A Study on Wear Resistance of Copper – Silica Sand Nanoparticles Based Composites

by

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
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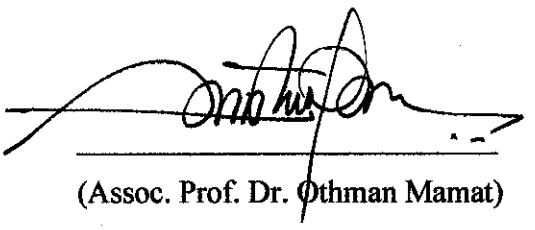
Approved by,

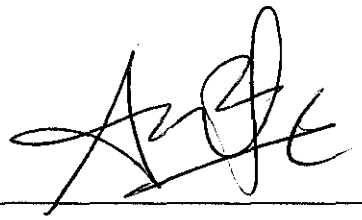


(Assoc. Prof. Dr. Othman Mamat)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
September 2011

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.



MOHAMAD ANUAR BIN MOS

ABSTRACT

Pure copper (Cu) and its composites reinforced with silica sand nanoparticles (SiO_2) were prepared by powder metallurgy process. These composites are expected to have high mechanical properties such as high melting point, hardness and good wear resistance. This study aims to find the compressibility curve for Cu and investigates the hardness and wear resistance of Cu-SiO_2 composites with different compositions of reinforcement of SiO_2. To create the compressibility curve for Cu, 6 different compression forces were applied (34MPa, 41MPa, 48MPa, 55MPa, 62MPa, and 69MPa) and the green densities values were measured and plotted. The following compositions were developed using the compression force that shows highest green density value: 5 samples of Cu, 5 samples of composites reinforced with 5wt%, 10wt%, 15wt% and 20wt% of SiO_2 nanoparticles. All samples were divided into 5 groups; each group consists of a sample of Cu, and composites reinforced with 5wt%, 10wt%, 15wt% and 20wt% of SiO_2 nanoparticles. Then the groups were sintered at 550^0C, 650^0C, 750^0C, 850^0C and 950^0C for 1 hour in argon atmosphere. The green and sintered densities were measured by following the Archimedes's method. The hardness values of the Cu-SiO_2 composites were measured using Vickers Microhardness Tester at constant load of 300gf and dwelling time of 15 seconds. The resistance test was conducted under 120N load using pin-on-disc apparatus and hardened cast iron was used as the counter face. It was observed that 41MPa compression force and 750^0C of sintering temperature are the suitable parameters which result in better green and sintered densities. The additions of SiO_2 nanoparticles up to 20wt% appeared to improve the hardness of the composites. The hardness increased from 38.2HV to 105.0HV. The sliding wear tests indicated that the composite with 20wt% SiO_2 nanoparticles exhibits a lower wear loss and wear rate compared to pure copper. The weight losses decreased from 0.18274g to 0.12824g and wear rate decreased from 5.153×10^{-2} mm^3/s to 2.474153×10^{-2} mm^3/s.

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1.0 INTRODUCTION

1.1 Project Background

1.1.1 Copper – Silica Sand Nanoparticles Based Composites

Copper is widely used as a material for electrical contacts and plumbing on account of its high electrical and thermal conductivities, low cost and ease of fabrication. However, some distinct shortcomings such as low hardness, low tensile strength, poor wear and arcing resistance limit its applications. "The mechanical strength of copper can be increased dramatically either by age hardening or by introducing dispersoid particles in its matrix [1]. "The wear resistance can be improved considerably by incorporating a ceramic reinforcing phase into copper, i.e. by developing copper based metal matrix composites (MMCs)" [4]. The copper alloys are highly potential alloys which exhibit good wear resistance, good machinability, cold workability, fatique resistance, corrosion resistance and can operate in very severe condition and up to temperature around 450°C [2]. The silica sand has high melting point (1725°C), excellent hardness and good wear resistance. The use of silica sand nanoparticles as a reinforcing phase for copper will be studied and analyzed by using some apparatus.

1.1.2 Powder Metallurgy

Powder metallurgy (PM) is the manufacture, processing and consolidation of fine metallic particles to produce the metal which often has superior properties resulting from a refined and uniform microstructure. The PM route consists of several processes like blending, compacting, sintering and secondary processing. Blending can be defined as the process where the metallic powders are mixed with the reinforced particles for example ceramic. Compacting is a mass-conserving shaping process. Compaction needs mold to shape the metal particles and the mold will be applied with high gas or fluid pressure. Sintering is a heating process to create new bonding between two or more materials. The powder will turn to solid composite after the new material is cooled. The secondary processing includes grinding and polishing the samples.

1.1.3 Wear Resistance

Wear is loss of material from a surface by means of some mechanical action [7]. In this experiment, the pin-on-disc is used to examine the loss of samples. After some scratches the weight will be measured by using Mettler Toledo AX 205 Density Measurement Instrument.

1.2 Problem Statement

The study of mechanical properties of copper-silica sand nanoparticles have not been established yet, and the same goes for the wear resistance. This study is to investigate on wear resistance of silica from the sand (95% purity) with the copper (99.7% purity). The powder methodology is used to produce the samples and the pin-on-disc to examine the wear. This study is important because it shows the improvement of mechanical properties of copper, which are hardness and wear resistance that can be applied in various sectors such as plumbing pipe. Copper plumbing is common and can be found in houses and building across United States. Wear problems can lead the pipe to corrode and causes leak. The improvement of hardness and wear resistance of copper after adding the silica sand nanoparticles is very beneficial and can be applied to this industry.

1.3 Objective and Scope of Study

The objective of this study is to analyze the wear resistance of silica sand nanoparticles reinforced copper composite. The study aims to find the compressibility curve for copper and to investigate the wear resistance of copper based composite with different compositions of reinforcement of silica sand nanoparticles. The composite will be interpreted by using Field Emission Scanning Electron Microscopic (FESEM). The works will be carried out in the period of 2 semesters in 2010/2011. All the tools and equipment are available in Level 17 laboratory, Universiti Teknologi PETRONAS, so this experiment is feasible to be conducted within these 2 semesters.

2.0 LITERATURE REVIEW

This study will be conducted based on the international journals as reference. 4 journals are selected as reference because of the same methodology and materials used. Below are the explanations about the journals:

The mechanical strength of copper can be increased dramatically either by age hardening or by introducing dispersoid particles in its matrix. In this experiment the pure copper and its composites reinforced with silica (SiC) particles were prepared by hot isostatic pressing (HIP). Methods for the production of dispersion-strengthened copper matrix composites involve ingot casting and powder metallurgy. Copper powder (99.9%) with size 50μm and SiC particles (99%) with size 75μm were used. The composites with the volume of SiC up to 20% were fabricated. The abrasive and dry sliding wear measurements were conducted using pin-on-disc tester. For the results and analysis, the pure copper exhibits an extremely high weight loss during abrasive sliding. The abrasive wear resistance of copper composites increases with the increasing SiC volume content. This is because SiC abrasives can penetrate easily to soft copper during sliding. The composites with higher SiC contents exhibit a better wear resistance due to SiC additions improve the hardness of specimens. Addition of 20wt% SiC particles to copper matrix increased the hardness of the composite and resulted in a reduction of the extent of plastic deformation of matrix [1]. This journal focuses on hardness and wear resistance of copper after adding some weight percentage of SiC (5wt%, 10wt%, 15wt%, 20%), so this journal is related to this study.

Copper-alumina metal matrix (Cu-Al_2O_3) were reinforced with alumina particles 5wt%, 10wt%, 15wt% of Al_2O_3 and prepared by using powder metallurgy method. Both copper and Al_2O_3 powders were mixed in a ball mill for 48 hours and were pressed into compacts at pressure ranging from 350MPa to 400MPa. Then the compacted samples were sintered at 850^0C for 1 hour. The densities were calculated using Archimedes's method, and the analysis is based on the XRD and microstructure. The sliding abrasive wear rates of the samples were conducted using pin-on-disc apparatus. The compressive

resistance increases with the increasing of Al_2O_3 weight fraction and therefore the porosity of 15wt% Al_2O_3 composites is the highest. The hardness values of the composite reinforced with Al_2O_3 particulate were increased. This can be attributed primarily to the presence of harder Al_2O_3 particulate in the copper matrix. The volume wear rate of the composite decreases with increasing Al_2O_3 volume fraction. The increasing hardness also increases the wear resistance of the composite and slows down in the wear rate [2]. This journal also focuses on wear resistance of copper composites, so it can be used as a reference for this study.

The copper-based composites can be obtained by powder metallurgy. Plenty of samples were made with different volume of silica sand nanoparticles (5wt%, 10wt%, 15wt%, 20wt%). The mixtures (copper and silica sand) with different volume of silica sand were ball milled for 1 hour. It is followed with compaction process using the hydraulic press of 6000Lb and a metallic mould of diameter of 13mm to make the pellets. Green densities of the samples are measured by following the Archimedes's method. The samples then will be sintered at 950^0C for 1 hour and the densities of the samples are measured again. The polish samples are analyzed by SEM and EDX analysis. Hardness and tensile strength are measured by using Rockwell Hardness Tester. For the results and analysis, it is clear that a decreasing trend in sintered densities, which show the reduction of porosity and full densification after sintering. These nanoparticles occupied the porous places and mixed with copper to fill these porosities to reduce the densities and enhance the mechanical properties. The increasing trend of silica sand also increases the presence of silica. XRD shows the silica sand nanoparticles still have the same crystalline structure during fabrication process and are homogeneously distributed in copper matrix which increases the mechanical properties of the composites. The addition of silica sand nanoparticles enhanced the hardness from 119HV to 555.4HV and tensile strength from 375.54MPa to 770.57MPa [3]. This journal is used as the guidelines for powder metallurgy method for this study.

The wear behavior of tungsten carbide (WC) particle reinforced copper matrix has been determined with pin-on-disc technique against a sintered SiC abrasive disc. The cold compression was used to get the sample. Then the sample will be heated using infrared heating. The sample was heated at 1083^0C in 2-8 minutes followed by rapid cooling down to room temperature. The densities of the sintered samples were measured by Archimedes's method. The microhardness was measured using a Vicker hardness tester at the load of 100gm. The wear test was carried out with a pin-on-disc method with a composite pin against a sintered SiC disc. The wear rate of copper increased with the increase in the applied load and the wear rate of copper is higher than Cu/WC composites at all load conditions. The increase in wear rate of Cu/WC composite with the increase in applied load is due to the increased wear of the WC particles in contact with the counter face followed by the re-appearance of the new soft copper matrix phase to the abrasive counter face. The better wear resistance of Cu/WC composite as compared to that of pure copper is due to better overall hardness of the Cu/WC composite as compared to the pure copper [4]. This journal used Vickers Microharness Testes to get the hardness values. So this journal is used as the guideline to evaluate the hardness of this study.

Table 1 shows the summary of the journals:

Table 1 : Summary of the journals

Title + Author(s)	Findings	Remarks
Tribologocal Behaviour of SiC Particle – Reinforced Copper Matrix Composites S.C Tjong, K.C Lau, "Tribologocal Behaviour of SiC Particle – Reinforced Copper Matrix Composites", Materials Letters, Volume 43, Issues 5-6, May 2000, Pages 274-280.	**Materials** - Copper powder, Cu (99.9% purity, 50μm) - Silica, SiC particles, SiC (99% purity, 75μm) - 5wt%, 10wt%, 15wt%, 20wt% of SiC **Methods** - HIP method - Mixing/blending - Archimedes's method - Tensile testing (Instron tensile tester) - Microhardness test (Vickers tester) - Pin on disc testing - Scanning Electron Microscopy(SEM) for observation **Results** - Wear volume increases as sliding distance increases. - The composites with higher SiC contents exhibit a better wear resistance. - Increasing sliding velocity leads to further reduction in volume wear. - The hardness of composite increases as SiC contents increase.	- This journal states the wear test and the relationship between hardness and wear resistance clearly - This journal only focuses about wear, not other mechanical properties except hardness, densities and tensile strength. - Relates to this study.
Development of Copper:Alumina Metal Matrix Composite by Powder Metallurgy Method Thiraviam, R., Somakumar, T. And Senthil Kumar, A., "Development of Copper:Alumina Metal Matrix Composite by Powder Metallurgy Method", Int. J. Material and Product Technology, Vol. 31, Nos. 2/3/4 (2008) pp.305-313.	**Materials** - Copper powder, Cu (45μm, density of 8.920g/cm^3) - Alumina powder, Al_2O_3 (1.2μm, density of 3.97/cm^3) - 5wt%, 10wt%, 15wt% of Al_2O_3 **Methods** - Mixing and Blending - Consolidation - Sintering - Secondary processing - Archimedes' principle - Philips X-ray Diffractometer (XRD) and optical microscope (observation)	- This journal states both mechanical properties and wear resistance results. - All methodology applied were written clearly. - As a reference for this study.

	- Brinell hardness testing - Pin-on disc testing - Eddy current method - Electrochemical technique **Results** - The porosity of pure copper is lower than copper-Al_2O_3. - The hardness value of the composite reinforced with Al_2O_3 particulate were increased as compared with pure copper. - The volume wear rate of the composite decreases with increasing Al_2O_3 volume fractions. - The increased wear resistance is attributed to the increase of the hardness of the composite and slow down in the wear rate of the composite. - The electrical conductivity decreased with increasing alumina because the weight fraction of non-conducting alumina increased.	
Characterization and Properties of Copper Silica Sand Nanoparticles Composites Tahir Ahmed, Othman Mamat, "Characterization and Properties of Copper Silica Sand Nanoparticles Composites", International Conferences on Plant Equipment and Reliability (ICPER) 15 June 2010.	**Materials** - Copper powder, Cu (99.7% purity, 63μm) - Silica sand nanoparticles, SiO_2 (95% purity, 100nm) - 5wt%, 10wt%, 15wt%, 20wt% of SiO_2 **Methods** - Ball milling - Green compaction - Archimedes's method - Sintering - Scanning Electron Microscopy(SEM) **Results** - Green and sintered density decrease as volume of silica decreases. - Hardness and tensile strength increase as volume of silica increases. - Conductivity of composite increases as volume of silica increases.	- Relates to this study. - This ICPER 2010 states the methodology to get the mechanical properties data of the composite clearly, but not included wear test.

Wear resistance of WC Particle Reinforced Copper Matrix Composites and the Effect of Porosity P.K Deshpande, R.Y Lin, "Wear Resistance of WC Particle Reinforced Copper Matrix Composites and the Effect of Porosity", Materials Science and Engineering, Volume 418, Issues 1-2, 25 February 2006, Pages 137-145.	**Materials** - Tungsten carbide (WC) - Copper **Methods** - Cold pressing technique - Sintering - Rapid cooling - Vickers hardness testing - Pin-on-disc testing **Results** - The applied wear stress increased , the composite was worn faster. - The temperature of the composites wear surface increased with the increase in applied load. - Wear rate of copper increased with the increase in the applied load. - The increase in wear rate of Cu/WC composite with the increase in applied load is due to the increased wear of the WC particles in contact with the counter face.	- Not state the contents/ratio/ volume of WC or copper in composite preparation part. - State the best volume of WC to make the Cu/WC composites in fully dense of wear which is 53 vol.%. - This journal can be used as guideline to evaluate the hardness. - Related as a reference.

3.0 METHODOLOGY

3.1 Materials

The test materials studies in this work are copper powder (99.7%) with size 63μm as a metallic matrix and silica sand nanoparticles (with average size less than 100nm). The silica sand was taken from outside of the main gate of Universiti Teknologi PETRONAS, Bandar Seri Iskandar, 31750, Tronoh, Perak, Malaysia and grounded to nanoparticles by using low ball mill with zirconium ball as grinding media and the production of silica sand nanoparticles. This silica sand nanoparticles had been verified by ZetaSizer, Nano ZS (ZEN 3600) (Malvern) [3].

3.2 Tools and Equipment

The tools and equipment required for this study are:

i. Ball mill
ii. Autopallet press machine
iii. Mettler Toledo AX 205 Density Measurement Instrument
iv. Sintering furnace
v. Grinder and polisher
vi. Field Emission Scanning Electron Microscope (FESEM)
vii. Vickers Microhardness Tester
viii. Pin-on-disc machine

3.3 Sample Preparation and Experiment

For sampling composites, firstly the silica is grounded for 6 hours using ball mill machine. The copper powder and silica sand nanoparticles were ball milled for 1hour, and followed by green compaction. The hydraulic press (made by USA, capacity of 80KN) at various forces (34MPa, 41MPa, 48MPa, 55MPa, 62MPa and 69MPa) by using a metallic mould of diameter of 13mm is used to make the pellets. The purpose is to create the compressibility curve for fine particles of copper. The following compositions were developed using the compression force that shows highest green density value: 5 samples of pure copper, 5 samples of 5wt% silica sand nanoparticles, 5 samples of 10wt% silica sand nanoparticles, 5 samples of 15wt% silica sand nanoparticles and 5 samples of 20wt% silica sand nanoparticles. The green densities of the samples were measured by following the Archimedes's method (Mettler Toledo AX 205 Density Measurement Instrument). All samples were divided into 5 groups; each group consists of of pure copper, 5wt% silica sand nanoparticles, 10wt% silica sand nanoparticles, 15wt% silica sand nanoparticles and 20wt% silica sand nanoparticles. Then the groups were sintered at 550^0C, 650^0C, 750^0C, 850^0C and 950^0C for 1 hour in argon atmosphere. The heating and cooling rates of sintering process are 5^0C/min and 10^0C/min respectively. After sintering, the densities of the samples were measured again. The selected samples were mounted for grinding and polishing process. The polish samples were analyzed by FESEM and EDX analysis. Hardness values were measured by using Vickers Microhardness Tester [3]. To analyze the wear resistance, the pin-on-disc is used.

3.4 Project flow chart

Figure 1 shows the flow chart for this project :

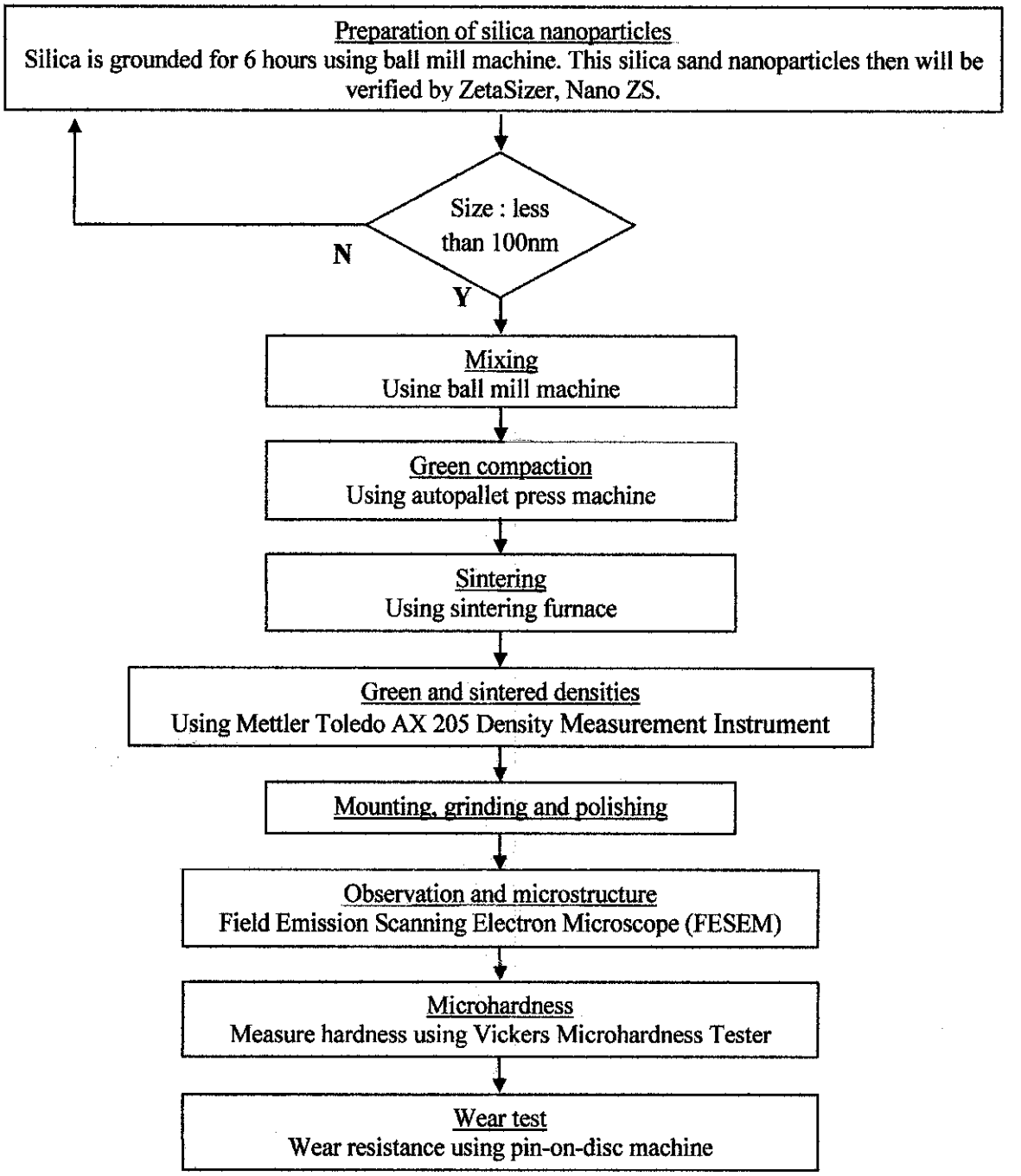


Figure 1 : Project flow chart

3.5 Project Planning for FYP 1

Table 2 shows the planning for Final Year Project 1 :

Table 2 : Final Year Project 1 planning

No	Activities	Week													
		1	2	3	4	5	6		7	8	9	10	11	12	13	14
1	Selection of Project Title															
2	Preliminary Research Work															
	2.1 Literature review															
	2.2 Preliminary report preparation															
	2.3 Submission of preliminary report					▲										
3	Methodology															
	3.1 Get familiar with equipment															
	3.2 Mixing the composite using ball milling															
	3.3 Compacting using hydraulic pressing to get the pellet															
	3.4 Sintering using sintering furnace at 750°C, 850 °C and 950°C															
4	Progress report and seminar															
	4.1 Data gathering and analysis for progress report															
	4.2 Submission of progress report									▲						
	4.3 Seminar									▲						
5	Methodology															
	5.1 Measuring the green density															
	5.2 Measuring the sintered density															
	5.3 Grinding and polishing the pallets															
6	Interim report															
	6.1 Collecting data and discussion															
	6.2 Submission of interim report														▲	
7	Oral presentation															▲

Mid-Semester Break

Process ▲ Key milestone

3.6 Project Planning for FYP 2

Table 3 shows the planning for Final Year Project 2 :

Table 3 : Final Year Project 2 planning

No	Activities	Week													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Methodology continues														
	1.1 Mixing the composites	■	■												
	1.2 Compacting the composites at 34MPa, 41MPa, 48MPa, 55MPa and 62MPa		■	■											
	1.3 Measuring the green density of the pallets (to choose the best green density)			■											
	1.4 Sintering at 550 °C, 650 °C, 750 °C , 850°C and 950 °C for 1 hour			■	■	■									
	1.5 Measure the sintered density for 550 °C, 650 °C, 750 °C , 850°C and 950 °C			■	■	■	■								
	1.6 Microstructures analysis using FESEM and EDX							■							
2	Progress report submission														
	2.1 Submission of progress report								▲						
3	Methodology continues														
	3.1 Grinding, polishing and mounting the pallets for testing purposes									■					
4	Tests														
	4.1 Hardness testing using Vickers Microhardness Tester										■	■			
	4.2 Wear resistance using Pin-on-Disc											■	■		
5	Pre-EDX														
	5.1 Pre-EDX											▲			
6	Poster, dissertation report, oral presentation and hard bound DR submission														
	6.1 Submission of draft report												▲		
	6.2 Submission of dissertation report (soft bound)													▲	
	6.3 Submission of technical report													▲	
	6.3 Oral presentation														▲
	6.4 Hard bound dissertation report submission														▲

■ Process ▲ Key milestone

4.0WORK DONE

4.1 Preparation of The Samples

The copper powder had been collected from the laboratory at Block 17, University Teknologi PETRONAS. The silica sand nanoparticles were produced after 6 hours of dry milling process. The production of the silica had been done with the help from a postgraduate student. Figure 2 and Figure 3 show the raw materials that had been used for this stdy :

Figure 2 : Copper powder

Figure 3 : Silica sand nanoparticles

4.2 Mixing

The composition of copper powder and silica sand nanoparticles were calculated to get 5wt% silica sand nanoparticles, 10wt% silica sand nanoparticles, 15wt% silica sand nanoparticles and 20wt% silica sand nanoparticles. Five 2 grams of mixtures of copper and silica sand nanoparticles had been made according to its ratio. For example, copper+5wt% silica sand nanoparticles was made from 1.9000g of copper and 0.1000g of silica sand nanoparticles. Table 4 shows the composition weights of copper and silica sand nanoparticles :

Table 4 : Composition weight of copper and silica sand nanoparticles

Composition (total = 2grams)	Copper powder (g)	Silica sand nanoparticles (g)
Pure copper	2.0000g	0.0000g
Copper + 5wt silica sand nanoparticles	1.9000g	0.1000g
Copper + 10wt% silica sand nanoparticles	1.8000g	0.2000g
Copper + 15wt% silica sand nanoparticles	1.7000g	0.3000g
Copper + 20wt% silica sand nanoparticles	1.6000g	0.4000g

The weights of the powder were measured using the Mettler Toledo AX 205 Density Measurement Instrument. Density can be also measured using this equipment. Figure 4 shows the Mettler Toledo AX 205 Density Measurement Instrument.

Figure 4 : Mettler Toledo AX 205 Density Measurement Instrument

After weighing the copper powder and silica sand nanoparticles, the mixing process between copper powder and silica sand nanoparticles was conducted by using ball mill machine. The mixing process was done for an hour to ensure that the mixing elements were perfectly homogeneous and uniform distribution of the mixture is got. The illustration of the ball mill machine is as in Figure 5 :

Figure 5 : Ball mill machine [9]

The mixtures of the copper powder and silica sand nanoparticles are showed in Figure 6 :

(a)

(b)

(c)

(d)

(e)

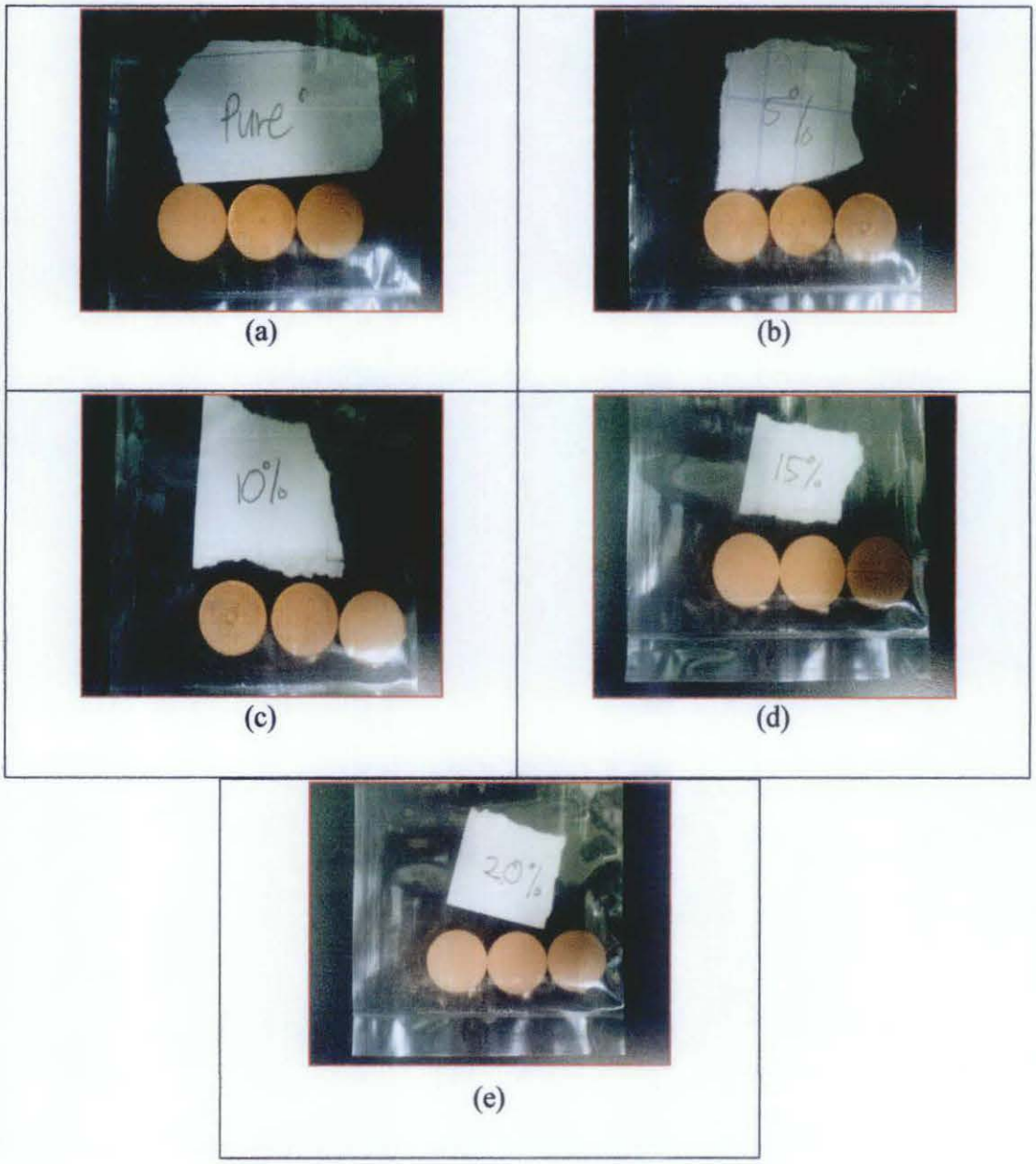
Figure 6 : Mixture of (a) copper powder with (b) 5wt% silica sand nanoparticles, (c) 10wt% silica sand nanoparticles, (d) 15wt% silica sand nanoparticles and (e) 20wt% silica sand nanoparticles

4.3 Compacting

After the mixture mixed using the ball mill machine, the mixture would be weighed (2grams) before compacting. The reason is to equalize the weight for all samples. The compaction is done using the Autopallet Press Machine. The green compaction is needed to get the required shape and to build the compressibility curve for the copper. The 34MPa, 41MPa, 48MPa, 55MPa, 62MPa forces applied and the compression force that results the highest green density will be chosen as the parameter for this study. Figure 7 shows the Autopallet Press Machine:

Figure 7 : Autopallet press machine

5 samples of pure copper, 5wt% silica sand nanoparticles, 10wt% silica sand nanoparticles, 15wt% silica sand nanoparticles and 20wt% silica sand nanoparticles were produced during the green compaction. The green parts are usually very fragile and can damage very easily. After the compaction, the samples would be sintered at three different temperature based on Binary Phase Diagram of copper and zinc. Figure 8 shows the pallets:



(a) (b) (c) (d) (e)

Figure 8: The pallet from (a) copper powder added with b) 5wt% silica sand nanoparticles, (c) 10wt% silica sand nanoparticles, (d) 15wt% silica sand nanoparticles and (e) 20wt% silica sand nanoparticles

The operation of the machine follows the standard of ASTM B331-95 for Standard Test Method for Compressibility of Metal Powders in Uniaxial Compaction.

4.4 Sintering

5 batches of samples were created, and each batch contains the pure copper, 5wt% silica sand nanoparticles, 10wt% silica sand nanoparticles, 15wt% silica sand nanoparticles and 20wt% silica sand nanoparticles each. Then, the batches were sintered in 5 different temperatures, which are 550°C, 650°C, 750°C, 850°C and 950°C for one hour under argon atmosphere by using the sintering furnace [3]. These temperatures were decided to be as the parameter based on the Binary Phase diagram of copper and zinc. The phase diagram for copper and silica sand is not published yet, so for the comparison, the copper-zinc phase diagram is used. Figure 9 shows the phase diagram of copper and zinc:

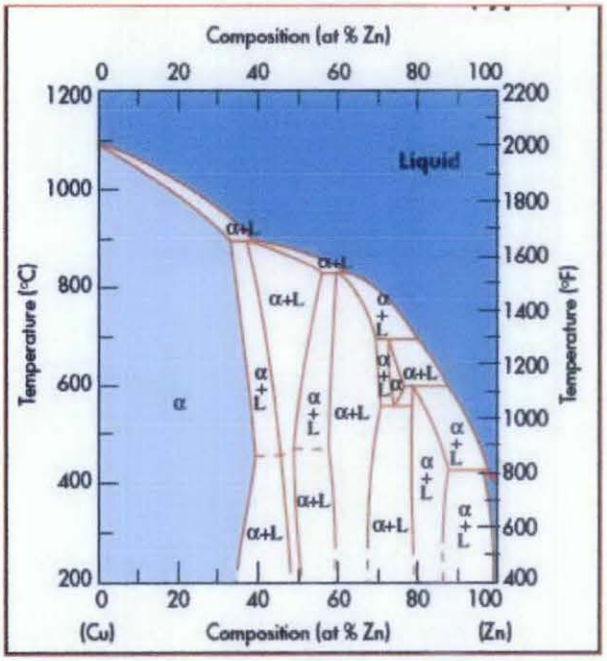


Figure 9 : The copper-zinc phase diagram [8]

The reason why this phase diagram was chosen as the reference is that the melting point of the zinc is below the silica sand. All the samples must have the same state, for example State a (Figure 9) during the cooling process. This is because the microstructure and properties of an alloy depends on such variables as the alloying elements present, their concentrations and the heat treatment of the alloy (i.e., the temperature, the heating time at temperature and the rate of cooling to room temperature). Based on the diagram, at 20wt% of zinc, the maximum temperature that can be applied for sintering is 1000°C and at that point, the microstructures of the samples will be the same. This means at 550°C, 650°C, 750°C, 850°C and 950°C, the samples would have the same microstructures and thus the samples could be compared to each other. Figure 10 and Figure 11 show the sintering furnace and the pallet after sintered :

Figure 10 : Sintering furnace

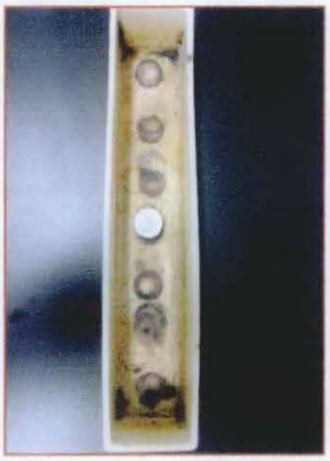


Figure 11 : Sintered pallet

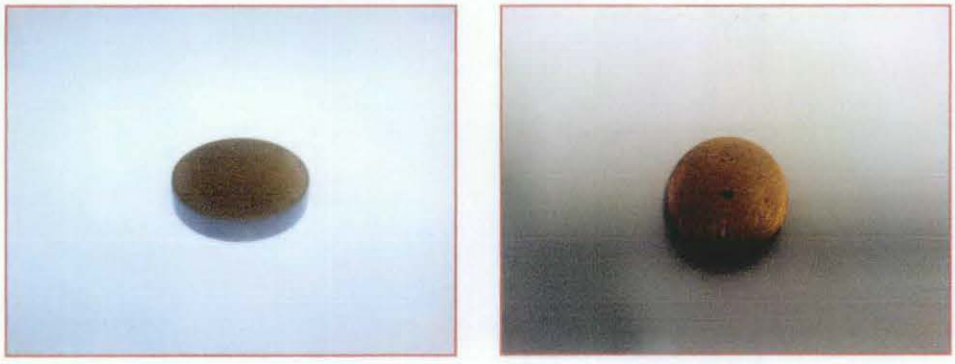
The sintering process for copper only needs 12 to 45 minutes to make the microstructures changed. Table 5 shows typical sintering temperature and time of copper alloys and steels :

Table 5 : Typical sintering temperature and time of copper alloys and steels [10]

Material	Temperature		Time, min
	°C	°F	
Bronze	760 – 780	1400 - 1600	10 – 20
Copper	840 – 900	1550 – 1650	12 – 45
Brass	840 – 900	1550 – 1650	10 – 45
Iron, iron graphite, etc	1010 – 1150	1850 2100	30 – 45
Nickel	1010 – 1150	1850 2100	30 – 45
Stainless steel	1095 - 1285	2000 – 2350	30 - 60

4.5 Grinding, polishing and mounting

The purpose is to get the smooth surface for testing purposes. The machines used were grinder and polisher. After grinding and polishing, the samples would be mounted. Figure 12 shows the surface of sample before and after grinding and polishing and Figure 13 shows the mounted samples :



(a) (b)

Figure 12 : The sample (a) before and (b) after grinding and polishing

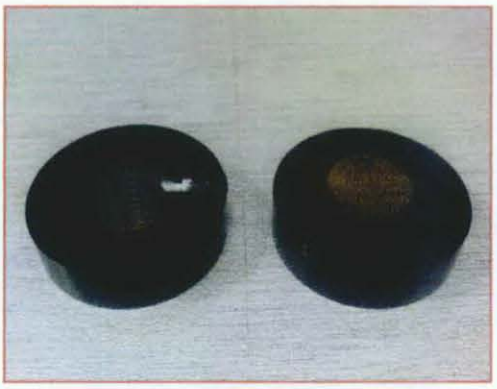


Figure 13 : Mounted samples

4.6 Field Emission Scanning Electron Microscopic (FESEM) and Energy Dispersive X-ray Spectroscopy (EDX)

A part of samples were cold mounted for the metallographic examination. The samples were polished and the images were taken using FESEM (Figure 14). FESEM is used to observe the microstructure of the sample. During the sintering period, the pores in the samples will be filled by silica sand nanoparticles. FESEM and EDX are used to observe the distribution of silica sand in copper and how silica sand nanoparticles are occupied the porosity places after sintering. There are 5 samples to be observed where each one of them has different silica sand nanoparticles contents (pure copper, 5wt% silica sand nanoparticles, 10wt% silica sand nanoparticles, 15wt% silica sand nanoparticles and 20wt% silica sand nanoparticles). The samples are observed at magnifications of 1000x with the resolution of 1nm.

Figure 14 : Field Emission Scanning Electron Microscopic (FESEM)

4.7 Microhardness

Microhardness was measured using Vickers Microhardness Tester at constant load of 300gf and dwelling time of 15 seconds. The values measured were taken up to 5 times. Figure 15 shows the Vickers Microharness Tester :

Figure 15 : Vickers Microhardness Tester

4.8 Wear test

The sliding abrasive wear test of pure copper and silica sand nanoparticles-copper composites were conducted under 120N load using pin-on-disc apparatus (as showed in Figure 16). The pure copper and composites were used as pin materials with 13mm diameter, and hardened cast iron was used as the counter face. After each test, the pin were carefully cleaned and weighted using Mettler Toledo AX 205 Density Measurement Instrument (also called electronic balance) with an accuracy of 0.01mg to determine the weight loss.

Figure 16 : DUCOM Multi Specimen Tester (Pin-on-Disc)

5.0 RESULTS AND DISCUSSION

5.1 Compressibility curve

Six (6) different pressures have been applied to produce the compressibility curve for copper. The green densities of each sample will be measured and the force with highest value of green density will be taken as according to developed compressibility curve for this experiment. Table 6 shows the green density values for 6 different compression forces. Better green density was obtained at compressibility forces 69MPa. Therefore, it was decided to use 69MPa compressions for developing these copper based silica sand nanoparticles composites.

Table 6 : Green density values for 6 different compression forces

Pressure (MPa)	Sample 1 (g/cm^3)	Sample 2 (g/cm^3)	Average (g/cm^3)
34	6.968	6.646	6.807
41	7.201	7.126	7.164
48	7.105	7.014	7.060
55	6.652	6.756	6.704
62	6.952	7.033	6.993
69	7.472	7.472	7.472

The green density of each sample was measured and the values were taken to produce the compressibility curve. It was observed that 69MPa is suitable pressure which results in better green density. Therefore, it was decided to use 69MPa compression force to develop copper based silica sand nanoparticles composite for further studies.

Figure 17 shows the compressibility curve for copper :

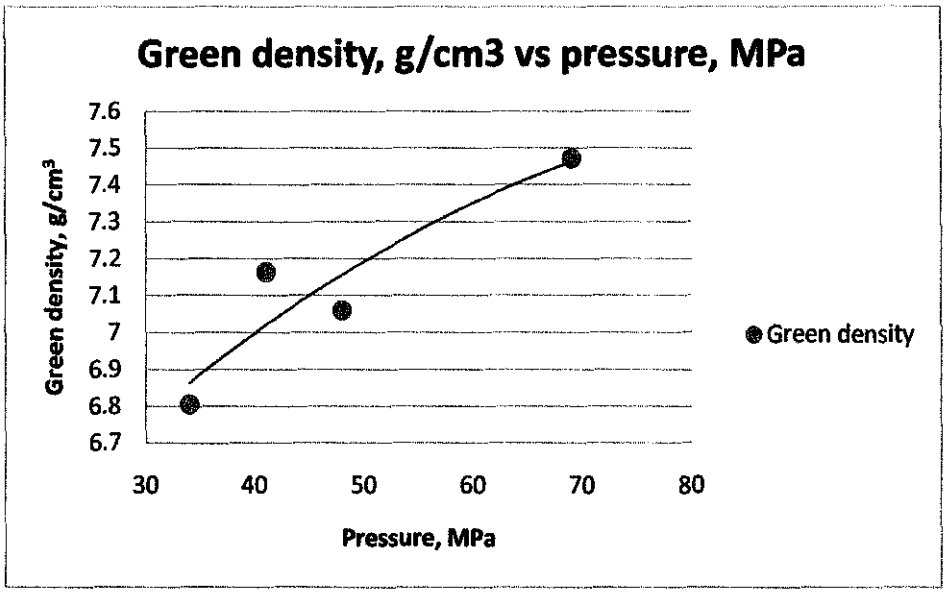


Figure 17 : Compressibility curve for copper

5.2 Green and sintered density at 69MPa

The green compacts compacted at 69MPa were sintered at different temperatures. Sintered densities of these copper based silica sand nanoparticles composites were calculated using the Mettler Toledo AX 205 Density Measurement Instrument, followed by sintering at 750^{0}C, 850^{0}C and 950°C. Table 7 shows the green and sintered densities for copper and silica sand nanoparticles :

Table 7 : Green and sintered density (g/cm^3) at 69MPa

Composition of SiO_2	Green density (g/cm^3)	Sintered density (g/cm^3)		
		750 °C	850 °C	950 °C
Pure Cu	7.472	7.198	7.318	6.762
5wt%	6.455	6.234	6.477	6.177
10wt%	5.557	4.846	5.357	5.595
15wt%	4.944	4.448	4.893	4.938
20wt%	4.551	4.222	4.610	4.658

Theoretically, the sintered density for the composites must be higher than green density. There are a lot of pores in powder body before compaction, so finer particles are easily filled into pores among coarser particles under impact force. The green density is improved mainly in the form of sliding, filling pores and rearranging of powder particles. As the force increases, the capacity of powder particles to deform and fill pores is increased achieving higher green density. The rule of sintered density of part is similar to the green density. As the force increased, the green density will increase gradually, and so does the sintered density.

It is observed that our results are not matching with other researchers [1,3]. This is the new area of research because still no researchers had studied the behavior of the copper based silica sand nanoparticles composites. This study also leads foundation for copper based silica sand nanoparticles composites. However, the following parameters should be counted for this research:

1. Applying high force causes destruction of the particles.

   A higher pressure is generally desirable when compacting a powder sample in the P/M practice, regardless of the sintering technologies employed. The higher the compaction pressure, the higher the sintering rate and the final density, because of reduced green porosity and increased dislocation population. However, excessively high pressure should be avoided because it is neither practical for intended applications nor beneficial to the final microstructures in most materials. Using higher compaction pressure will destroy the particles especially when dealing with nanoparticles. The particles are liable to be crushed if too high pressure is applied. There likely exists an optimum range of compacting pressure. Compaction pressure does have a determinative influence on the bonding between the particles formed in the subsequent sintering stage.

   Excessively high compaction pressure is not only difficult to achieve in practice but also likely to bring about undesirable defects in the final form. In addition to the aforementioned cracking and fracture problems, it can also lead to

fragmentation of the other particles. At a lower compaction pressure, there was severe spilling of the particles yielding an imperfect geometry. At a higher compaction pressure, cracks were often induced in the samples, sometimes leading to complete fracture, although strong bonding was observed in the undamaged regions in the samples. The samples produced under medium compaction pressures exhibited the highest qualities.

The copper and silica sand nanoparticles are very fine. When high force was applied during compaction, it may cause damages or destruction of the fine particles. According to some researchers [11], the compaction with high force will destruct the particles. When it happens, less particle contact area between copper powders will occur and porosity will increase. The metals also have their own compressibility curve, and according to this curve, 69MPa force for compacting the copper is not recommended.

2. Use the suitable temperature for sintering.

   If the sintering temperature is excessively high, however, some molten particles near the surface of the compact often ooze out from the compact to form globules. This results in non-uniform distribution of cell size and density in the foam as well as undesired shape distortion. In optimum sintering temperature range, the molten particles has a high enough fluidity to flow through the disrupted surface and to penetrate the nearby interstices between the particles but not sufficient to separate from the compact.

3. The copper powder may oxides during sintering process and the sintering process is not homogenous.

   Pure copper metal is reactive and oxidized very quickly. When the copper powder was added with silica sand nanoparticles, the copper powder may react with silica sand nanoparticles during the sintering process. The silica sand (SiO_2) will decompose to become Si and O_2. Then the copper powder will react with O_2

and oxides. When this situation happens, it may affect the volume of the samples/pallets.

Effect of oxidation of copper powder – The non-homogenous sintering process will occur, and the volume of samples/pallets will increase. The oxidation of copper powder will happens at any area of the pallets. The oxidation will increase the void between the particles, and makes the samples/pallets become larger. In less oxidizing atmosphere, the grain boundary mobility is more rapid for the composites. In general, the density for any metal will decreases when the volume increases, or the density is inversely proportional to the volume as stated in the formula below :

$$\rho = m/v \quad \text{..... Equation 1}$$

As conclusion, the sintered densities for pallets will decrease when the volume of the pallets increase. Figure 18 shows the example of sintered pallet which was oxidized :

Figure 18 : The example of sintered pallet which was oxidized

4. Density measuring instrument requires calibration and density should be measured using solid porous method.

All of the above readings were taken using solid method, which is not suitable for measuring the compacted metals. There are 2 methods to combine the metals which are casting and compacting. In casting process, the metals will be melted first and the melted metals will be cooled in the die. For casting, there is no porosity, so the density can be measured by using the solid method. For compacting process, the compacted metals have the porosity. So, the exact method to measure the density for this category is by using the solid porous method.

5.3 Green and sintered density at 41MPa

From the previous results, it seems wrong because the sintered density should be higher than green density. So another force (41MPa) was taken to prove the assumption made above. Table 8 and Table 9 show the green and sintered density values of pure copper and its composites :

Table 8 : Green density (g/cm^3) at 41MPa

Composition of SiO_2	Green density (g/cm^3)				
	550 °C	650 °C	750 °C	850 °C	950 °C
Pure Cu	6.997	6.985	7.201	7.126	7.050
5wt%	5.984	6.119	5.850	6.087	5.996
10wt%	5.044	5.123	4.830	5.091	5.168
15wt%	4.610	4.408	4.502	4.649	4.626
20wt%	4.016	4.242	4.212	4.270	4.258

Table 9 : Sintered density (g/cm^3) at 41MPa

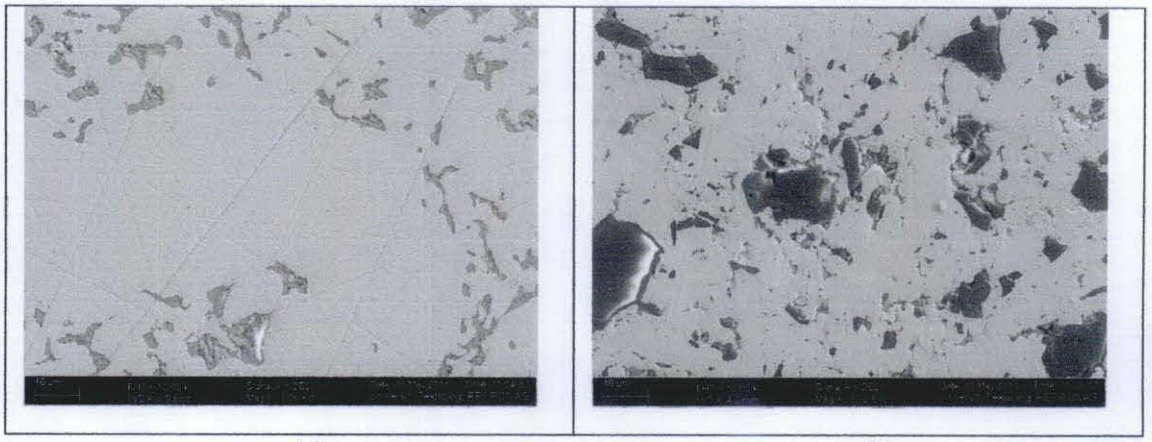
Composition of SiO_2	Sintered density (g/cm^3)				
	550 °C	650 °C	750 °C	850 °C	950 °C
Pure Cu	6.664	6.656	7.418	6.968	6.636
5wt%	5.848	6.088	6.306	6.039	6.319
10wt%	5.008	4.909	5.604	5.079	4.933
15wt%	4.449	4.490	4.601	4.386	4.375
20wt%	4.199	4.278	4.298	4.217	4.198

From our results it is observed that 41 MPa is the compression force which result in best green as well as sintered densities. From our research, it was observed that 41MPa compression force and 750°C of sintering temperature are the suitable parameters which result in better green and sintered densities. Therefore, it is recommended to develop copper based silica sand nanoparticles composites using these parameters for further studies.

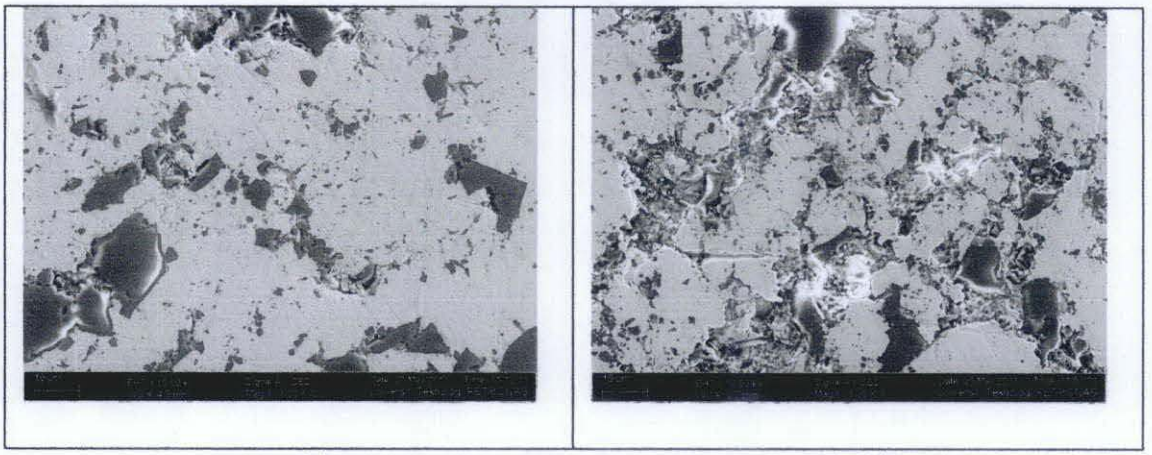
5.4 Microstructure

5.4.1 FESEM image of samples using magnification of 1000 X resolutions

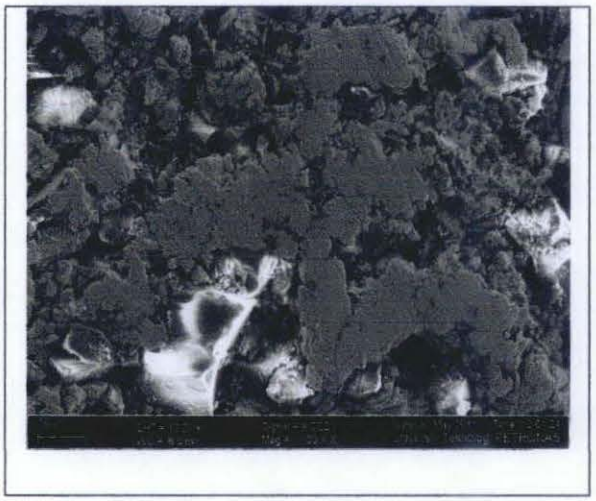
Figure 19 shows FESEM images (1000X) for (a) pure copper and Cu+SiO_2 nanoparticles composites with (b) 5wt% SiO_2, (c) 10wt% SiO_2, (d) 15wt% SiO_2 and (e) 20wt% SiO_2.



(a) (b)



(c) (d)



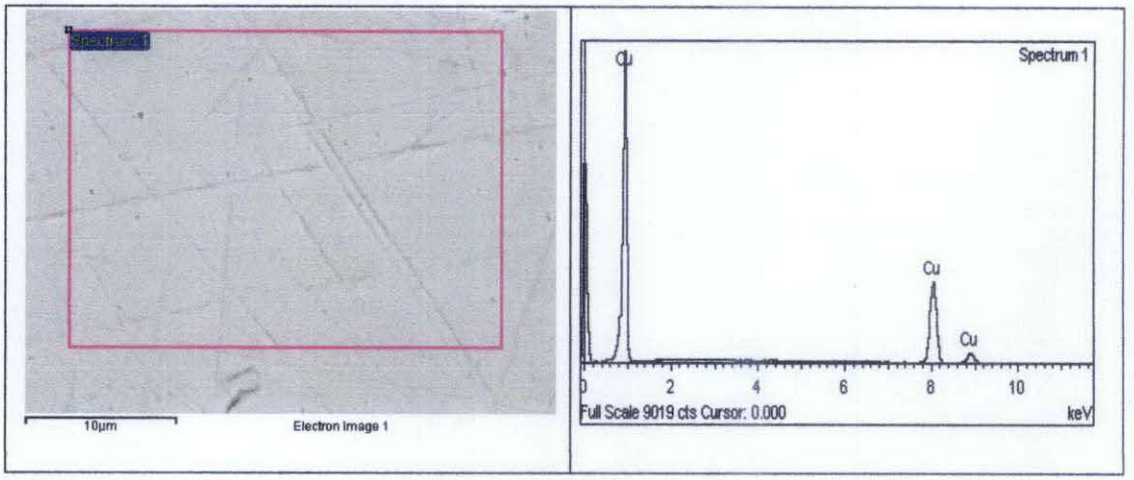
(e)

Figure 19 : FESEM images (1000X) for (a) pure copper and Cu+SiO_2 nanoparticles composites with (b) 5wt% SiO_2, (c) 10wt% SiO_2, (d) 15wt% SiO_2 and (e) 20wt% SiO_2

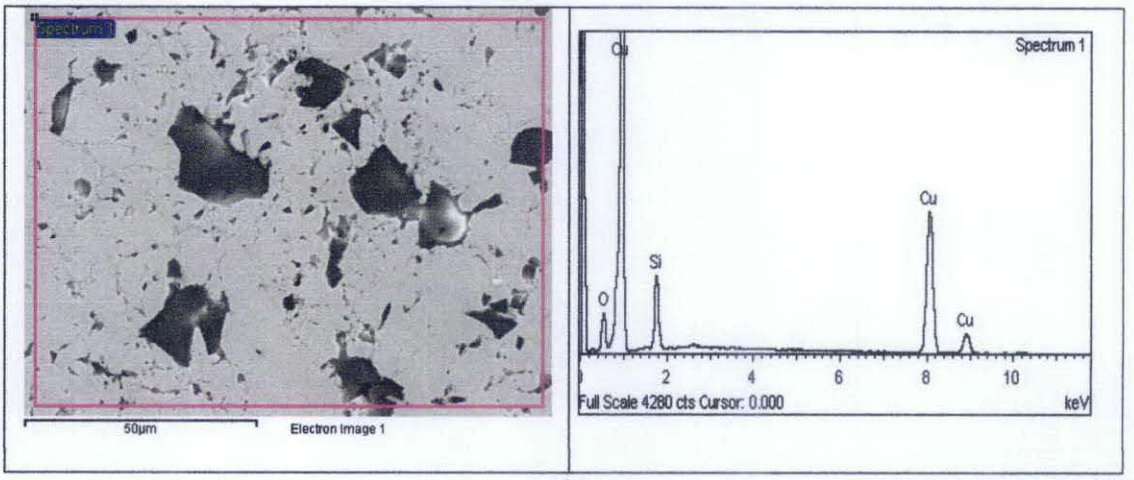
Figures 19 (a, b, c, d and e) shows that the fine silica sand nanoparticles are distributed over the entire area of the copper matrix and occupied the porosity places after sintering. During the powder metallurgy process, milling plays the important role in distributing the silica sand nanoparticles and during sintering, the silica sand nanoparticles will fill the pores between the copper matrix. Increasing trend of silica sand nanoparticles indicated that more pores are filled with 20wt% silica sand nanoparticles as compared to 5wt% silica sand nanoparticles [3]. Pore spaces at Figure 19 (a) will be filled with silica sand nanoparticles after sintering.

5.4.2 FESEM images (whole area) and EDX analysis

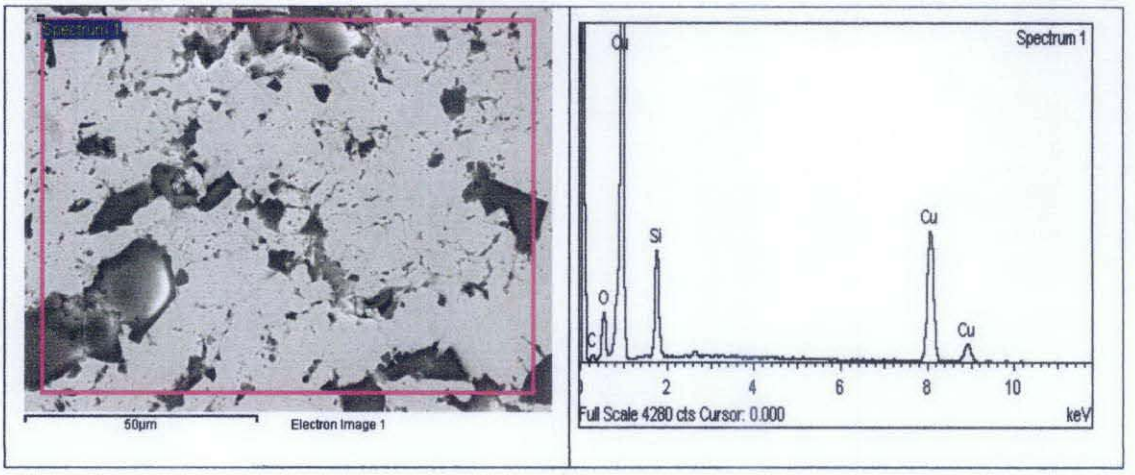
Figure 20 shows EDX (whole area) of (a) pure copper and Cu-SiO2 nanoparticles composites with (b) 5wt% SiO_2, (c) 10wt% SiO_2, (d) 15wt% SiO_2, (e) 20wt% SiO_2.


Spectrum 1
10µm
Electron Image 1
Spectrum 1
Cu
Cu
Cu
0
2
4
6
8
10
Full Scale 9019 cts Cursor: 0.000
keV

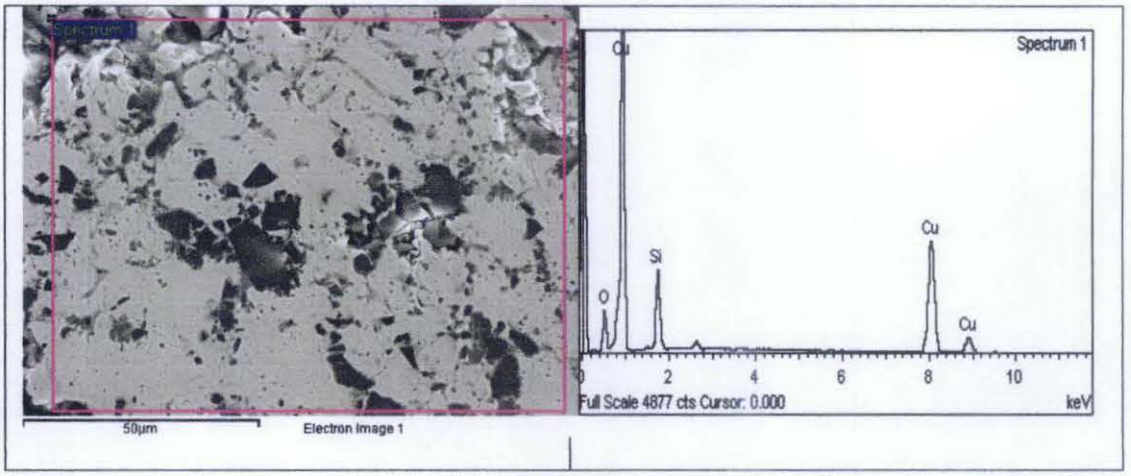
(a)


Spectrum 1
50µm
Electron Image 1
Spectrum 1
Cu
O
Si
Cu
Cu
0
2
4
6
8
10
Full Scale 4260 cts Cursor: 0.000
keV

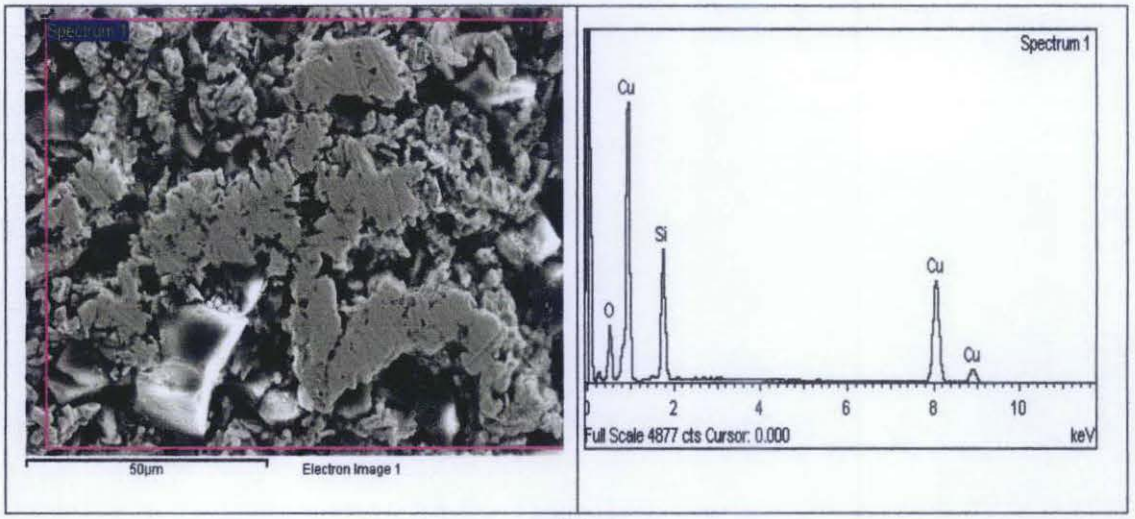
(b)


Spectrum 1
50µm
Electron Image 1
Spectrum 1
Cu
Si
O
C
Cu
Cu
0
2
4
6
8
10
Full Scale 4260 cts Cursor: 0.000
keV

(c)



(d)



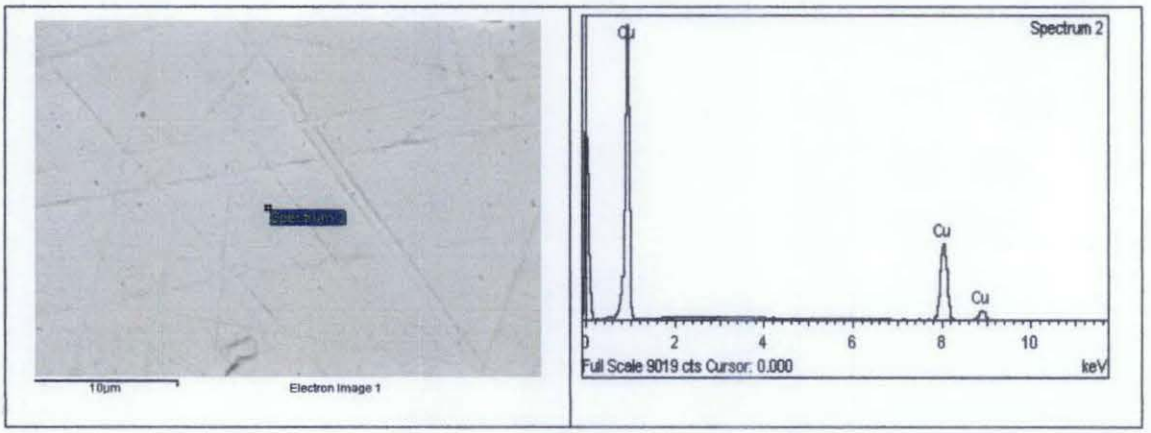
(e)

Figure 20 : EDX (whole area) of (a) pure copper and Cu-SiO2 nanoparticles composites with (b) 5wt% SiO_2, (c) 10wt% SiO_2, (d) 15wt% SiO_2, (e) 20wt% SiO_2

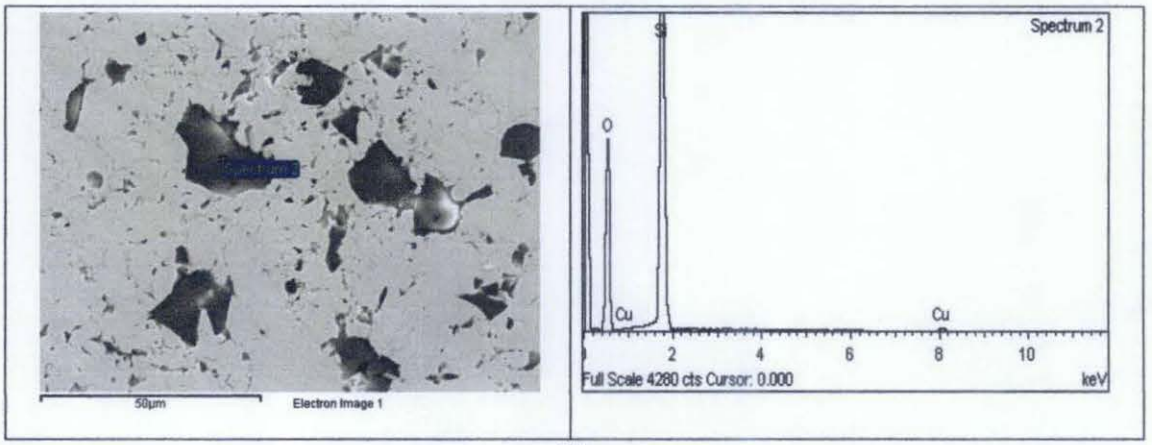
Figures 20 (a, b, c, d and e) shows the EDX analysis that verifies the presence of silica sand nanoparticles in 5%, 10%, 15% and 20wt% copper based composites. The presence of silica sand nanoparticles in copper matrix can be seen from the peak.

5.4.3 FESEM images (pointed area) and EDX analysis

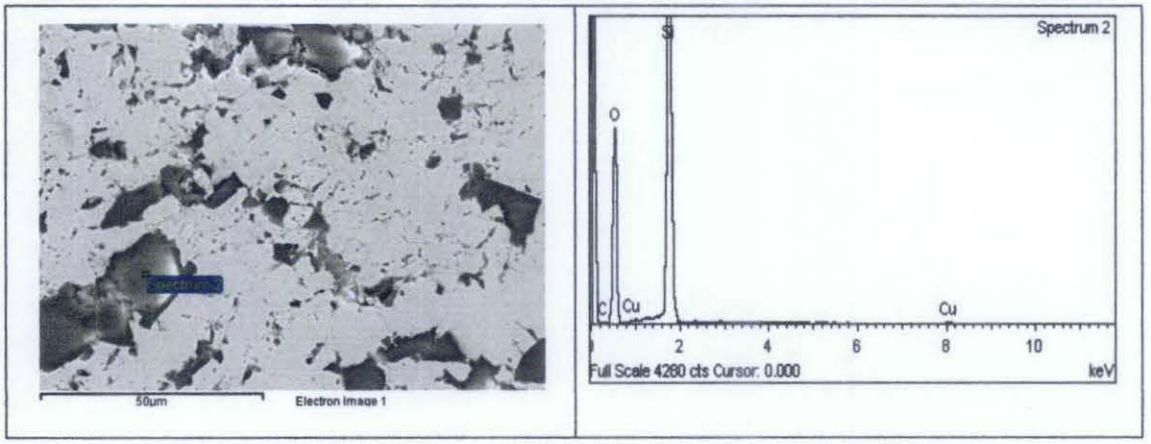
Figure 21 shows FESEM images and EDX (pointed area) analysis of (a) pure copper and Cu-SiO2 nanoparticles composites with (b) 5wt% SiO_2, (c) 10wt% SiO_2, (d) 15wt% SiO_2 and (e) 20wt% SiO_2.



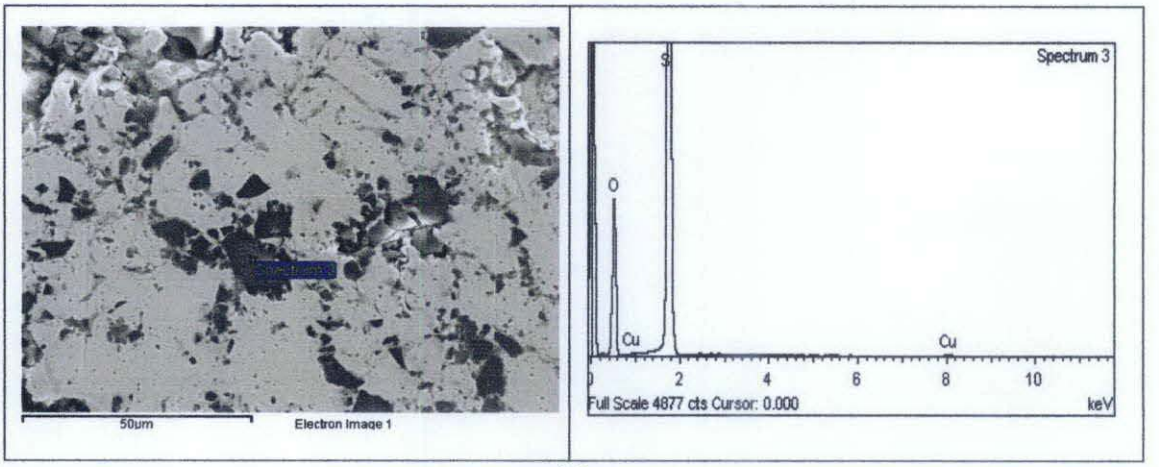
(a)



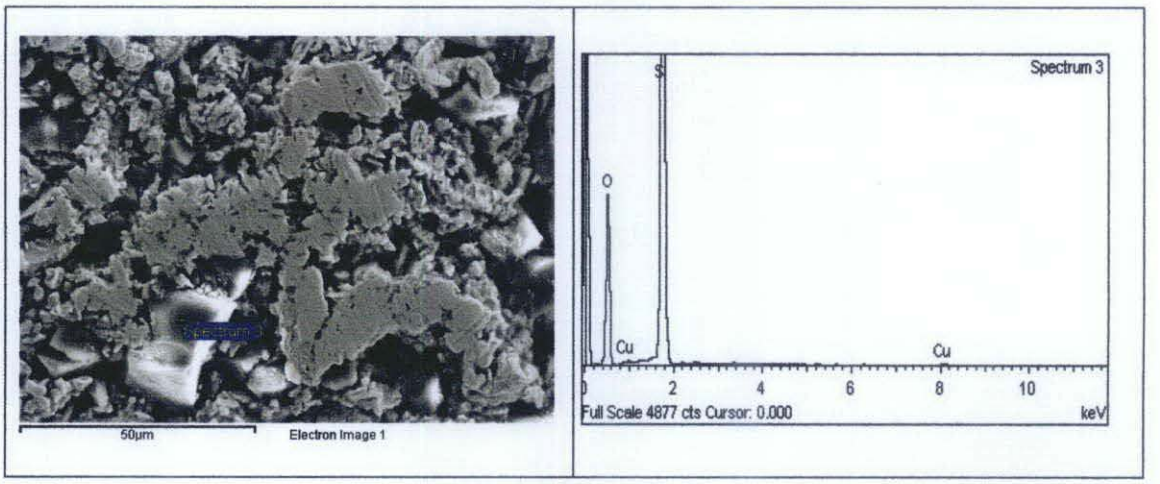
(b)



(c)



(d)



(e)

Figure 21 : FESEM images and EDX (pointed area) analysis of (a) pure copper and Cu-SiO2 nanoparticles composites with (b) 5wt% SiO_2, (c) 10wt% SiO_2, (d) 15wt% SiO_2 and (e) 20wt% SiO_2

Figure 21 (a, b, c, d and e) shows the point EDX analysis where the porous point has been taken to verify the presence of silica sand nanoparticles in 5wt%, 10wt%, 15wt% and 20wt% copper based composites. These nanoparticles occupied the porous places and mixed with copper to fill these porosities to reduce the densities and enhance the mechanical properties. The curve of 20wt% silica sand nanaoparticles is more pronounced as compared to 5wt% silica sand nanoparticles.

5.5 Microhardness

Table 10 gives the hardness values of copper-silica sand nanoparticles composites. As compared to the pure copper, the hardness values of the composite reinforced with 5wt%, 10wt%, 15wt% and 20wt% of silica sand nanoparticles were increased. The hardness of copper generally improves significantly with the additions of silica sand nanoparticles. This can be attributed primarily to the presence of harder silica sand nanoparticles in the copper matrix.

Table 10 : The hardness properties of copper and composite reinforced with 5wt%, 10wt%, 15wt% and 20wt% of silica sand nanoparticles

Composition of SiO_2	Reading 1 (HV)	Reading 2 (HV)	Reading 3 (HV)	Reading 4 (HV)	Reading 5 (HV)	Average (HV)
Pure Cu	36.7	36.5	37.6	37.3	42.8	38.2
5wt%	47.9	52.8	51.7	55.3	45.7	50.7
10wt%	59.9	63.0	53.2	66.1	50.0	58.4
15wt%	73.3	81.3	71.8	82.2	84.5	78.6
20wt%	109.3	125.6	96.9	97.1	96.3	105.0

5.6 Wear resistance

Table 11 and Figure 22 show the weight loss of the pure copper and composite reinforced with 5wt%, 10wt%, 15wt% and 20wt% of silica sand nanoparticles. The pure copper exhibits the maximum weight loss due to its relatively low hardness. The weight losses of the composite decreases with increasing the silica sand nanoparticles volume fractions, from 5wt% to 20wt%.

Table 11 : The weight losses of pure copper and composite reinforced with 5wt%, 10wt%, 15wt% and 20wt% of silica sand nanoparticles

Composition of SiO_2	Before (g)	After (g)	Weight loss (g)
Pure Cu	4.14150	3.95876	0.18274
5wt%	4.23793	4.09205	0.14588
10wt%	4.22975	4.08974	0.14001
15wt%	4.27218	4.13674	0.13544
20wt%	4.33577	4.20753	0.12824

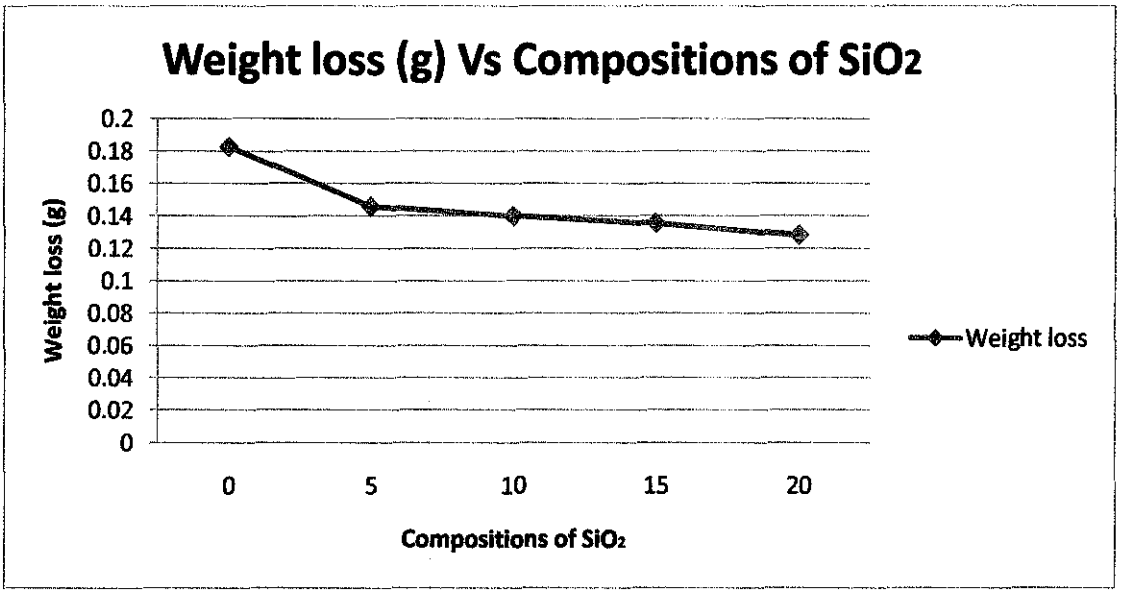


Figure 22 : Weight loss (g) vs compositions of SiO_2

To calculate the wear rate, the mass in air of the sample, m_a and in water, m_w were measured and recorded. The following formulas were used to calculate the density of samples:

$$\rho = \frac{m_a}{m_a - m_w} X \rho_w \quad \text{..... Equation 2}$$

Where ρ_w is the density of water ($1x10^{-3}$ g/mm^3).

Volume loss, W was calculated using the following formula :

$$W = \frac{\Delta m}{\rho} \quad \text{..... Equation 3}$$

Where Δm is the mass loss of specimen due to wear

Table 12 shows the measured m_a, m_w and ρ_{Cu} which will be used to calculate the wear rate.

Table 12 : The measured m_a, m_w and ρ_{Cu}

Composition of SiO_2	m_a (g)	m_w (g)	ρ_{Cu} (g/mm^3)	W (mm^3)
Pure Cu	3.95876	3.55683	0.00985	18.55228
5wt%	4.09205	3.70205	0.01049	13.90658
10wt%	4.08974	3.72535	0.01122	12.47861
15wt%	4.13674	3.83206	0.01358	9.97349
20wt%	4.20753	3.91535	0.01440	8.90556

The wear rate was calculated using the following formula :

$$W(t) = \frac{W}{t} \quad \text{..... Equation 4}$$

Where t is time in seconds (in this experiment, the time is 360 seconds).

Table 13 summaries the calculation and shows the wear rate of each samples.

Table 13 : The wear rate of pure copper and composite reinforced with 5wt%, 10wt%, 15wt% and 20wt% of silica sand nanoparticles

Composition of SiO_2	Wear rate, W(t) (10^{-2} mm^3/s)
Pure Cu	5.153
5wt%	3.863
10wt%	3.466
15wt%	2.770
20wt%	2.474

The wear rates of composite decreases as the silica sand nanoparticles contents increases. The composite with 20wt% of silica sand nanoparticles has higher wear resistance compared to pure copper and composite with 5wt% of silica sand nanoparticles [1,2,4,5,6]. The abrasive wear resistance is defined as the reverse of volume loss [1]. As the conclusion, the increased wear resistance of copper-silica sand nanoparticles in attributed to the increase of the hardness of the composite and slow down in the wear rate of the composite.

6.0 CONCLUSION AND RECOMMENDATION

6.1 Conclusion

One of the objectives for this study is to create compressibility chart for copper, and it was successfully created. The best parameters for this study are 41MPa of compression force and 750°C of sintering temperature. 69MPa is notably high for compacting the copper as this large force could destruct the particles of the sample. The temperature for sintering must be considered in order to get the high densities. This study was done with various temperatures; 550°C, 650°C, 750°C, 850°C and 950°C. 750°C was found to be the suitable temperature because the sintered densities at this temperature were higher than the green densities. The hardness of the composites affects the weight losses and the wear rate of the composites. The hardness of copper matrix increases as the silica sand nanoparticles contents increases from 38.2HV to 105.0HV. As compared with pure copper, the hardness values of the copper-silica sand nanoparticles composite were increased significantly. The wear results showed that the wear resistance of composites increased with increase of the reinforcement weight fraction. The weight losses decreased from 0.18274g to 0.12824g and wear rate decreased from 5.153×10^{-2} mm^3/s to 2.474153×10^{-2} mm^3/s. These are due to the strong particulate matrix bonding and high hardness of the silica sand nanoparticles.

6.2 Recommendation

The experiment of copper composite with silica sand is still new and fresh. Currently, there is only one research paper done. Thus, other properties such as porosity, thermal properties and abrasive wear test with lubricant can be studied for further development of this project.

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