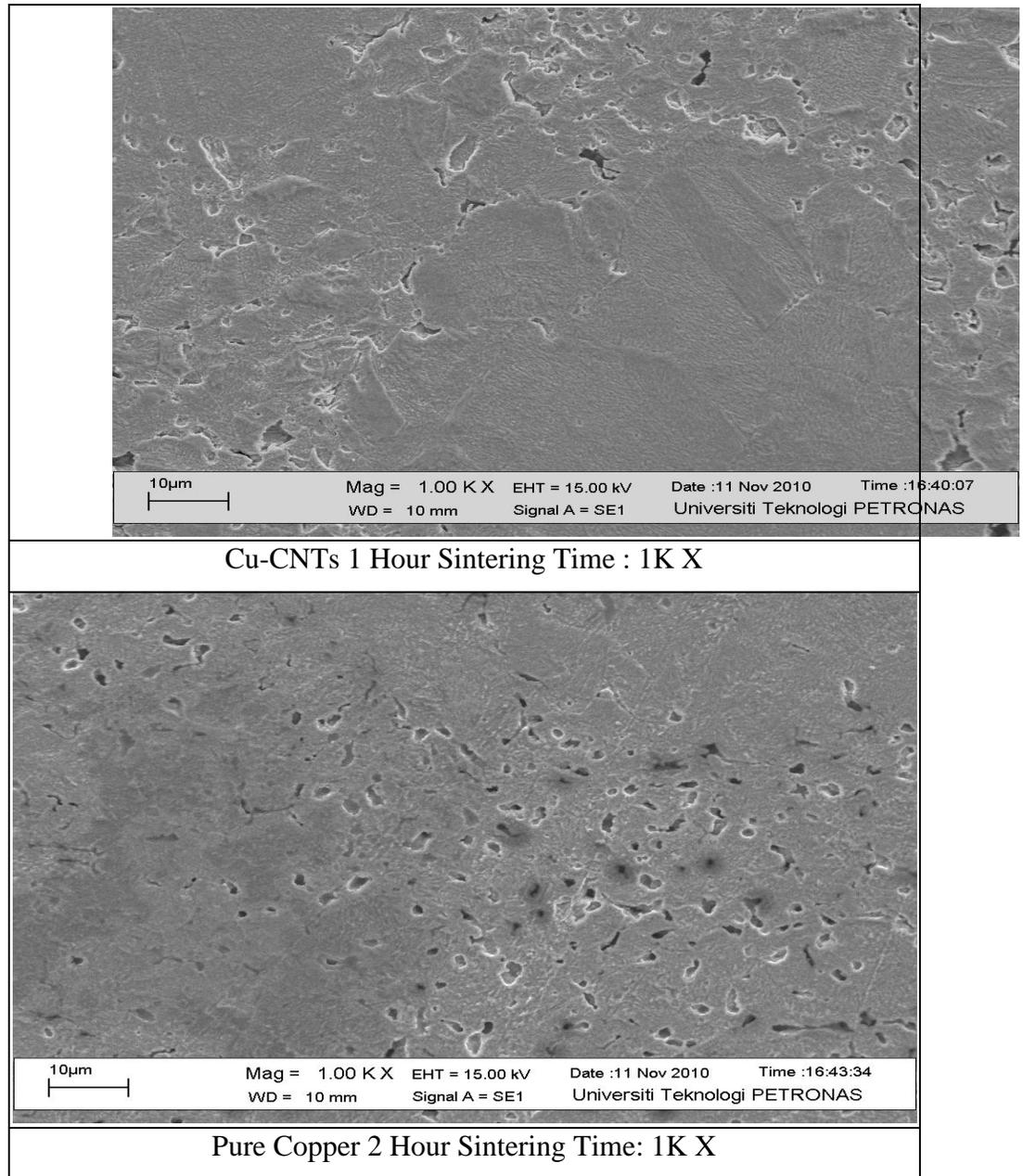


Figure 4.6: SEM micrograph of samples composites which sintered at 850°C under Argon atmosphere

From the micrographs above (**Figure 4.5 and 4.6**), it shows the diffusion of powder particles are well distributed. The micrographs also showed grain growth of copper powder. However, there are some pores are spotted at few location of the surface's samples. Micrograph also shows the leftover of CNTs from the

result of grinding and polishing process, where the CNTs were removed from the surface and leaving small pits.

4.4 HARDNESS OF SINTERED SAMPLES

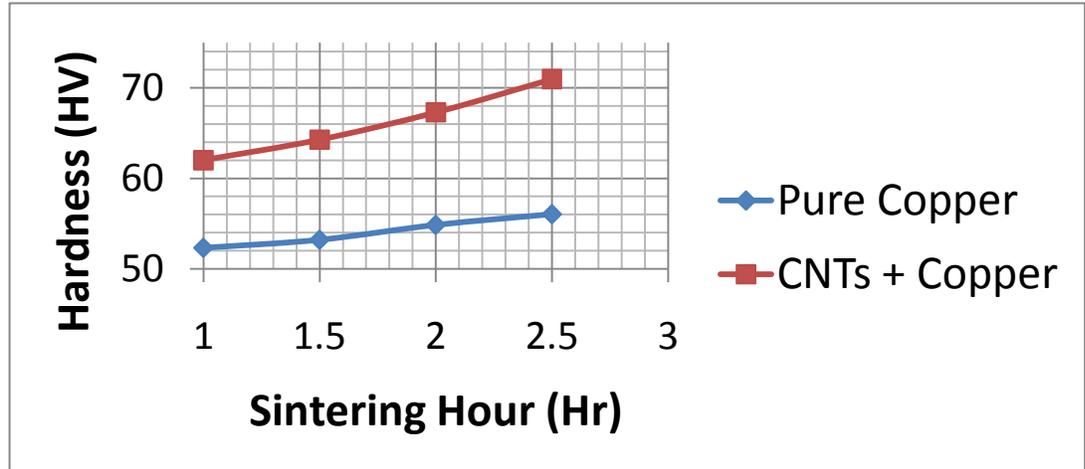


Figure 4.7: Relationship between the hardness and the sintering time of pure copper and Cu-CNTs samples

The hardness curves basically specify how strong the intermolecular bond between the particles. Usually the hardness value which obtained from the microhardness testing is used to determine the yield strength of the composites. In order to convert HV to MPa below equation is used;

$$\text{Tensile Strength (MPa)} = 3.45 \times \text{Hardness (HB)}$$

The values of hardness which obtained from the test need to be convert into another unit first which is in HB. By referring to the hardness table, the value possibly converted by using interpolation method.

Figure 4.6 shows that the hardness values for Cu-CNTs composites are increase with the sintering time. As the sintering time is longer, the grain size was increased. Therefore the hardness of the samples was increase as well.

Refer appendices for the tensile strength values of the samples.

4.5 SIGNIFICANCE OF THE STUDY

The importance of this study is mainly about to fill the need of the industry in order to develop light weight, low cost, and high performance of Copper metal matrix composites using Carbon nanotubes particle as reinforcement.

Nanotechnology such as nanocomposites can make a contribution to the following areas:

- Damage protection
- Optimizing of existing product
- A more efficient use of materials
- Reduced need for maintenance and/or operational upkeep
- Reduction in weight and/or volume

And as a direct result:

- Reduction in the consumption of raw materials and energy
- Conservations of resources
- Greater economy

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSION

The objective of this project was completely achieved where the development samples of Carbon Nanotubes (CNTs) reinforced Copper matrix composites were successfully done by using powder metallurgy process with 70KN or approximately about 530MPa load of compaction and sintered at 850°C with four different sintering times.

The existence of reinforced materials helps industry to provide lightweight material by reducing the density of materials. The physical behavior of the composites were investigated and explained by analyzed the microstructure of the composites surface. The microstructure behaviors reflect the hardness of the composite. Sintered Copper and CNTS composite show the high hardness values since the grain was enlarge during the sintering time.

5.2 RECOMMENDATION

In order to improve the diffusion bonding between the copper powder and reinforced materials (CNTs) during the mixing process, a type of binder should be used. For example, copper powder need to be coated with the reinforcement using electroless copper coating during the mixing process to get a better mixture of mixed powder.

Extended effort on the testing methods such as TEM or FESEM on the Copper and CNTs to see the clearer images and bonding between the composites material will be give more valuable information for this project.

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APPENDIX I
Properties of research materials

Properties of research material

Material	SWCNTs (f)	Copper, Cu (m)
Properties		
Density, ρ (g/cm³)	1.4	8.94
Melting temperature, T_m (°C)	\approx 3400	1083.4
Young's Modulus	\approx 1.5TPa	110 - 128 GPa
Yield Strenght	62000MPa	70 MPa
Thermal Conductivity	\approx 3000W/m.K	401 W/m.K

APPENDIX II

Sample calculation of Volume, Mass, and the theoretical density of the composites

Calculation

Composite volume is calculated from the sample volume such below dimension:

Thickness, t : 3mm

Diameter, D : 13mm

Composite Volume, v_c

$$\begin{aligned}
&= \pi \times r^2 \times t \\
&= \frac{\pi D^2}{4} t \\
&= \frac{\pi \times 13^2 \times 3}{4} \\
&= 398.20 \text{ mm}^3 \\
&= 3.982 \times 10^{-7} \text{ m}^3
\end{aligned}$$

At $V_f = 1\%$ $V_m = 99\%$

$$\begin{aligned}
V_f &= \frac{v_f}{v_c} \\
v_f &= V_f v_c
\end{aligned}$$

Volume of fiber (CNTs), v_f

$$\begin{aligned}
&= 0.01 \times (3.982 \times 10^{-7}) \\
&= 3.982 \times 10^{-9} \text{ m}^3
\end{aligned}$$

$$\begin{aligned}
V_m &= \frac{v_m}{v_c} \\
v_m &= V_m v_c
\end{aligned}$$

Volume of matrix (Copper), v_m

$$\begin{aligned}
&= 0.99 \times (3.982 \times 10^{-7}) \\
&= 3.942 \times 10^{-7} \text{ m}^3
\end{aligned}$$

From the density-mass relation, we can find the mass portion of the reinforcement and matrix material in the composites.

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

$$m = \rho v$$

$$\begin{aligned} m_f &= \rho_f v_f \\ &= (1.4 \times 10^6) \times (3.982 \times 10^{-9}) \\ &= 0.0056 \text{ g} \end{aligned}$$

$$\begin{aligned} m_m &= \rho_m v_m \\ &= (8.94 \times 10^6) \times (3.492 \times 10^{-7}) \\ &= 3.122 \text{ g} \end{aligned}$$

The theoretical density of the composite can be calculating using equation (2.5.3);

$$\begin{aligned} \rho_c &= \rho_f V_f + \rho_m V_m \\ &= (1.4 \times 10^6)(0.01) + (8.94 \times 10^6)(0.99) \\ &= 8.865 \times 10^6 \text{ g/m}^3 \\ &= 8.865 \text{ g/cm}^3 \end{aligned}$$

APPENDIX III
Dimensions change of composites samples

Dimension of green and sintered Cu-CNTs composites

Sintering Time	Volume Green Cu-CNTs (mm ³)	Volume Sintered Cu-CNTs (mm ³)	Increment (%)
1	444.78	486.09	9.29
1 1/2	451.49	493.27	9.25
2	446.06	492.96	10.51
2 1/2	440.74	495.67	12.46

APPENDIX IV
Density variations of composites samples

Density variations of the pure copper and Cu-CNTs composites samples

Sintering Time	Average Green Sample of Pure copper Density (g/cm^3)	Average Sintered Pure Copper Density (g/cm^3)	Average Green Sample of CNTs + Copper Density (g/cm^3)	Average Sintered CNTs + Copper Density (g/cm^3)
1	7.658	7.648	7.487	7.435
1 1/2	7.672	7.664	7.466	7.409
2	7.691	7.686	7.458	7.367
2 1/2	7.689	7.676	7.453	7.287

APPENDIX V
Hardness value of the composites

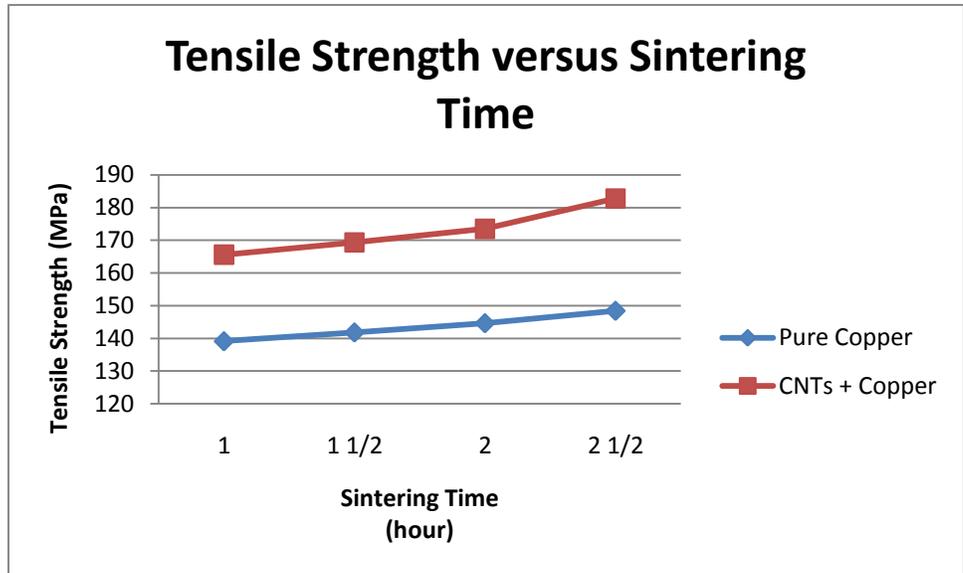
Sintering Time	Pure Copper (HV)	CNTs + Copper (HV)
1	52.33	62.01
1 1/2	53.22	64.27
2	54.86	67.29
2 1/2	56.05	70.97

Sintering Time	Pure Copper (MPa)	CNTs + Copper (MPa)
1	139.14	165.60
1 1/2	141.83	169.36
2	144.66	173.50
2 1/2	148.45	182.75

Hardness of the Cu-CNTs composites

Tensile Strength of the samples

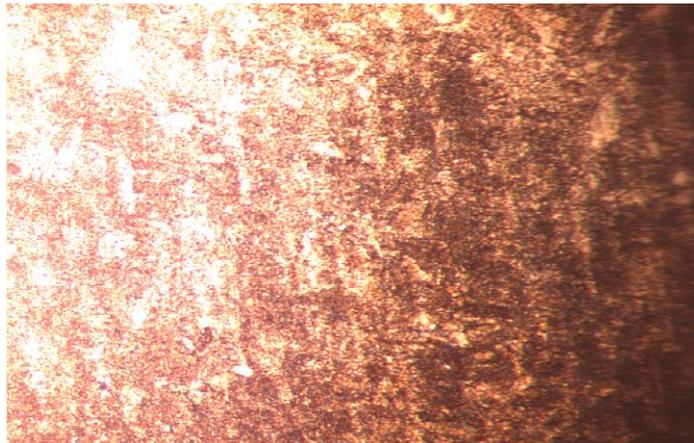
Graph of tensile strength versus Sintering time of composites



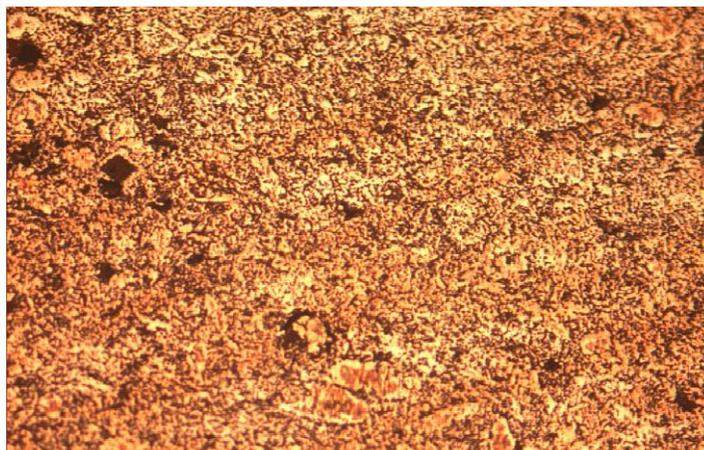
APPENDIX VI
Micrograph of SEM and OM

Optical Microscopy

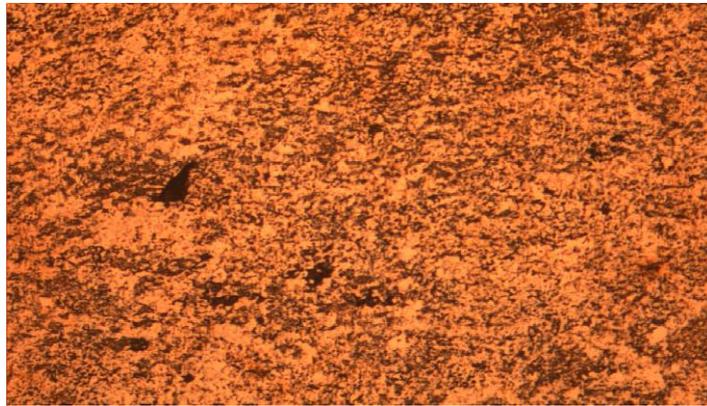
10 Magnifications



Pure Copper 2 Hour Sintering Time under 850⁰C and Argon atmosphere

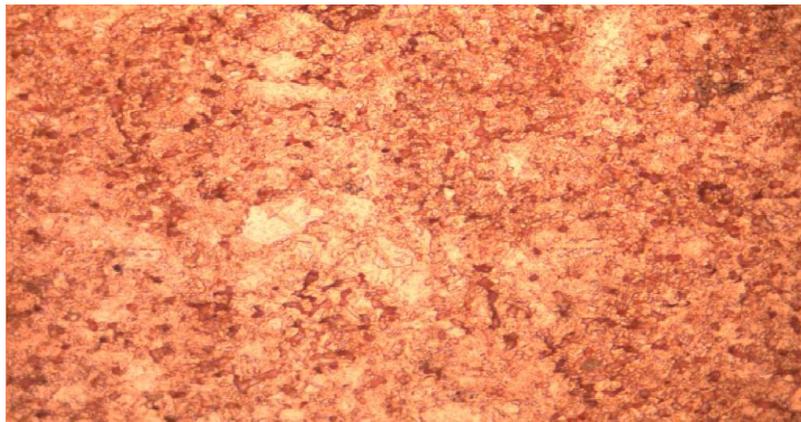


Cu-CNTs 1 Hour Sintering Time under 850⁰C and Argon atmosphere

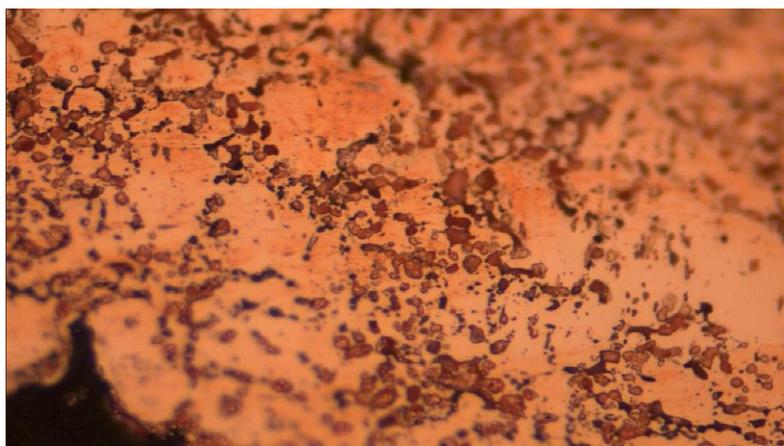


Cu-CNTs 2 Hour Sintering Time under 850⁰C and Argon atmosphere

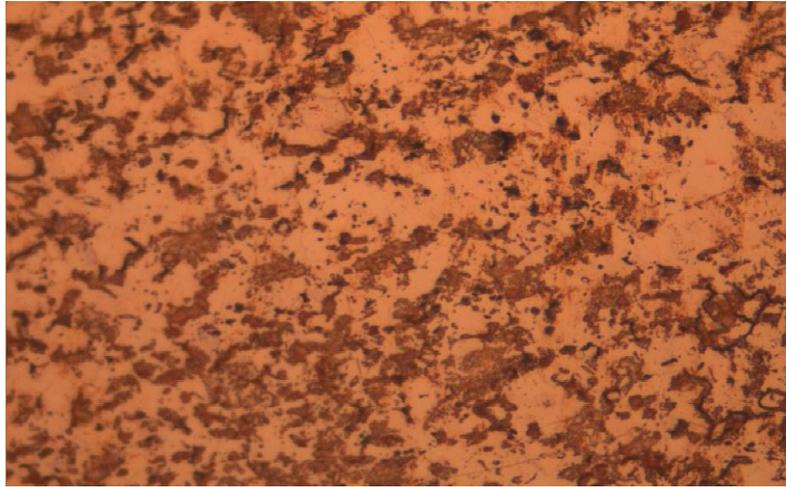
50 Magnifications



Pure Copper 2 Hour Sintering Time under 850⁰C and Argon atmosphere

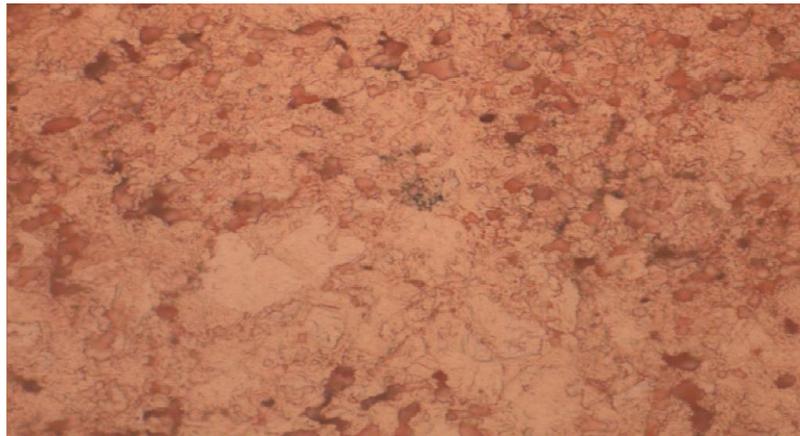


Cu-CNTs 1 Hour Sintering Time under 850⁰C and Argon atmosphere

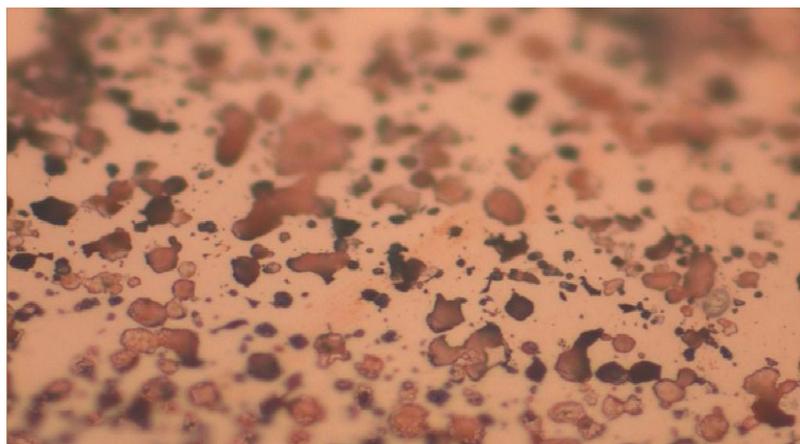


Cu-CNTs 2 Hour Sintering Time under 850⁰C and Argon atmosphere

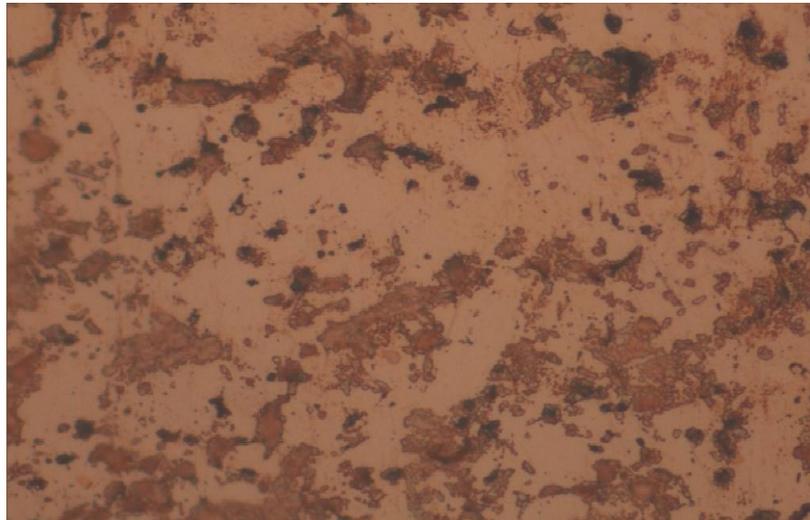
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Pure Copper 2 Hour Sintering Time under 850⁰C and Argon atmosphere



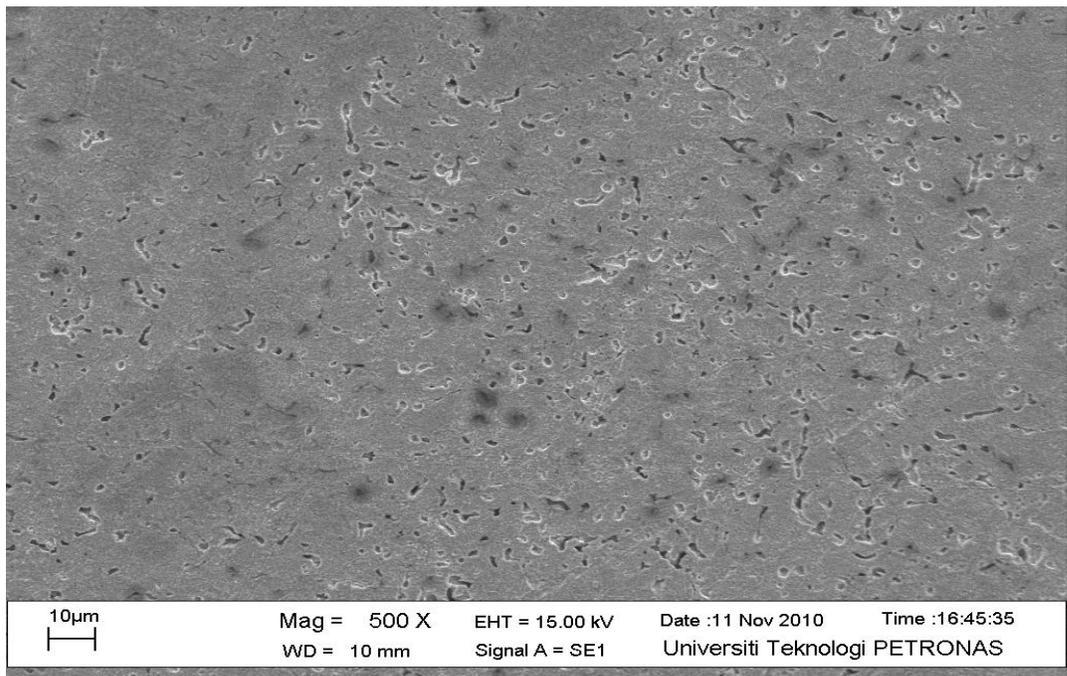
Cu-CNTs 1 Hour Sintering Time under 850⁰C and Argon atmosphere



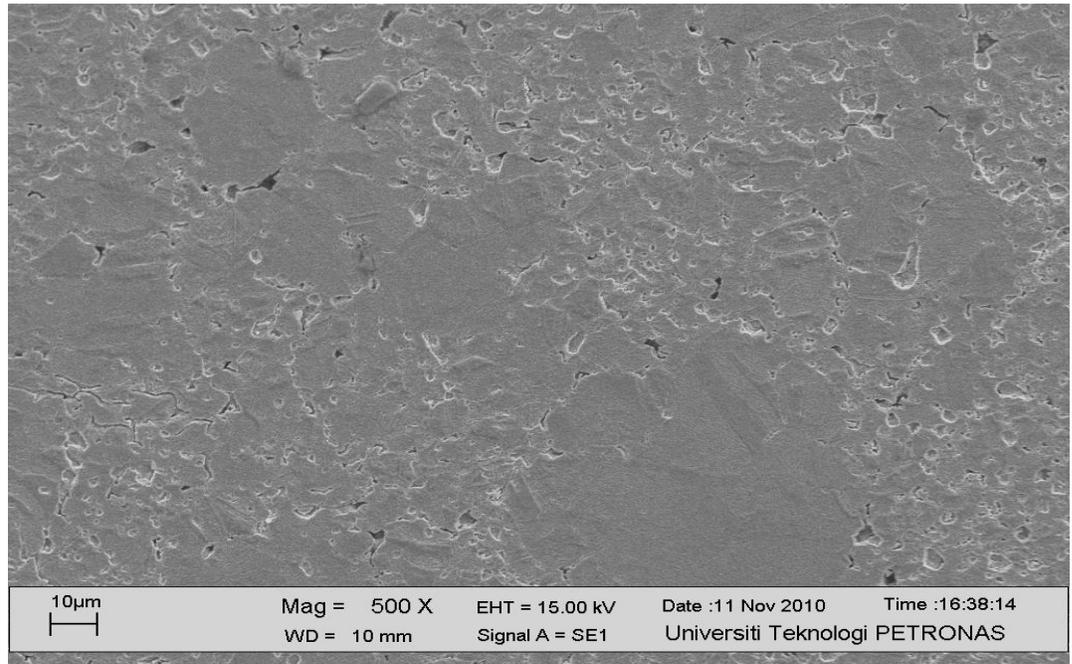
\Cu-CNTs 2 Hour Sintering Time under 850⁰C and Argon atmosphere
Scanning Electron Microscopy

At Surface of the sample

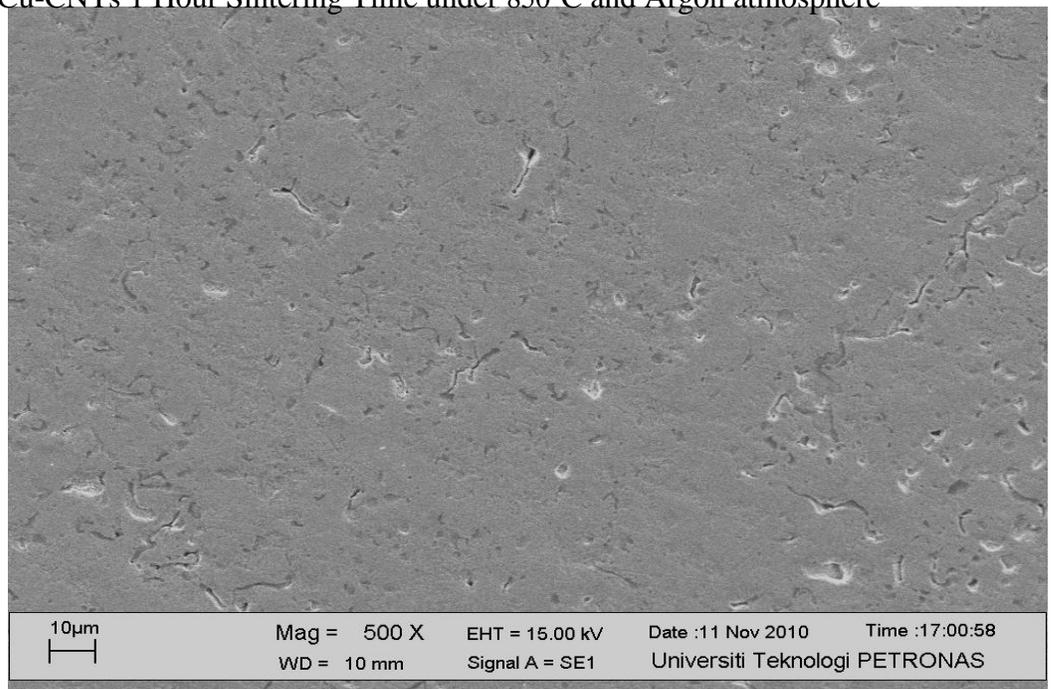
500 Magnifications



Pure Copper 2 Hour Sintering Time under 850⁰C and Argon atmosphere

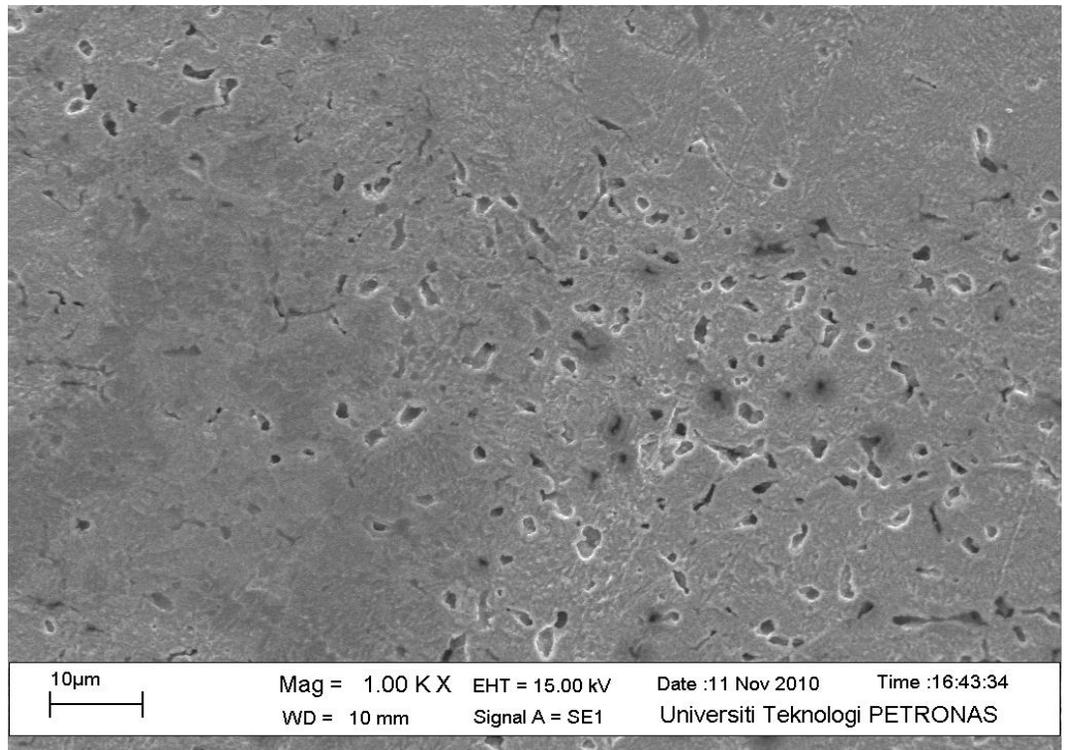


Cu-CNTs 1 Hour Sintering Time under 850⁰C and Argon atmosphere

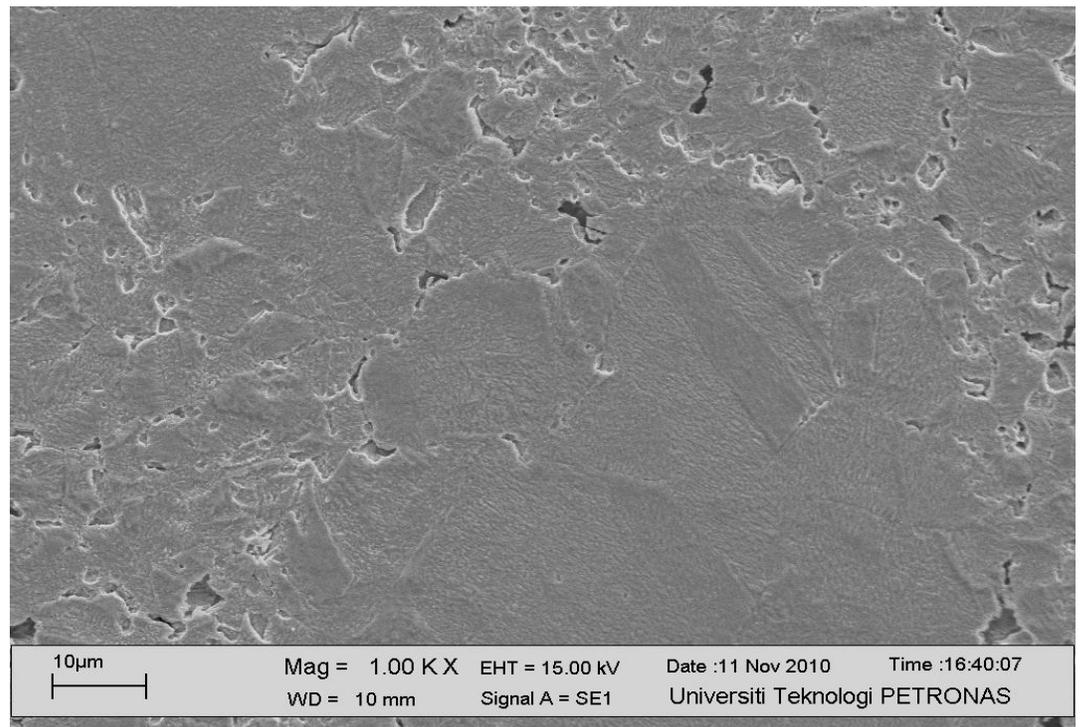


Cu-CNTs 2 Hour Sintering Time under 850⁰C and Argon atmosphere

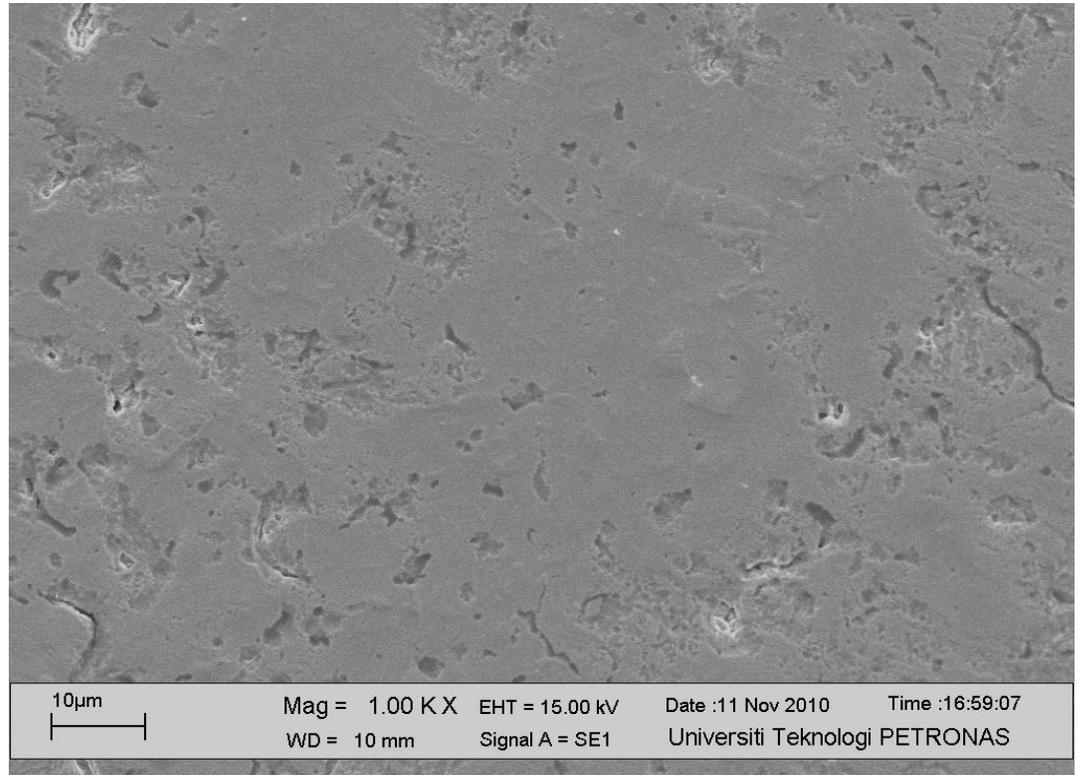
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Pure Copper Sintering Time under 850⁰C and Argon atmosphere



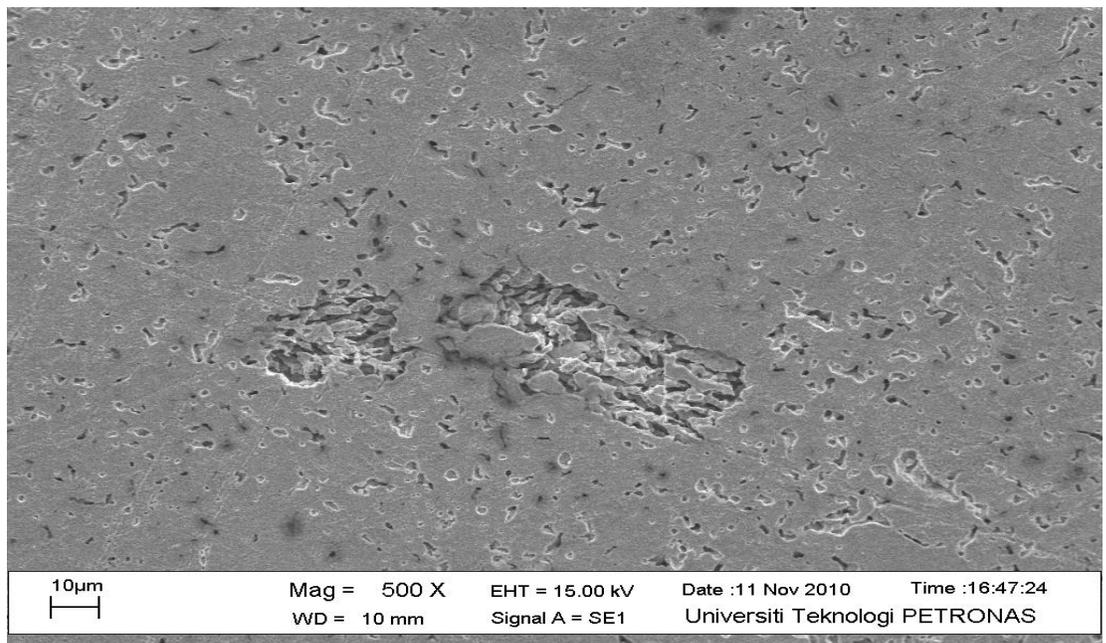
Cu-CNTs 1 Hour Sintering Time under 850⁰C and Argon atmosphere



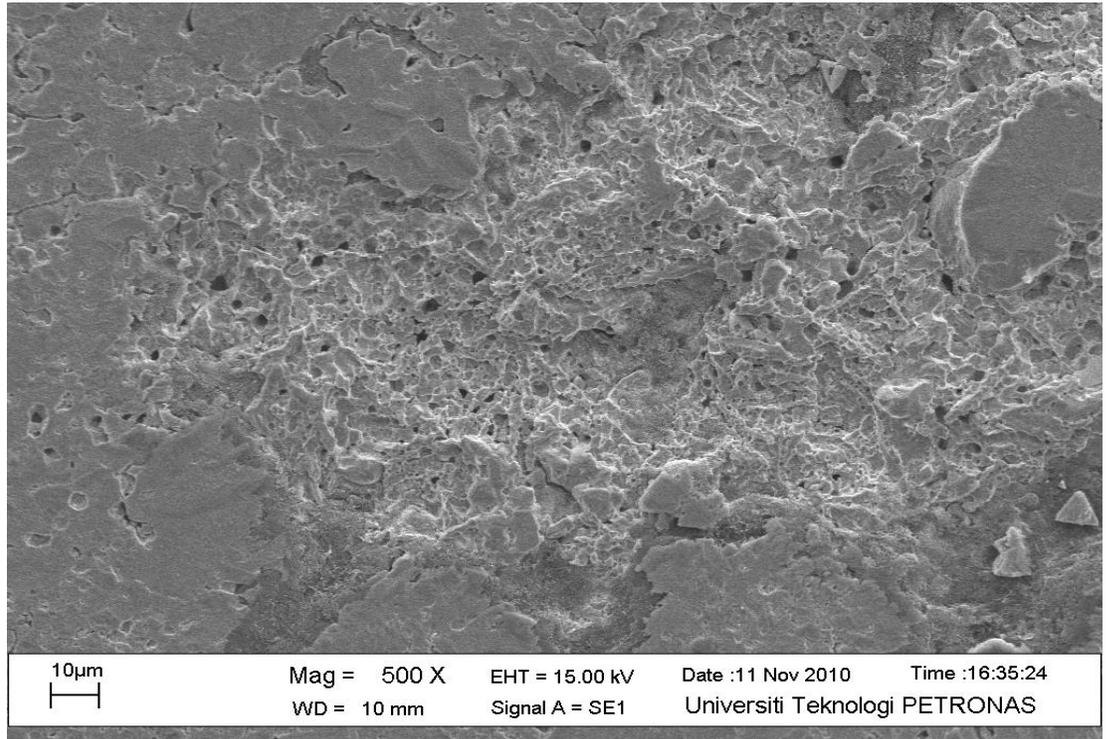
Cu-CNTs 2 Hour Sintering Time under 850⁰C and Argon atmosphere

Diffusion of Particles

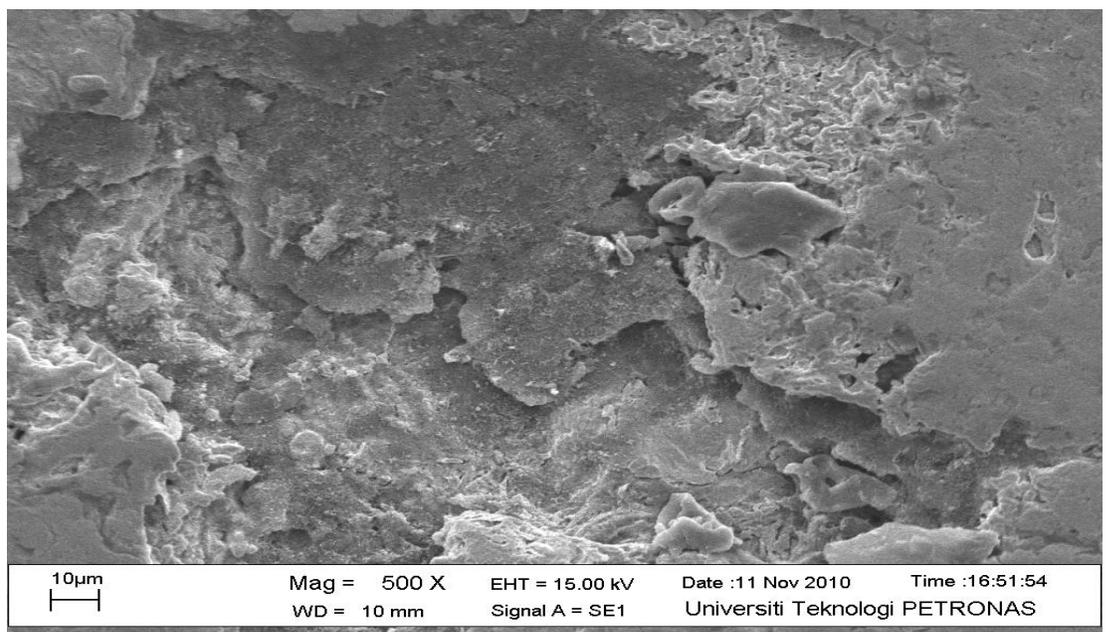
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Pure Copper 2 Hour Sintering Time under 850⁰C and Argon atmosphere

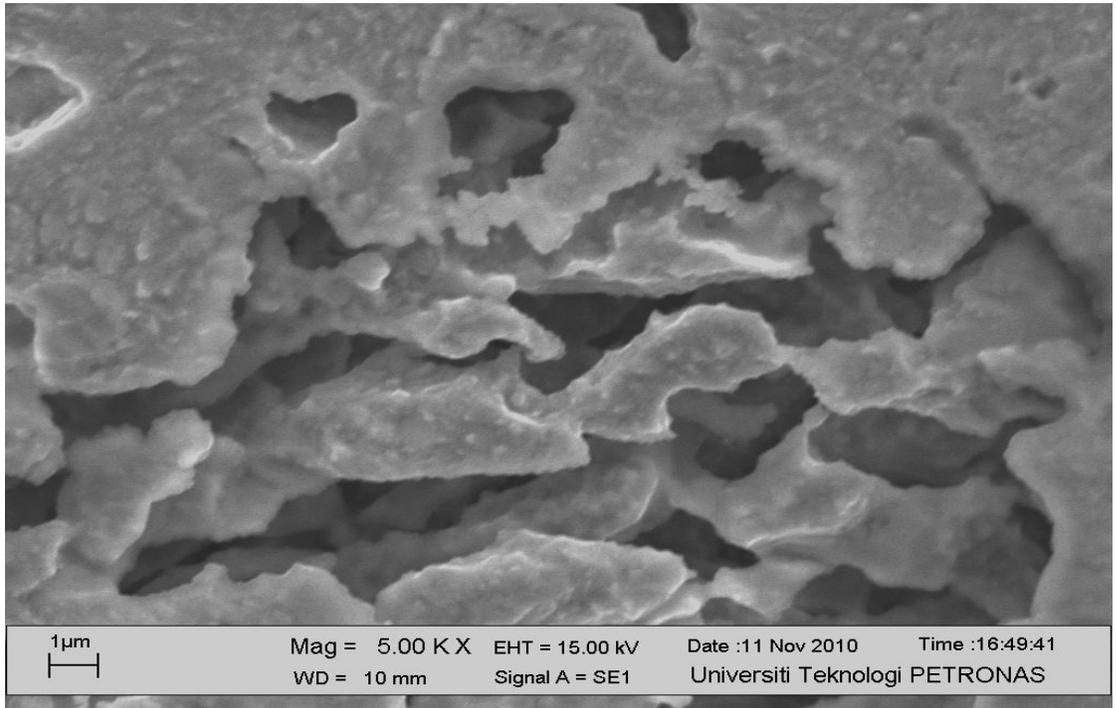


Cu-CNTs 1 Hour Sintering Time under 850⁰C and Argon atmosphere

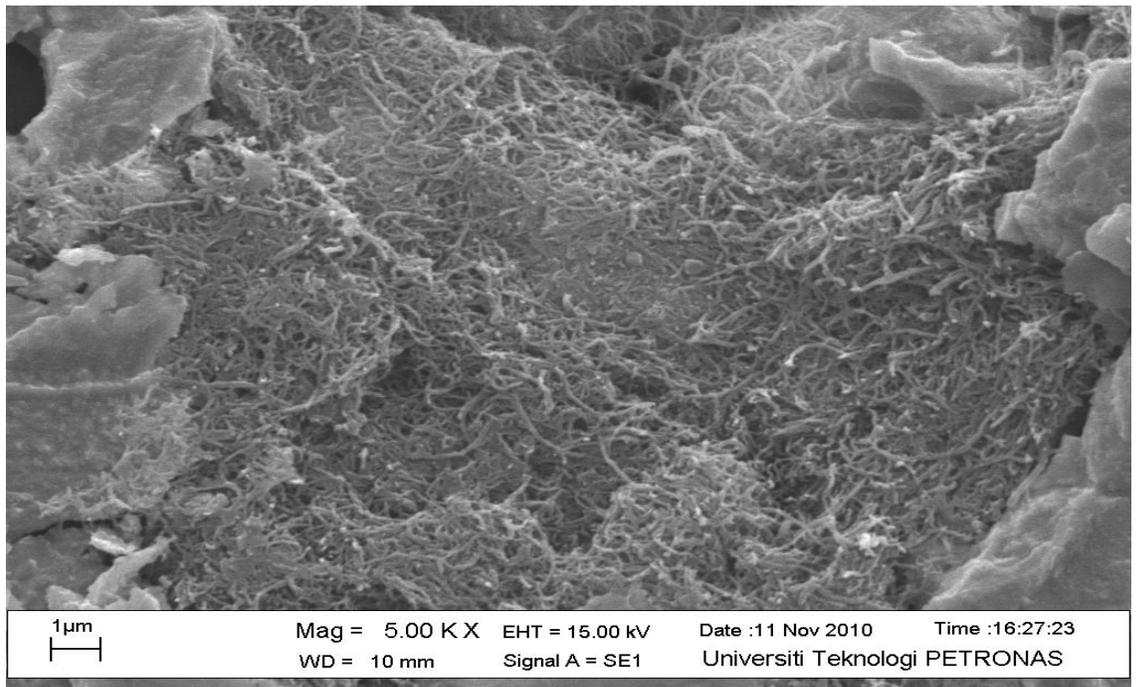


Cu-CNTs 2 Hour Sintering Time under 850⁰C and Argon atmosphere

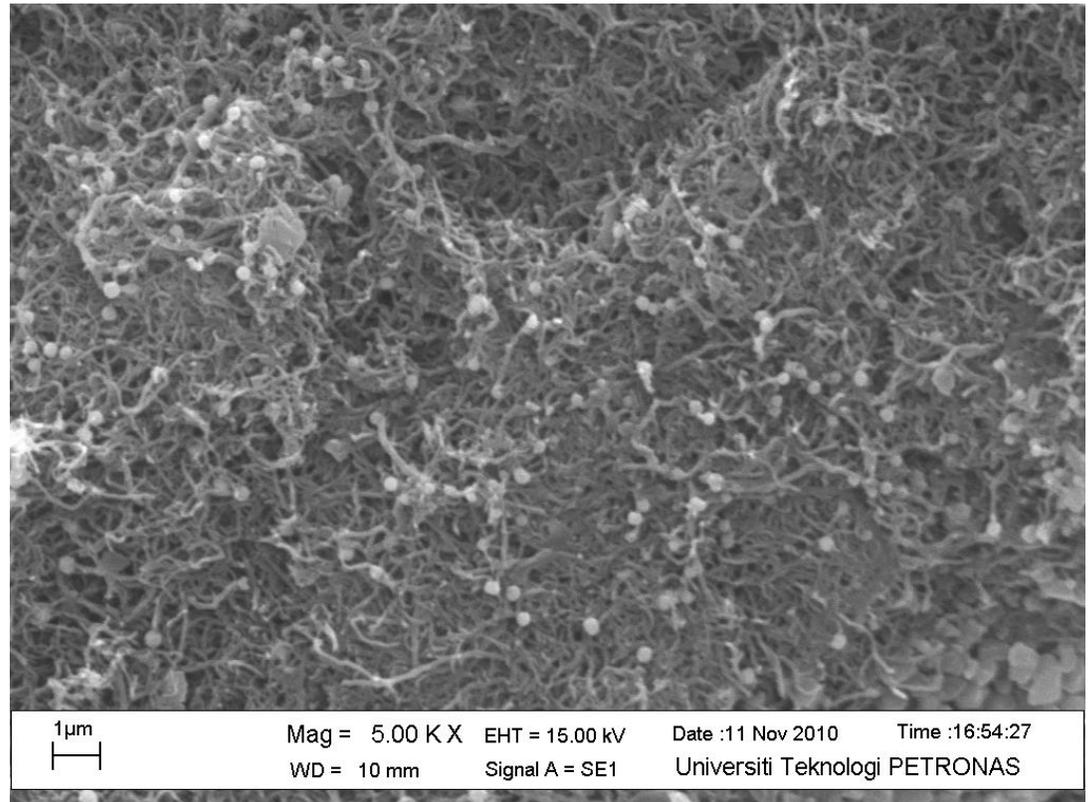
5000 Magnifications



Pure Copper 2 Hour Sintering Time under 850⁰C and Argon atmosphere



Cu-CNTs 1 Hour Sintering Time under 850⁰C and Argon atmosphere



Cu-CNTs 2 Hour Sintering Time under 850⁰C and Argon atmosphere