STUDY ON THE CHARACTERIZATION OF SILICA SAND NANOPARTICLES-IRON BASED COMPOSITES (Fe-SiO_2) USING NITROGEN ATMOSPHERE AS SINTERING ENVIRONMENT

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CERTIFICATION OF APPROVAL

Of Research Project

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by

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CERTIFICATION OF ORIGINALITY

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

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ABSTRACT

Metal Matrix Composite has become an attractive alternative in various applications especially in automotive and aerospace industries nowadays. Therefore, in order to study the characterization of Silica Sand Nanoparticles-Iron Based Composites (Fe-SiO_2) using Nitrogen atmosphere as sintering environment, samples were fabricated by mixing pure iron with different weight percentages (5%, 10%, 15% and 20%) of silica sand nanoparticles and undergone several testing and observation before being evaluate.

The silica sand nanoparticles-iron based composites were developed by powder metallurgy technique and being sintered in Nitrogen atmosphere at 1100^{0}C. The characterization that being evaluated include physical properties (density of green and sintered silica sand nanoparticles-iron based composites), microstructure analysis (Optical Microscopy & FESEM), elemental composition (EDX Analysis) and mechanical properties (microhardness test of silica sand nanoparticles-iron based composites).

The research shows densities were reduced from pure iron to 20wt% of silica sand nanoparticles. However, there were increments in each density of the composite after being sintered which at the same time improved its hardness as well. During sintering, the microstructure also exhibits changes in porosity, pore size also pore shape and with the high rate of atomic motion progressively (diffusion) leads to growth of bonds between particles, shown by the microstructure analysis being conducted.

Theoretically stated those properties will improved after sintering process were proven and the composite with highest weight percentage of silica sand nanoparticles (20wt%) offered the best characteristics compared to others.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Iron is one of the well-known ferromagnetic metals and compared to aluminum, iron is much softer which made it more useful for numerous applications [German, 1998]. At the moment, many studies had been made in order to improve the properties of iron such as by smelting process with carbon and by alloying a partial or complete solid solution of one or more elements in a metallic matrix.

Silica sand is one of the most common found sand in the world which can be commercially utilized for various applications. Besides that, it can also be introduced as alloying element for composite production.

Composite is considered to be any multi-phase material that exhibits a significant proportion of the properties of both constituent phases such that a better combination of properties is realized [Callister, 2007]. This project will work on a new composite which is Metal Matrix Composite (MMC). MMC is composite material formed by the reinforcement of organic compound or ceramics (silica sand nanoparticles, SiO_2) and a matrix (pure iron, Fe). More studies have been done on MMC lately since it has a potential for aerospace and automotive industries. Developing MMC allows it to be utilized at higher service temperatures than their base metal counterpart; furthermore, the reinforcement may improve some mechanical properties as well [Callister, 2007]. At the same time, the coefficient of linear thermal expansion of MMC can also be reduced due to the addition of ceramic reinforcement which is important in some application nowadays [Gul, 2008].

1.2 Problem Statement

Both metal (iron) and ceramic (silica sand) have their own interesting characteristics and abilities which can be utilized for various applications. Iron or metal is known as a material with high density, thermal and electric conductivity besides able to be deformed without cleaving under stress. However, pure iron is not suitable to be used in certain application involving high temperature. [German, 1998]. Meanwhile ceramic are known as a material with high temperature capabilities and mechanical strength besides low coefficient of thermal expansion. Therefore, in order to enhance pure iron's properties, the idea to produce Metal Matrix Composite (MMC) will be used with the combination of both materials (pure iron, Fe as matrix and Silica Sand Nanoparticles, SiO_2 as ceramics) by using powder metallurgy technique. Thus, some properties of the Silica Sand Nanoparticles-Iron Based Composites will be evaluated throughout this project.

1.3 Significance of the Project

Metal Matrix Composite's (MMC) development has become an attractive alternative in numerous industrial applications especially in aerospace and automotive industries. Previously, the development of MMC focus on reinforcement using Micro-sized particles. As the technologies grows rapidly nowadays, the stage of MMC's development has evolved and more focus on Nano-sized particles reinforcement. Therefore, the research will not only contribute something good for the engineering material field, but also will develop the capabilities among UTP students, hence building the credibility of UTP itself towards the successfulness of UTP's aim which is to lead in research and development area.

1.4 Objectives

This project is a research to study/investigate the effect of silica sand nanoparticles reinforcement on the mechanical properties of iron based composites

1.5 Scope of Study

In this research, pure iron (Fe) is chosen as the matrix base metal meanwhile silica sand nanoparticles (SiO_2) are chosen as the ceramic reinforcement.

Metal Matrix Composite (MMC) can provide many advantageous characteristic due to the reinforcement of ceramic into the base metal and hence, become an attractive research in recent years. In this project, MMC is developed using powder metallurgical technique with different weight percentages of Silica Sand Nanoparticles which are 5%, 10%, 15% and 20%.

After fabrication, physical properties of MMC or silica sand nanoparticles-iron based composites are evaluated by comparison between the density of green and sintered composites. This will be determined by using Mettler Toledo AX205 instrument.

The process will be continued by analyzing the microstructure of silica sand nanoparticles-iron based composites using Optical Microscope (OM) and Field Emission Scanning Electron Microscopic (FESEM).

Besides, the elemental composition of silica sand nanoparticles-iron based composites will also be determined using Energy Dispersive X-Ray Spectroscopy (EDX) that being attached together with the FESEM machine.

The silica sand nanoparticles-iron based composites will also undergo Microhardness test using hardness testing instrument. The measurement that will be used in this testing is Vickers Hardness test.

All the details involve in this research can be simplified as the Table 1.1 below for better understanding.

Table 1.1: Details of Research

Matrix	Pure Iron (Fe)
Reinforcement	Silica Sand Nanoparticles (SiO_2)
Fabrication Method	Powder Metallurgic Technic 1. Blending/Mixing 2. Compaction 3. Sintering using Nitrogen atmosphere as sintering environment at 1100^0C
Physical Properties	1. Theoretical Density 2. Green Density 3. Sintered Density
Microstructure Analysis	1. Optical Microscope Analysis 2. Field Emission Scanning Electron Microscope
Elemental Composition Properties	Energy Dispersive X-Ray Spectroscopy
Mechanical Properties	Microhardness Testing

1.6 Relevancy of the Project

As a future mechanical engineer, conducting this project will give more understanding in manufacturing process and mechanical behaviour of material besides enhancing capabilities among students. Furthermore, this project is relevant to Mechanical Engineering academic syllabus of Universiti Teknologi PETRONAS (UTP) especially in Material field. This project is also known as an attractive alternative for aerospace and automotive industries.

1.7 Feasibility of the Project

Mainly, this project will need more research on the microstructure besides physical and mechanical properties of silica sand nanoparticles-iron based composites. Some laboratory works such as observations and testing will be involved in order to complete it. In addition, all laboratory equipment are provided by the university. Therefore, this project could be done, hence achieve the objectives if the work flows according to the planning.

CHAPTER 2

LITERATURE REVIEW

2.1 Materials

Iron is the most common element found from in earth's crust and the symbol for this element is Fe. Pure iron is known to be softer than aluminum which made it more useful for numerous applications since the ancient times. The properties of iron such as strength and hardness can be improved, for example by smelting process with carbon to produce steels which can lead up to 1000 times harder than pure iron. Iron also exists in wide range of oxidation states besides reactive to oxygen and water [Fu Lin, 2002]. Table 2.1 below represents general properties of iron [German, 1998].

Table 2.1: General Properties of Iron [German, 1998]

Material	Melting Point	Boiling Point	Density
Iron	1538^0C	2862^0C	7.874g/cm^3

Silica sand is one of the most common found sand in the world which can be commercially utilized for various applications. Silica is another name for Silicon Dioxide (SiO2), of which quartz is a specific latticed structure. Quartz, Tridynamite and Cristobalite are the three crystalline form of silica. Silica has several interesting characteristics such as good abrasion resistance, electric insulation besides having high thermal stability which makes it useful for industrial applications. At the same time, silica is known to be insoluble in most acids [Chawla, 2006]. Table 2.2 represents general properties of Silica [Callister, 2007].

Table 2.2: General Properties of Silica [Callister, 2007]

Material	Melting Point	Boiling Point	Density
Silica	1830^0C	2230^0C	2.65g/cm^3

Composite is a material formed by combining two or more constituent materials with having quite different physical, chemical and mechanical properties. Those different materials work together in order to offer unique properties for the composite produced. Engineers and scientists have ingeniously combined various type of material such as metals, ceramics and polymers to produce new generation of extraordinary composites. There are three types of common composites nowadays which are [Kainer, 2006]:

1. Polymer Matrix Composite (PMC) – Combination of polymer resin as the matrix and fibers as the reinforcement
2. Metal Matrix Composite (MMC) – Combination of metals as the matrix and ceramic or organic compound as the reinforcement
3. Ceramic Matrix Composites (CMC) – Combination of ceramic matrix with reinforced ceramic phase

Since this project involves Metal Matrix Composite (MMC), it is important to understand the basic concept of MMC first. In developing MMC, metal or metallic ally will be the matrix while ceramic or organic compound as the reinforcement. Generally, there are three kinds of MMC commonly produced shown in Figure 2.1 which are [Chawla, 2006]:

1. Particle reinforced MMC
2. Short fiber or whisker reinforced MMC
3. Continuous fiber or sheet reinforced MMC

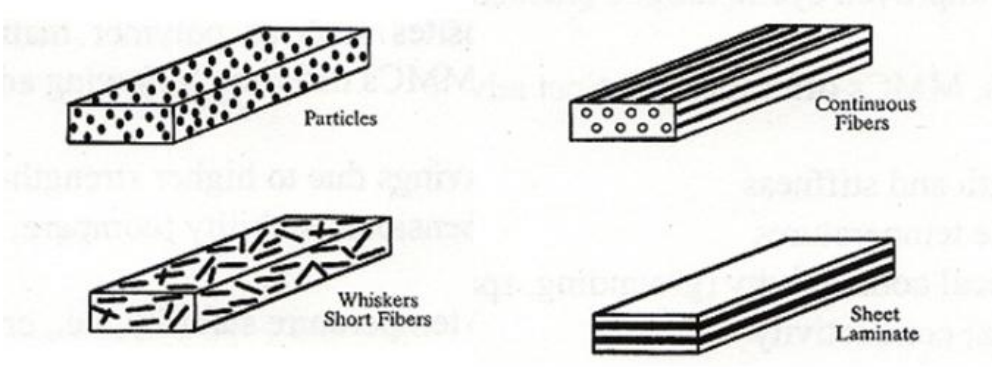


Figure 2.1: Type of MMC Commonly Produced [Chawla, 2006]

It is stated that particle reinforced MMC is the most preferable MMC for most industrial applications. This is because, compared to the other types of MMC, particle reinforced MMC is less expensive, which mean it can save cost in for the production. Besides, the techniques to produce MMC using particle reinforcement are not so complicated. Conventional metallurgical processing technique such as casting or powder metallurgy, then followed by conventional secondary processing by rolling, forging and extrusion can be used. The properties of MMC developed by using particle reinforcement is relatively isotropic, hence it is preferable compared to other type of MMC [D. Huda, 1995], [Kainer, 2006], [Chawla, 2006].

2.2 Fabrication (Powder Metallurgy Processing)

In this project, powder metallurgy technique will be used to produce particle reinforced MMC. Blending/mixing, compressing/compaction and sintering are three main steps involved in powder metallurgy processing. Figure 2.2 below shows the summary of powder metallurgy technique which will be used in the project [Baglyuk, 2001], [Liu, 1994].

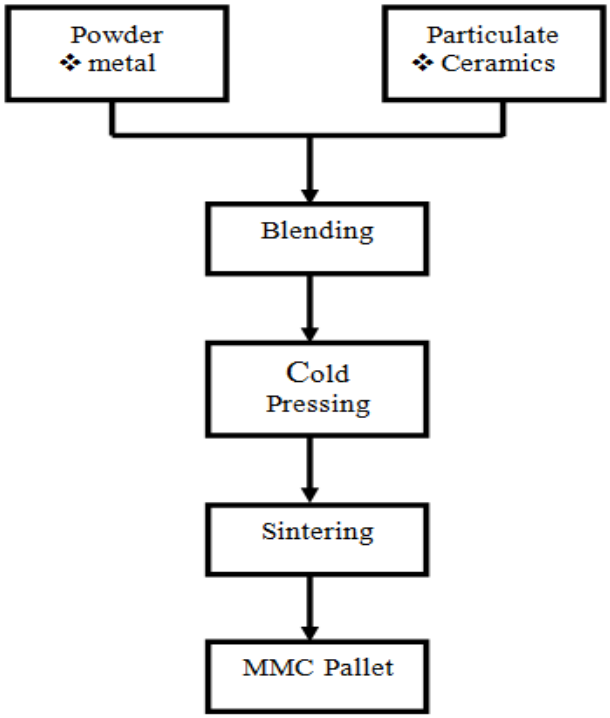


Figure 2.2: Powder Metallurgy Process Technique [Liu, 1994]

2.2.1 Blending/Mixing

After materials had been prepared according to each composition, they will be blended and mixed together. During this process, materials can be mixed using a steel container with steel balls in a planetary ball mill, a glass vessel in a tumble mixer or in attritor [Eroglu, 2000]. It is important to add binding agent into the mixture so that it is more adhesive and hence, the particles are hold into compacted shape until sintering process. Wax is one of commonly used binding agent since it is low cost, easy to use and less hazardous. It is important to ensure the mixture to be homogeneously mixed to avoid internal porosity which can cause uneven shape, non-uniformed viscosities and lead to some difficulties during sintering. In addition, mixing time and rotation rate are also important during this preparation. The mixture will become more difficult to be compacted if the time taken is too fast or too long. Therefore, mixing process is usually performed for about 30 minutes [Staniek, 1993], [German, 1998].

2.2.2 Compressing/Compaction

The particles will deform, bond and harden during compressing/ compaction process. The compacted particles will become harder and higher in density if the compaction pressure is greater. However, the output from the compaction is usually 80% dense compared to theoretical value. For iron powder, the compaction pressure that usually used is around 550-700MPa or 30-50psi. In order to ease ejection process and minimize die wear because of friction between die wall and the powder during compaction, lubricant is required [Liu, 1994].

2.2.3 Sintering

Sintering is a process of heating the green compact at certain temperature which can lead the particles to weld their bond between themselves due to high rate of atomic motion (diffusion), so that the green

compact will be transformed into a high strength structure. Some degree of liquid phase will be obtained to allow it flows through the pores resulting in densification of the composite during sintering process. Other than that, the microstructure of compacted composite will experienced changes in the grain size, porosity, pore size and pore shape. Theoretically, the higher temperature of sintering process will increase in atomic motion rate [German, 1998], [S. Azis, 2007].

In order to allow heat uniformly soak the green compact, they usually are heated to a peak temperature for few minutes. Both binder and lubricants will be burn out and extracted at the temperature of 120^0C until below 550^0C. Then, the atomic motion is increased as well as sintering bonding at the temperature range of 1100-1350^0C for iron [Anand, 2006].

During sintering, atmosphere that been provided throughout the entire process play an important role to avoid any inconvenience to the compact (iron) which can affect the final properties of samples later on. In normal air, iron can forms an oxide and with the increasing in temperature, the rate of oxidation will also increase. Therefore to avoid this problem from happened, sintering of iron must be conducted in protective atmosphere, either by using nitrogen, hydrogen or argon gasses [German, 1998], [Anand, 2006].

2.3 Related Works

2.3.1 Tahir Ahmed, Othman Mamat, "*Characterization and Properties of Iron-Silica Sand Nanoparticles Composites",* Universiti Teknologi PETRONAS, Malaysia, 2010

This research is basically set as the main reference, since it is a study on characterization and properties of iron-silica sand nanoparticles composites. The MMC is made of pure iron powder with the reinforcement of silica sand nanoparticles (5-20%) using powder metallurgy technique and being sintered at different temperatures (900^0C, 1000^0C and 1100^0C) in Argon

atmosphere. The characteristics that being evaluated include density, microstructure analysis, elemental composition, hardness and wear-resistance. It is stated that the properties of metal matrix particulate composite depend mainly upon the microstructure and properties of matrix material, nature of particles, the distribution, size and shape of particles besides the interfacial bahaviour between particles and matrix. Increasing the sintered temperature will improve the mechanical properties and the microstructure of composite due to more diffusion of silica sand nanoparticles into porous sites. An optimum value of 20wt% of silica sand nanoparticles reinforcement is found to have the best microhardness values for all sintering temperature as shown in **Appendix I**.

2.3.2 Saidatulakmar Shamsuddin, Shamsul Baharin Jamaludin, Zuhailawati Hussain, Zainal Arifin Ahmad, *Characterization of Fe-Cr-Al2O3 Composite Fabricated by Powder Metallurgy Method with Varying Weight Percentage of Alumina,* Faculty of Applied Science, Universiti Teknologi Mara, 2008

This paper provides information and guideline for the development of composite by using powder metallurgy technique with different weight percentages of reinforcement. In this research, alumina particles with the weight percentages of 5-25% are added into the matrix which is iron-chromium alloy. Theoretically, varying the weight percentages of reinforcement can affect the density porosity, hardness and shrinkage of a composite. This is then proved by the results in **Appendix II** that stated higher in weight percentages of reinforcement will cause higher in porosity which will reduce the density of composites. However, at the same time the hardness of composite is increasing. This research provides fundamental understanding of composite development which is beneficial for others.

2.3.3 H. Gul, F. Kilic, S. Aslan, A. Alp, H. Akbulut, "*Characteristics of Electro-Co-Deposited Ni-Al2O3 Nano-Particle Reinforced Metal Matrix Composite (MMC) Coatings",* Department of Metallurgical & Materials Engineering, Sakarya University, 2008

In this research, a pallet of metal matrix composite coating is developed where Al_2O_3 nanoparticle with the average size of 80nm are reinforced into nickel matrix. This research mainly focus on how surface hardness and wear resistance of electrodeposited can be increased. Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD) is being utilized to investigate the characteristic of this metal matrix coating, for example in **Appendix III**. As the result, there is increment in wear resistance of the nano composites compared to pure Ni at about 2-3.5 times higher. Therefore, it is shown that mechanical properties of MMC can be improved by the addition of nano-particulate reinforcement.

2.3.4 G.Staniek, F. Lehnert, M. Peters, W.Bunk and W.A.Kaysser, "*Powder Metallurgical Processing of a SiC Particle reinforced Al-6wt% Fe Alloy",* Institute of Material Research, German Aerospace Research, 1993

This paper basically focus on the study of different fraction of SiC particulate (10-15%) reinforcement towards the Al-6wt% Fe-Alloy matrix developed using powder metallurgy technique. There are some guidelines provided by this research on how to conduct powder metallurgy technique including proper mixing, compaction and sintering method. Besides, this research also study on the mechanical properties of composite with different type of mixing which are tumble mixer, ball mill and attritor. By the end of this research, there are some improvement in yield strength and elastic modulus of composites by the addition of 10-15% of SiC particle as shown in **Appendix IV**.

2.3.5 Saurabh Anand and Neerav Verma, "*Effect of sintering temperature, heat treatment and tempering on hardness of sintered hardened grade steels",* Department of Materials and Metallurgical Engineering, Indian Institute of Technology, 2006

This research is conducted to study the effect of different sintering temperatures which are 1120^0C, 1180^0C and 1250^0C towards the hardness of sintered hardened grade steel. The characteristic of density and densification are also evaluated as shown in **Appendix V**. The methodology and parameter used to conduct the research are related and can be used for other researches.

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

There are several methodology/steps/activity that need to be conducted in order to complete the research successfully. The Figure 3.1 below represents the flow of activities throughout the project.

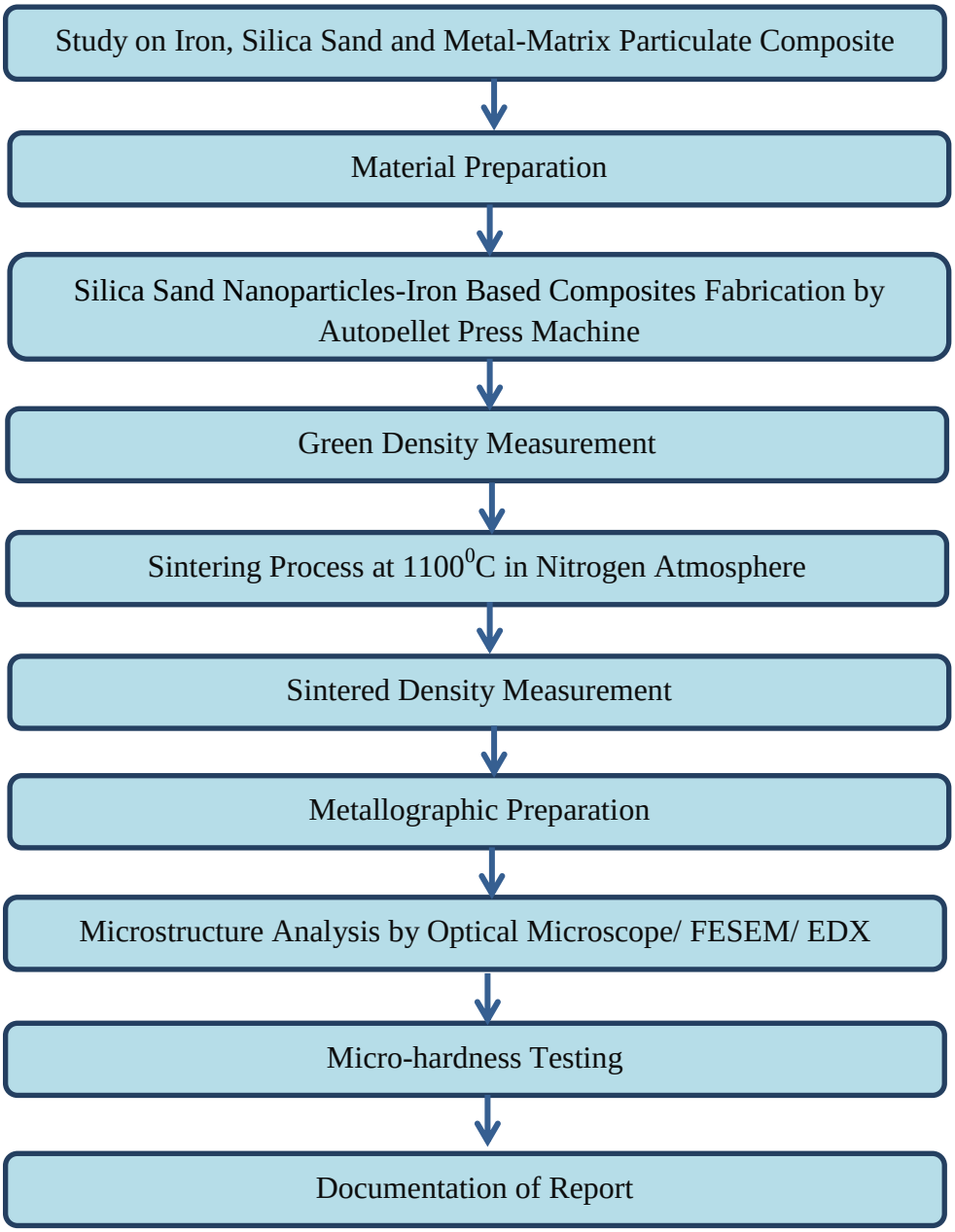


Figure 3.1 : Flow of Activities

3.2 Specific Project Activities

3.2.1 Study on Iron, Silica Sand and Metal Matrix Particulate Composite

First and for most, the project began with research on several issues which were on iron, silica sand and metal matrix particulate composite. These were the materials used throughout the project. The information from journal papers and books were gather and the area that been stressed include the material background, fabrication, testing that can be conducted with the standard procedure.

3.2.2 Material Preparation

The iron powder (99.5%) that was used is commercially available with average size of 10um. Meanwhile for the matrix, silica sand nanoparticles (95%) with average size of 54.18 nm (intensity produced by Zetasizer Nano Analyzer) originated from Tronoh, Perak were utilized.

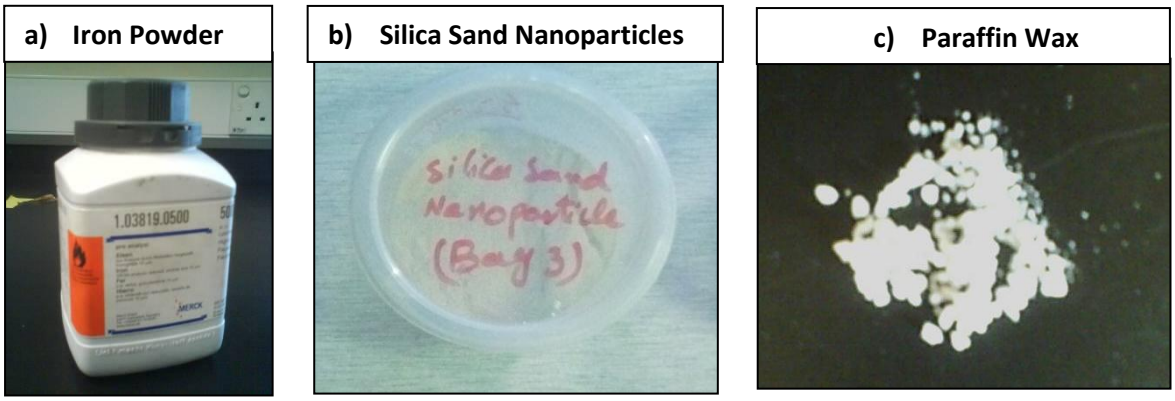


Figure 3.2 : Material Used Throughout The Project; a) Iron Powder b) Silica Sand Nanoparticles c) Paraffin Wax

Figure 3.3 shows the average size for the silica particles which were qualified to be considered as SiO_2 nanoparticles with the size of 54.18 nm [Tahir, 2010].

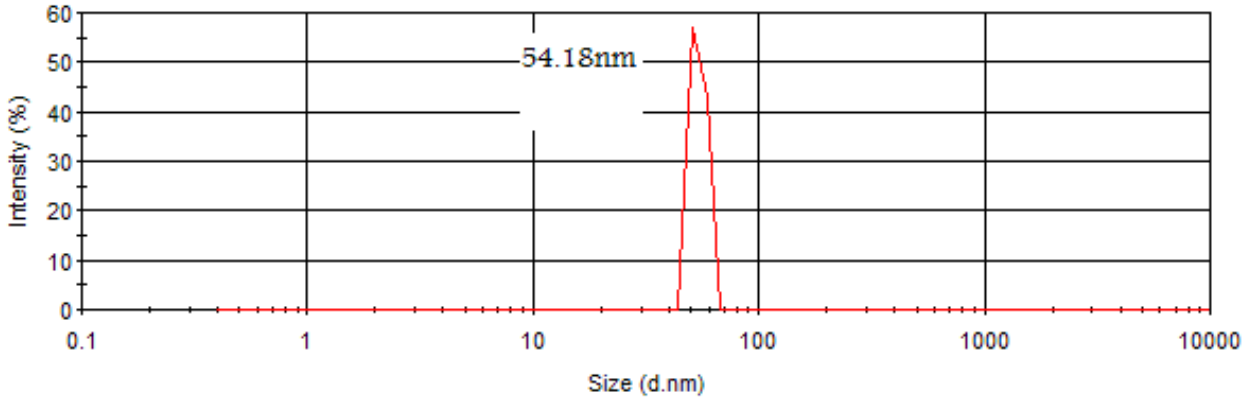


Figure 3.3 : Particle Size Distribution by Intensity Produced by Zetasizer Nano Analyser with the Average Size of 54.18 nm [Tahir, 2010]

Figure 3.4 shows the morphology, size and distribution of the SiO_2 sand nanoparticles. It can be seen that some of SiO_2 particulates had fused together resulted from squeezing action of the ball-milling process. Figure 3.5 shows the EDX analysis on the silica sand rich phase which indicate the present of high Si with little amount of Al [Tahir, 2010].

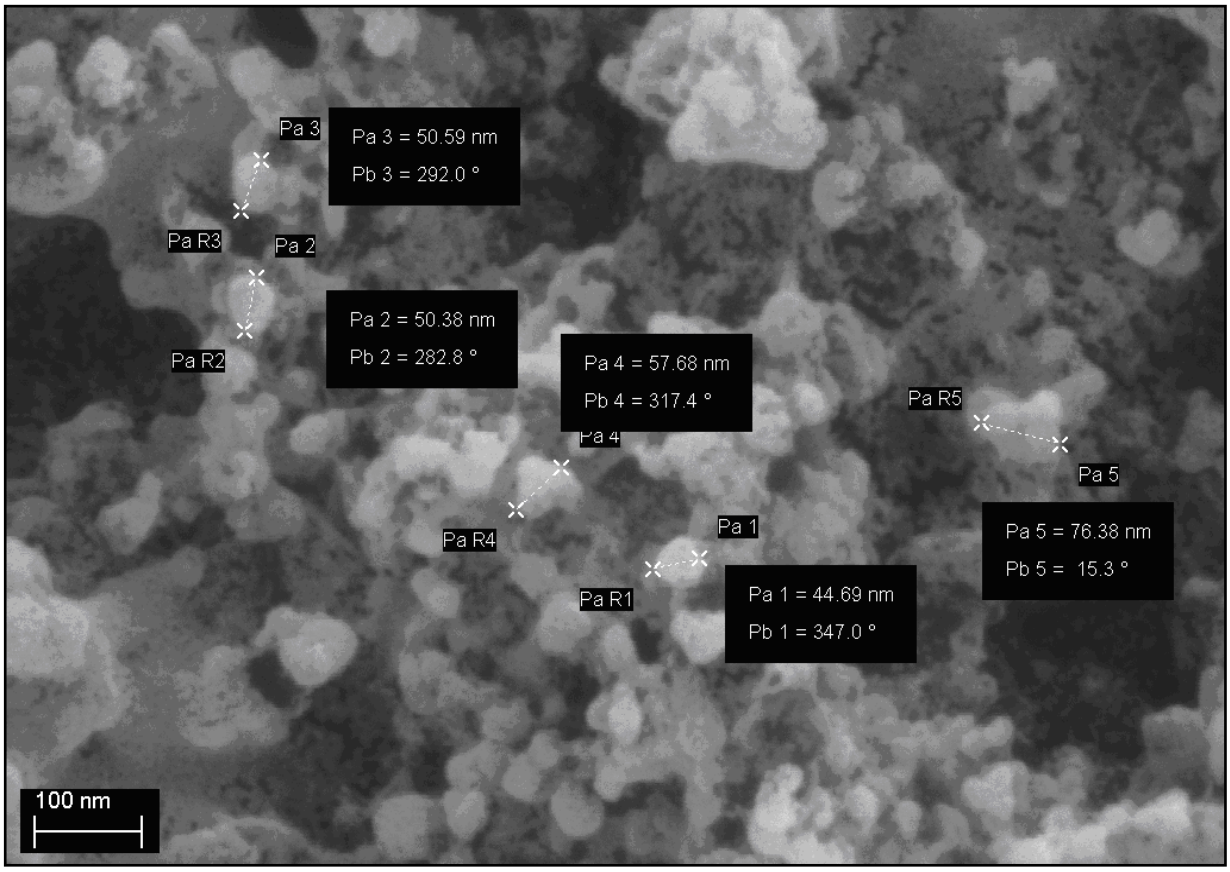
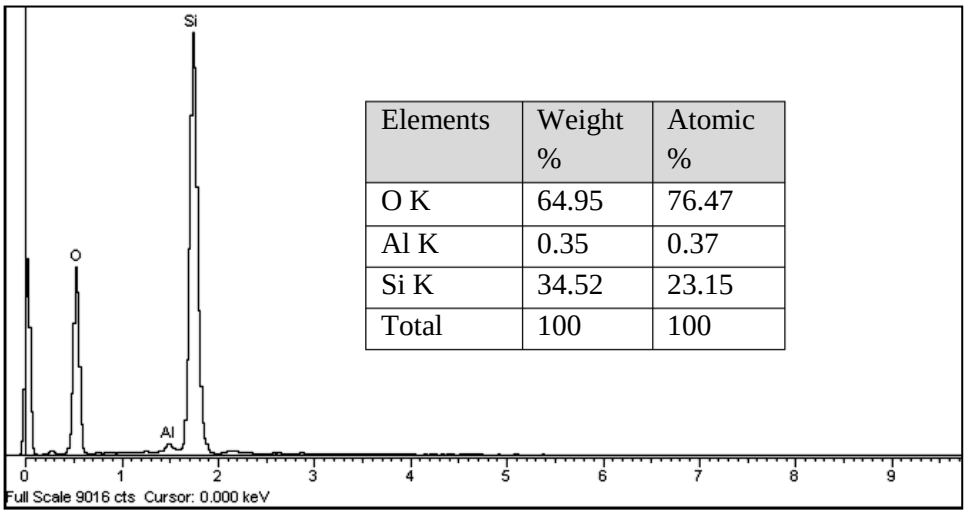


Figure 3.4 : FESEM Image of the Silica Sand Nanoparticles [Tahir, 2010]



Elements	Weight %	Atomic %
O K	64.95	76.47
Al K	0.35	0.37
Si K	34.52	23.15
Total	100	100

Figure 3.5 : EDX Analysis of Silica Sand Nanoparticles [Tahir, 2010]

Both iron powder and silica sand nanoparticles were mixed according to the composition which are pure iron, 5wt% of silica sand, 10wt% of silica sand, 15wt% of silica sand and 20wt% of silica sand with the addition of wax. Binding agent which was 0.1% Paraffin Wax was used to ensure the mixtures more adhesive. 5grams of mixture were needed in order to prepare a sample. The weight ratio for each mixture was measured by using Mettler Toledo AX205 instrument in Figure 3.6 below.

Figure 3.6 : Mettler Toledo AX205 (Weight)

The composition weight of iron and silica sand nanoparticles that were made are represented by the Table 3.1 below:

Table 3.1 : Weight Composition of Iron and Silica Sand Nanoparticles

Composition (total 5grams)	Iron Powder (g)	Silica Sand Nanoparticles (g)
Iron + 5%wt silica sand nanoparticles	4.7500	0.2500
Iron + 10%wt silica sand nanoparticles	4.5000	0.5000
Iron + 15%wt silica sand nanoparticles	4.2500	0.7500
Iron + 20%wt silica sand nanoparticles	4.0000	1.0000

Each composition was then being ball mill for one hour to make the mixture homogeneous as shown in Figure 3.7 below. This process was done to ensure the consistency of results later.

Figure 3.7 : Ball Mill Machine

3.2.3 Silica Sand Nanoparticles-Iron Based Composites Fabrication

The 5grams mixture of iron powder and silica sand nanoparticles was then compacted by using Auto Pallet Press Machine (Figure 3.8). The mixture was compacted at the force of 200MPa, dwell time was 3minutes and decomposition time was 1minute. The dimension and shape of the

samples followed the metallic mould shape. In case of broken specimens, additional specimens were fabricated as backup.

Figure 3.8 : Auto Pallet Press Machine

3.2.4 Green Density Measurement

The basic information to be obtained for this project was each sample's green density. This is because, the values will be compared to the sintered density afterwards. In order to measure the density, Mettler Toledo AX205 instrument in Figure 3.9 below was used with respect to Archimedes' Method.

Figure 3.9 : Mettler Toledo AX205 (Density)

3.2.5 Sintering Process

All compacted samples were sintered by using sintering furnace (Figure 3.10) at 1100^{o}C for two hours under nitrogen atmosphere as sintering environment. Throughout this process, the heating and cooling rates that been used were 5^{o}C/min and 10^{o}C/min respectively. All parameters were being set as referred to the iron-carbon phase diagram shown in Figure 3.11 below.

Figure 3.10 : Sintering Furnace

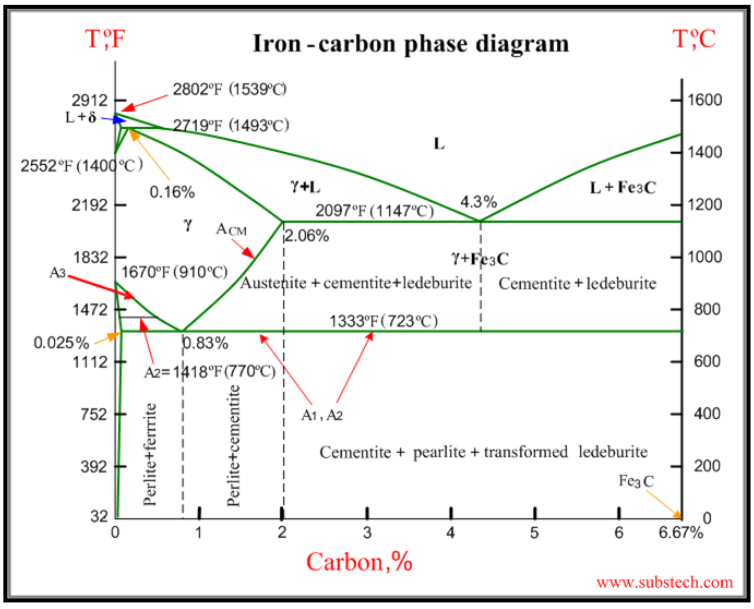


Figure 3.11 : The Iron-Carbon Phase Diagram [Callister, 2007]

3.2.6 Sintered Density Measurement

The densities of all samples after being sintered were measured by using Mettler Toledo AX205 instrument with respect to Archimedes' Method. Then, the values will be compared with green density values.

3.2.7 .Microstructure Analysis

The purpose of this analysis was to examine the distribution of silica sand nanoparticles in iron for each composition before and after sintering process. There are two types of observation were done which are Optical Microscope (OM) and Field Emission Scanning Electron Microscopic (FESEM).

a) Optical Microscope (Figure 3.12)

Analysis was being made by taking pictures in the magnification range of 5X, 10X, 50X and 100X.

b) FESEM Analysis (Figure 3.13)

FESEM Analysis was used to observe microstructure at the magnification of 1000X with the resolution of 1nm. The observation covered whole area analysis and point analysis. In addition, the elemental composition were also be determined using Energy Dispersive X-Ray Spectroscopy (EDX) that being attached together with the FESEM machine

Figure 3.12 : Optical Microscope

Figure 3.13 : FESEM

There are few steps taken before conducting microstructure analysis which called metallographic preparation. The samples need to be cut into smaller size, mounted, grinded and polished first.

Step 1: Cutting Process

The samples were cut into smaller size for mounting purpose by using abrasive water jet machine.

Step 2: Mounting Process

In order to make the process more convenient, the samples were mounted using Auto Mounting Press machine in Figure 3.14 under certain parameters which were:

- Material: Bakelite powder
- Quantity: 1 full scoop
- Pressure: 4000 psi
- Dwell Time: 10minutes

Figure 3.14 : Auto Mounting Press

Step 2: Mounting Process

The mounted samples were grinded to produce a flat surface. During grinding, water is supplied as lubrication to keep the process cool and to remove grinding waste. This grinding process required user to use from small grade (rough) to high grade (smooth) of SiC waterproof paper. After finish grinding, process were continued with polishing. This process was conducted to make sure the samples' surface are perfectly

flat and shine as a mirror after fine scratches have been removed. Both processes used the same machine which was grinder as Figure 3.15 below. The difference was by changing the SiC waterproof paper to cloth impregnated with a very fine abrasive (1 μm diamond) compound.

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Figure 3.15 : Grinder Machine

3.2.8 Microhardness Testing

There were three most commonly methods used for hardness testing which were Brinell, Vikers and Rockwell hardness test. The samples' surface must be bright and smooth to ensure the accuracy for hardness value. For this project, the method used for the hardness measurement was Vickers Hardness Test. The load used for this test was 300-gf for 15 second with diamond indenter as shown in Figure 3.16 below.

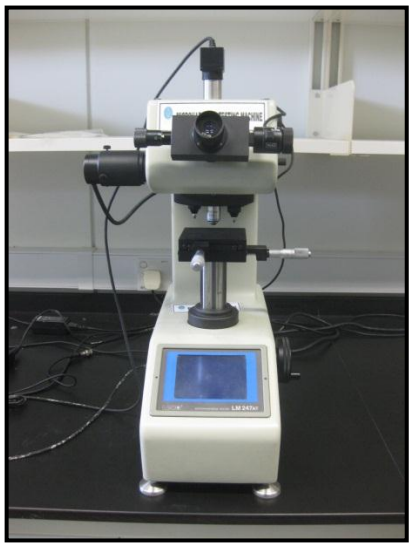


Figure 3.16 : Microhardness Testing Instrument

3.3 Gantt Chart and Key Milestone for FYP II

Table below represents the workflow for FYP II (September2011)

Table 3.2: Gantt chart for FYP II

ACTIVITIES	Weeks										
	6	7	8	9	10	11	12	13	14	15	16
Research and Background Studies of the Project											
Material Preparation											
Sample Fabrication											
Green Density Measurement			▲								
Sintering Process											
Sintered Density Measurement				▲							
Preparation of Progress Report											
Submission of Progress Report							▲				
Metallographic Preparation											
Optical Microscopy						▲					
FESEM and EDX								▲			
Microhardness Test								▲			
Data Analysis											
Preparation of Final Dissertation and Technical Paper											
Oral Presentation									▲		
Submission of Final Dissertation									▲		
Submission of Technical Paper										▲	
Submission of Project Hardbound											▲

▲ = Key Milestone

CHAPTER 4

RESULT AND DISCUSSION

4.1 Silica Sand Nanoparticles-Iron Based Composites Samples

Silica sand nanoparticles-iron based composites samples were compacted by using Auto Pallet Press Machine. Figure 4.1 below shows the samples that were fabricated:

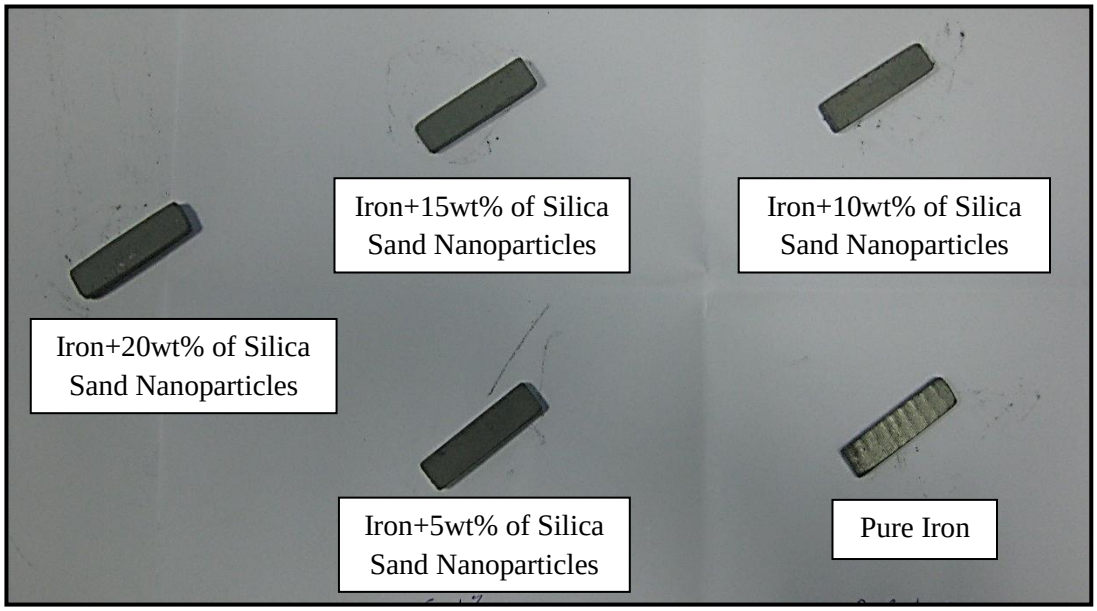
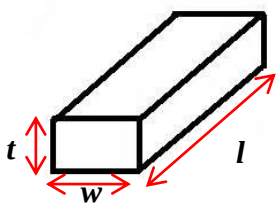


Figure 4.1 : Samples Produced by Auto Pallet Press Machine

Next, the dimensions of all samples were being measured. All measurements are represented in Table 4.1 below:

Table 4.1 : Samples Measurement



	Pure Iron	Iron+5wt% of Silica Sand Nanoparticles	Iron+5wt% of Silica Sand Nanoparticles	Iron+5wt% of Silica Sand Nanoparticles	Iron+5wt% of Silica Sand Nanoparticles
Length (mm)	35	35	35	35	35
Width (mm)	9	9	9	9	9
Thickness (mm)	3	3	3.5	3.5	4

4.2 Density Measurement

4.2.1 Theoretical Density of Silica Sand Nanoparticles-Iron Based Composites

Before performing the project, theoretical densities of a composite were determine as a reference for the results onwards by using the formula given below:

$\boldsymbol{\rho_c = 1 / (W_p/ \rho_p + W_m/ \rho_m)}$ where; ρ_c = *total density of composite*

W_p = *weight percentage of particulate*

W_m = *weight percentage of metal*

ρ_p = *density of particulate*

ρ_m = *density of metal*

The results for theoretical density of composites calculated are represented by the Table 4.2 below which shows the decreasing in density with the increasing in percentages of silica sand nanoparticles:

Table 4.2 : Theoretical Density of Silica Sand Nanoparticles-Iron Based Composites

Composition (total 5grams)	Theoretical Density (g/cm^3)
Pure Iron	7.874
Iron + 5%wt silica sand nanoparticles	7.168
Iron + 10%wt silica sand nanoparticles	6.577
Iron + 15%wt silica sand nanoparticles	6.077
Iron + 20%wt silica sand nanoparticles	5.647

4.2.2 Green and Sintered Density of Silica Sand Nanoparticles-Iron Based Composites

Green density values are represented by the Table 4.3 below and it shows that there were differences compared with theoretical density at about 75% of dense.

Table 4.3 : Green Density of Silica Sand Nanoparticles-Iron Based Composites

Composition (total 5grams)	Green Density (g/cm^3)
Pure Iron	5.643
Iron + 5%wt silica sand nanoparticles	5.125
Iron + 10%wt silica sand nanoparticles	4.878
Iron + 15%wt silica sand nanoparticles	4.423
Iron + 20%wt silica sand nanoparticles	4.264

After being sintered at 1100^0C in Nitrogen atmosphere for two hours, the sintered density were measured by using Mettler Toledo AX205 with respect to Archimedes' method. Table 4.4 below shows the comparison between green and sintered density.

Table 4.4 : Comparison of Green and Sintered Density of Silica Sand Nanoparticles-Iron Based Composites

Composition (total 5grams)	Green Density (g/cm^3)	Sintered Density (g/cm^3)
Pure Iron	5.643	5.840
Iron + 5%wt silica sand nanoparticles	5.125	5.202
Iron + 10%wt silica sand nanoparticles	4.878	4.928
Iron + 15%wt silica sand nanoparticles	4.423	4.617
Iron + 20%wt silica sand nanoparticles	4.264	4.366

From the Figure 4.2 below, it shows that both green and sintered densities reduced from pure iron to 20wt% of silica sand nanoparticles. However, there are increments in each density of silica sand nanoparticles-iron based composites after being sintered. The sintered density is slightly improved at the average of 1.6% compared to the green density. This is because, silica sand nanoparticles act to fill pores to reduce the porosity during the sintering process.

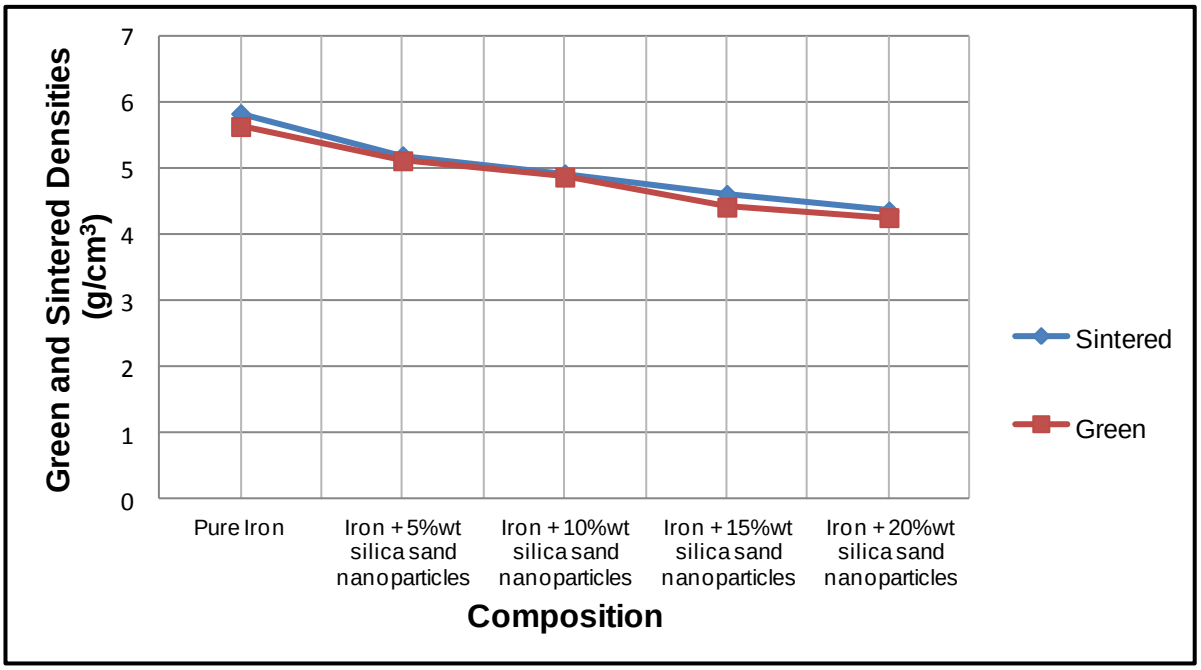


Figure 4.2 : Comparison of Green and Sintered Density of Silica Sand Nanoparticles-Iron Based Composites at 1100^0C

4.3 Microstructure Analysis

Before performing microstructure analysis, all samples had undergone metallographic preparation. The steps included cutting, mounting, grinding and polishing process. Figure 4.3 below shows the samples produced after metallographic preparation being done:

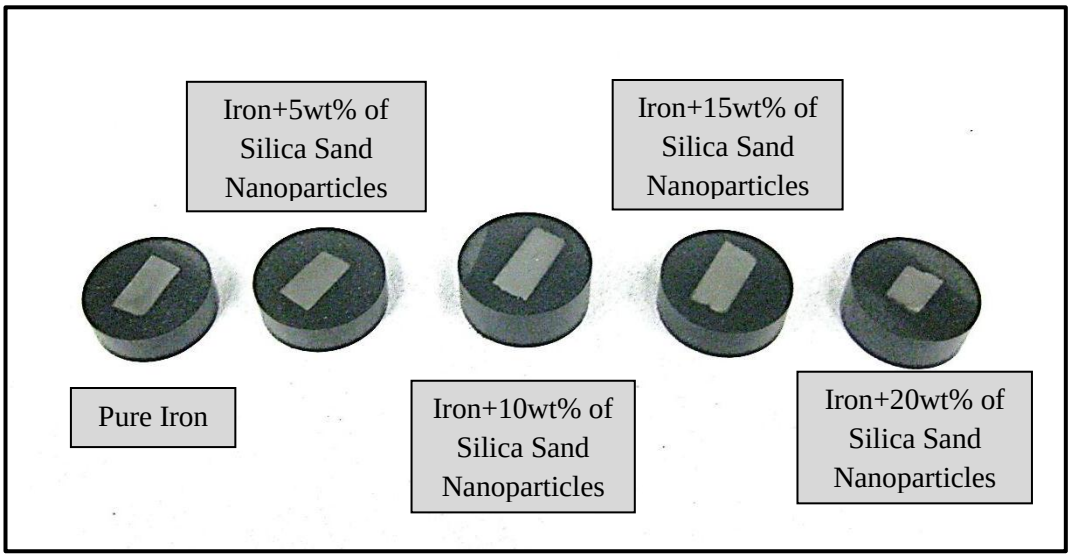
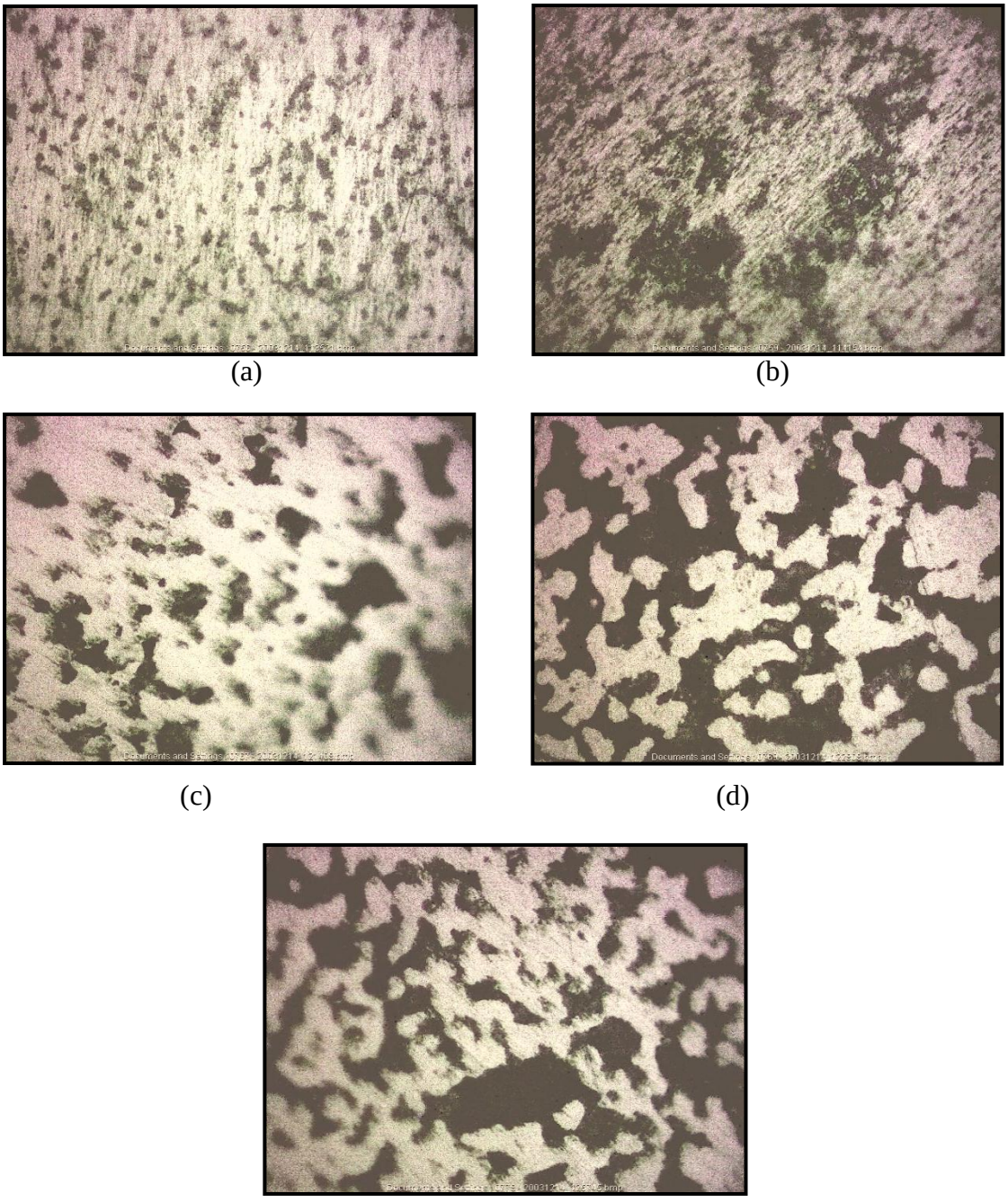


Figure 4.3 : Samples After Metallographic Preparation

4.3.1 Optical Microscope Analysis of Silica Sand Nanoparticles-Iron Based Composites

Figure 4.4 (a, b, c, and d) show the image of sintered Fe-SiO_2 nanoparticles composite at 1100^{o}C. It is clearly shown how silica sand nanoparticles are distributed in iron and occupied the porosity places after sintering. Increasing wt% trend of silica sand nanoparticles shows more pores are filled throughout Fe particle. It is clearly indicates sintering temperature effect the diffusion of SiO_2 into the Fe particles. During sintering of Fe-SiO_2 compacts, some SiO_2 particles decomposed into Si and O_2 atoms that could diffuse into Fe particles. The higher sintering temperature, more diffusion occurs.



(a) (b)

(c) (d)

(e)

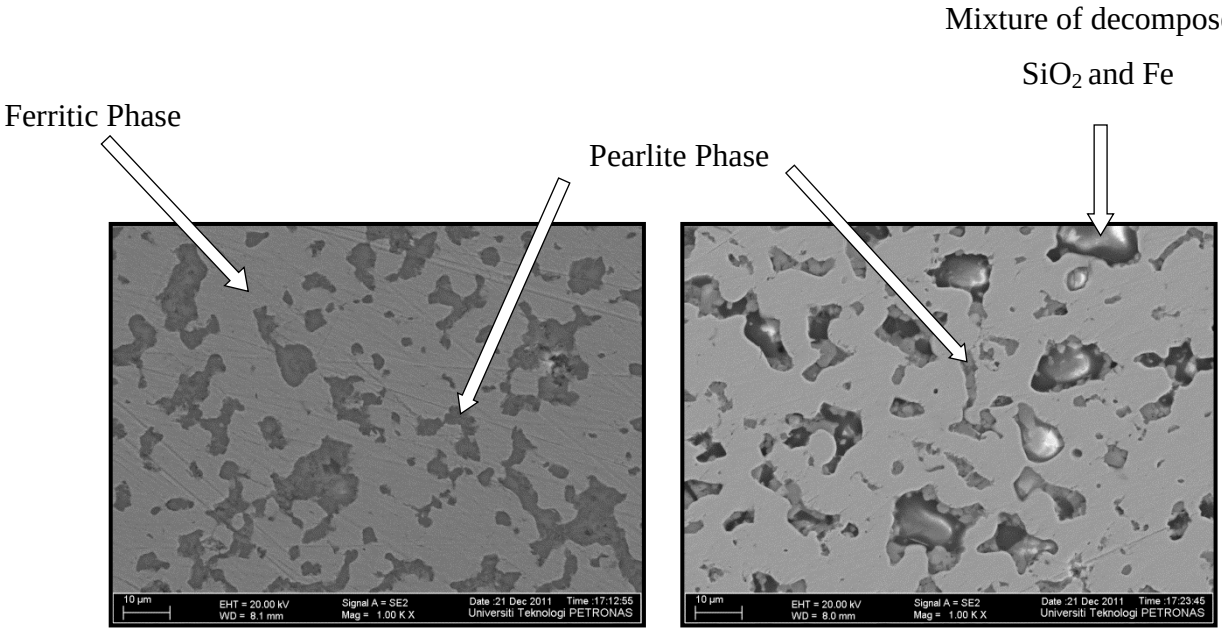
Figure 4.4 : Optical Microscope Image at 50x Resolution of Silica Sand Nanoparticles-Iron Based Composites at 1100^0C Sintering Temperature in Nitrogen Atmosphere ; (a) Pure Iron (b) 5wt% of SiO_2 (c) 10wt% of SiO_2 (d) 15wt% of SiO_2 (e) 20wt% of SiO_2

4.3.1 FESEM Analysis of Silica Sand Nanoparticles-Iron Based Composites

From figure 4.5 (a, b, c and d) shows the diffusion of silica sand nanoparticles in iron based composites. Same goes for the the ferritic and pealite iron phase which are also clearer and more visible. There a few important zones present form the FESEM images which are:

1. The light zones represent ferritic iron
2. The light grey zones represent lamellar structure (hair type structure) of pearlite phase
3. The dark zones represent voids surrounding the decomposed SiO_2 particles

Some of SiO_2 particles decompose into Si and O_2 atoms could diffuse into Fe particles which lead to the growth of voids during the sintering process of silica sand nanoparticles-iron based composites. A clearer and more homogeneous structure can be seen at the sintering temperature of 1100^0C due to better diffusion welding between silica sand nanoparticles and iron particles.



(a) (b)

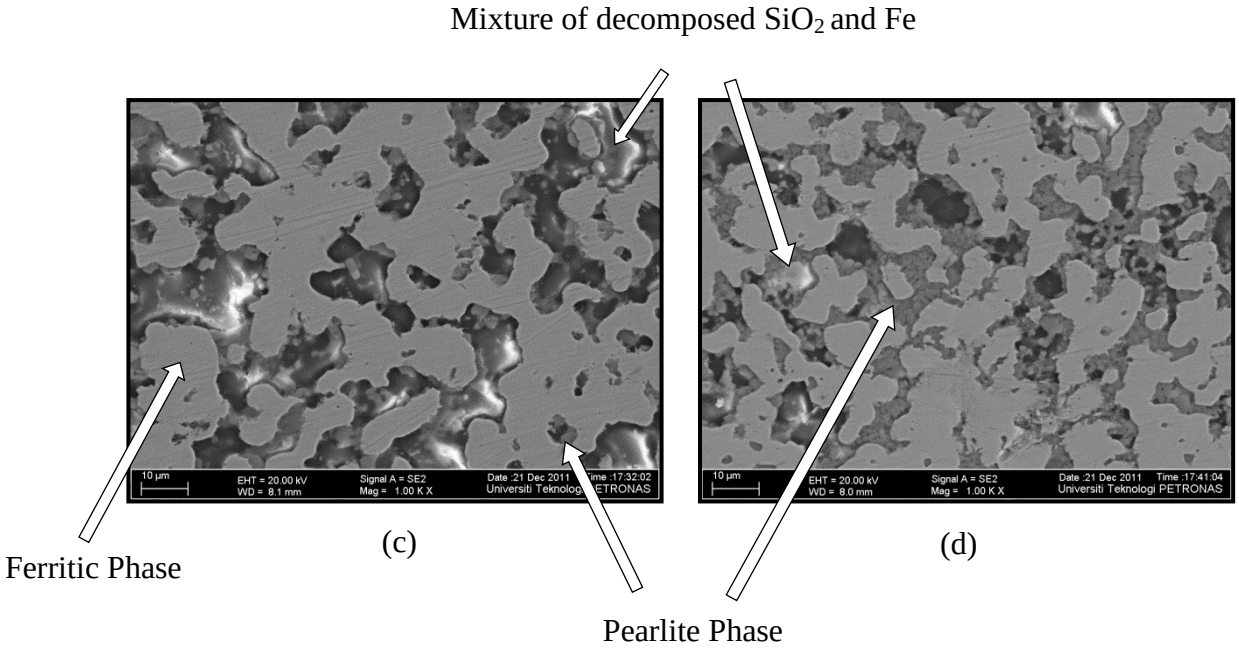


Figure 4.5 : FESEM Image at 1000x Resolution of Silica Sand Nanoparticles-Iron Based Composites at 1100^0C Sintering Temperature in Nitrogen Atmosphere ; (a) 5wt% of SiO_2 (b) 10wt% of SiO_2 (c) 15wt% of SiO_2 (d) 20wt% of SiO_2

4.4 Elemental Composition Analysis

4.4.1 EDX Analysis of Silica Sand Nanoparticles-Iron Based Composites

Figure 4.6 until 4.9 show the point and EDX analysis where the different points have been taken to verify the description of FESEM images. The light zones represent ferritic iron while the light grey zones represent lamellar structure (hair type structure) of pearlite phase containing a little bit silicon content and make the surface harder. The dark zones represent voids surrounding the decomposed SiO_2 particles. The decomposed Si from SiO_2 diffuses inside iron during sintering and forming a new compound FeSi. This phase or compound is a main causing of hardening of Iron based composites. These results have been here verified by EDX analysis which indicates elemental composition for each mixture.

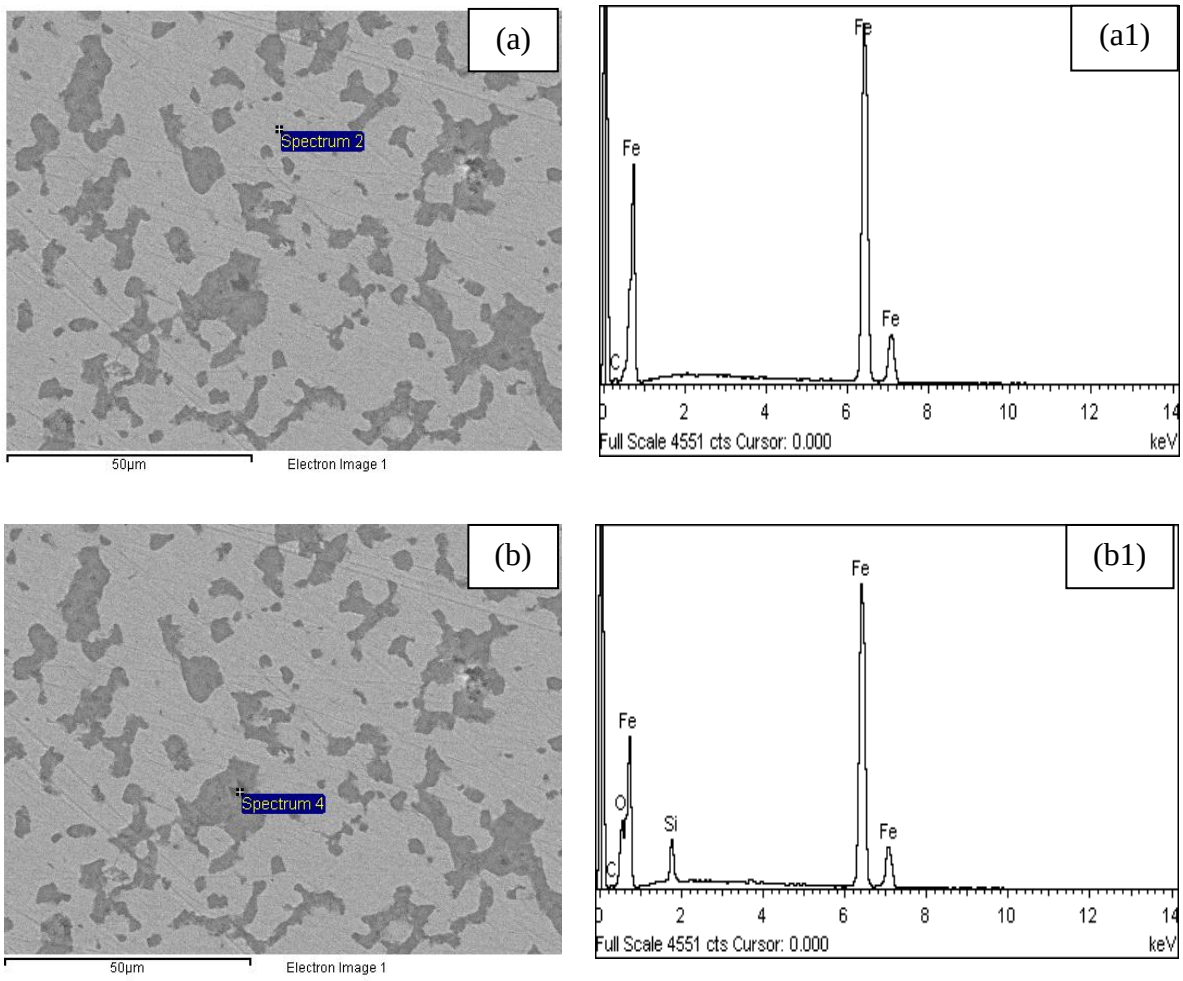


Figure 4.6 : EDX Analysis for 5wt% of Silica Sand Nanoparticles (a & b)

Table 4.5 below shows the elemental composition of 5wt% of silica sand reinforced into pure iron for Figure 4.6 (a & b)

Table 4.5 : Element Composition for 5wt% of Silica Sand Nanoparticles (a & b)

(a)

Element	Weight %	Atomic %
C K	5.22	20.40
Fe K	94.78	79.60
Totals	100.00	100.00

(b)

Element	Weight %	Atomic %
C K	3.79	12.19
O K	10.88	26.24
Si K	3.85	5.29
Fe K	81.47	56.28
Totals	100.00	100.00

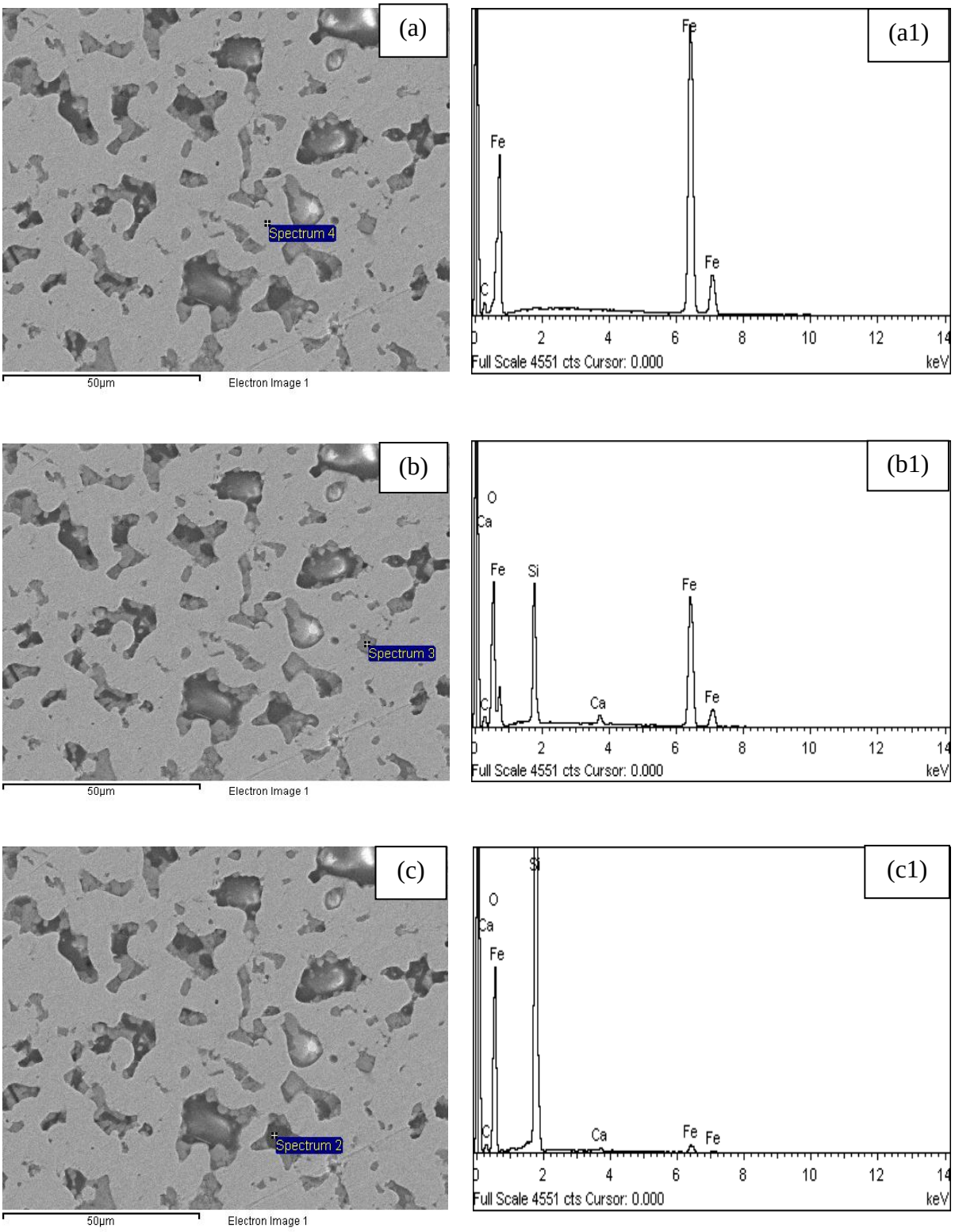


Figure 4.7 : EDX Analysis for 10wt% of Silica Sand Nanoparticles

Table 4.6 below shows the elemental composition of 10wt% of silica sand reinforced into pure iron for Figure 4.7 (a, b & c)

Table 4.6 : Element Composition for 10wt% of Silica Sand Nanoparticles (a, b & c)

(a)

Element	Weight %	Atomic %
C K	10.83	36.10
Fe K	89.17	63.90
Totals	100.00	100.00

(b)

Element	Weight %	Atomic %
C K	11.57	22.09
O K	35.94	51.53
Si K	11.53	9.41
Ca K	0.90	0.52
Fe K	40.06	16.45
Totals	100.00	100.00

(c)

Element	Weight %	Atomic %
C K	12.63	19.12
O K	51.15	58.13
Si K	33.96	21.99
Ca K	0.22	0.10
Fe K	2.04	0.66
Totals	100.00	100.00

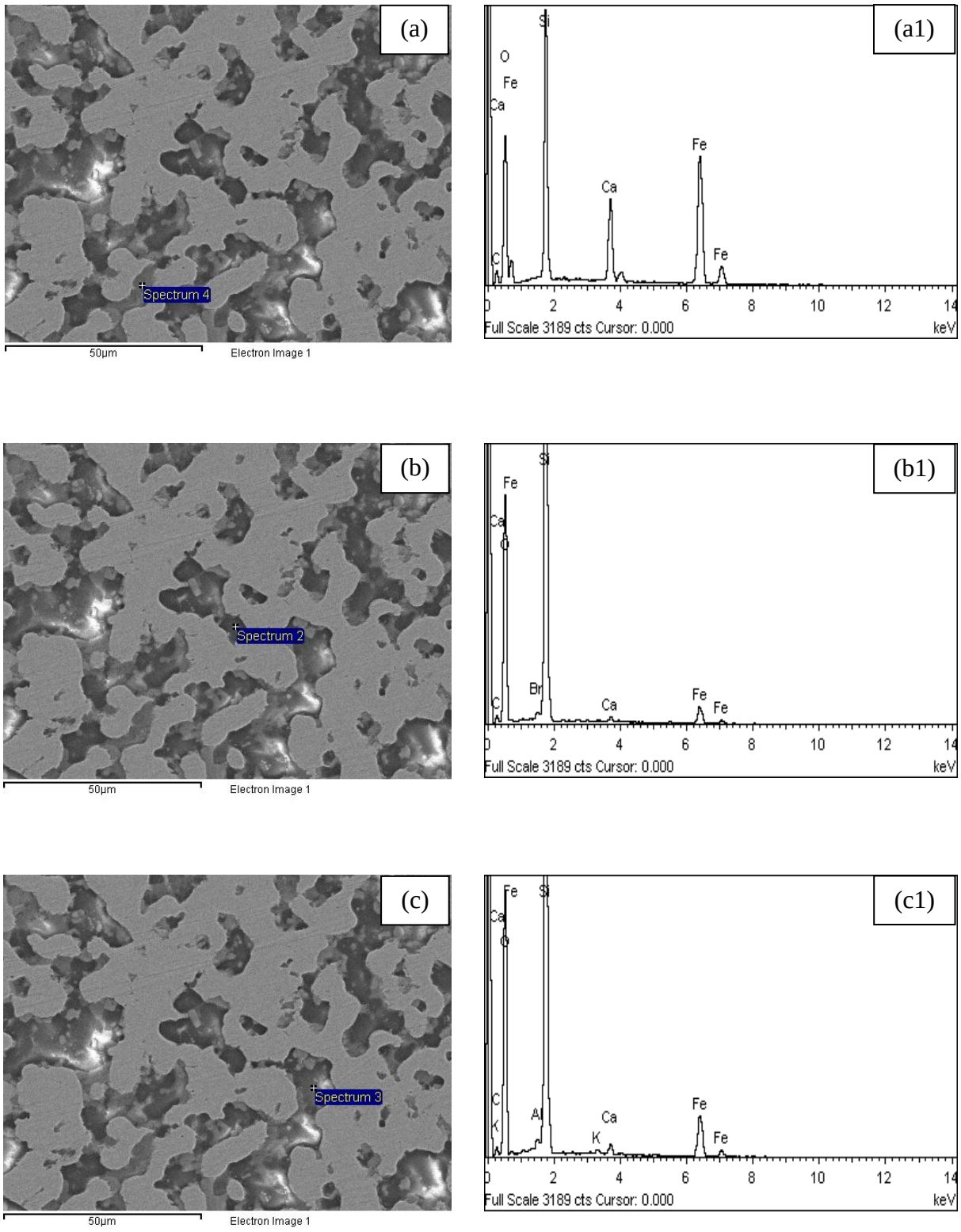


Figure 4.8 : EDX Analysis for 15wt% of Silica Sand Nanoparticles

Table 4.7 below shows the elemental composition of 15wt% of silica sand reinforced into pure iron for Figure 4.8 (a, b & c)

Table 4.7 : Element Composition for 15wt% of Silica Sand Nanoparticles (a, b & c)

(a)

Element	Weight %	Atomic %
C K	11.22	20.96
O K	36.06	50.56
Si K	15.68	12.52
Ca K	6.86	3.84
Fe K	30.18	12.12
Totals	100.00	100.00

(b)

Element	Weight %	Atomic %
C K	10.50	16.25
O K	52.30	60.75
Si K	32.29	21.36
Ca K	0.36	0.17
Fe K	4.03	1.34
Br L	0.52	0.12
Totals	100.00	100.00

(c)

Element	Weight %	Atomic %
C K	-1.37	-2.42
O K	52.43	69.51
Al K	0.48	0.37
Si K	37.13	28.04
K K	0.30	0.16
Ca K	0.89	0.47
Fe K	10.14	3.85
Totals	100.00	100.00

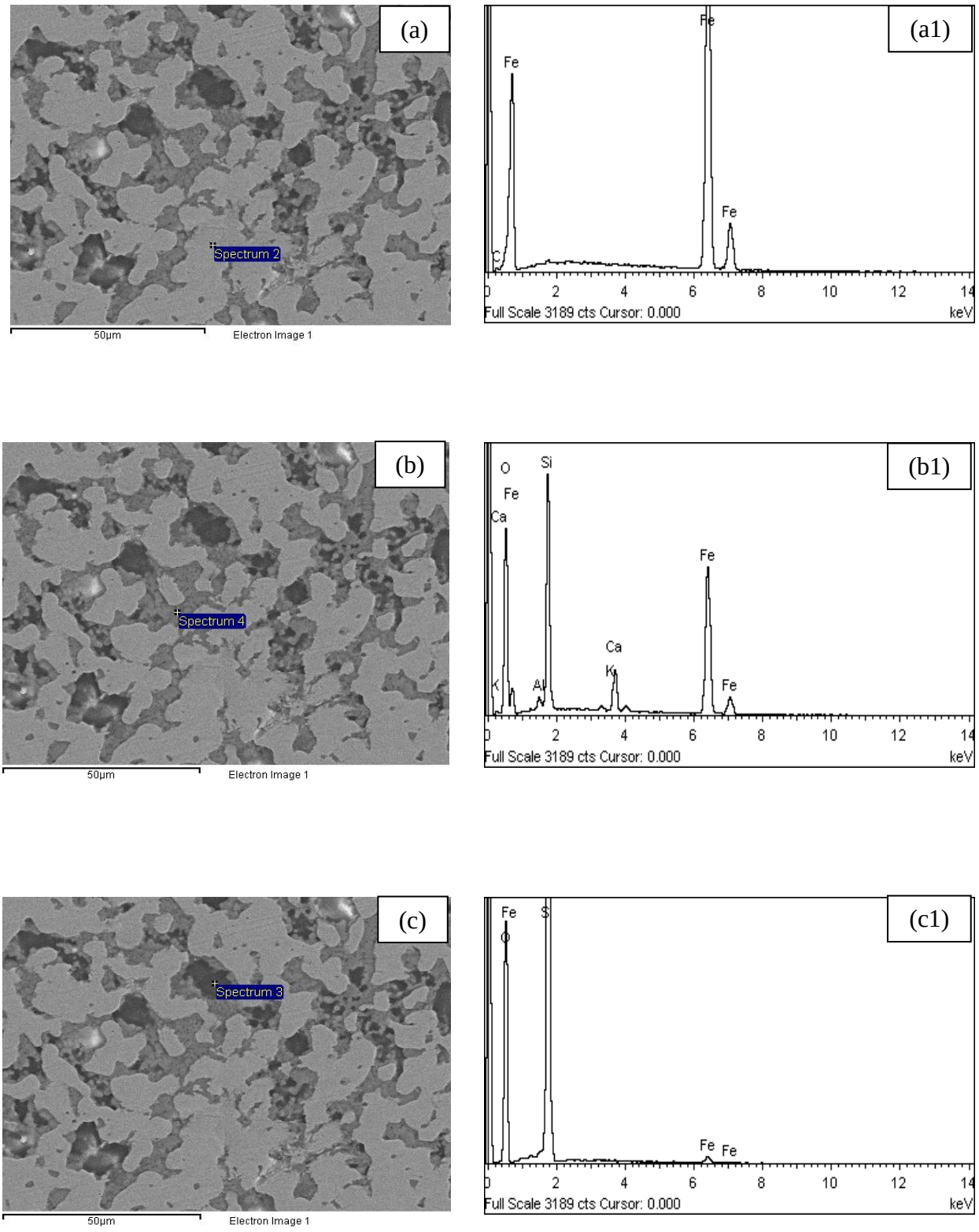


Figure 4.9 : EDX Analysis for 20wt% of Silica Sand Nanoparticles

Table 4.8 below shows the elemental composition of 20wt% of silica sand reinforced into pure iron for Figure 4.9 (a, b & c)

Table 4.8 : Element Composition for 20wt% of Silica Sand Nanoparticles (a, b & c)

(a)

Element	Weight %	Atomic %
C K	3.72	15.22
Fe K	96.28	84.78
Totals	100.00	100.00

(b)

Element	Weight %	Atomic %
O K	38.87	62.75
Al K	0.82	0.78
Si K	16.97	15.61
K K	0.37	0.24
Ca K	4.12	2.65
Fe K	38.85	17.96
Totals	100.00	100.00

(c)

Element	Weight %	Atomic %
O K	54.82	68.46
Si K	43.48	30.93
Fe K	1.70	0.61
Totals	100.00	100.00

4.5 Microhardness Analysis

Increasing in the weight percentages of silica sand nanoparticle reinforcement into pure iron has increased the hardness value of the composite as well. As shown in Table 4.5 below, the maximum hardness 152.1 HV was achieved in the composite with 20 wt% of silica sand nanoparticles. This may be due to the strengthening in solid solution by Si diffusion, hence forming a pearlite phase. Besides, good binding interface between materials can also give good mechanical properties of composite.

Silicon rich phase had more hardness as compared to iron rich phase. Sintering process had cause the diffusion of Si into Fe which created Fe-Si phase and lead to the increasing in hardness of the composites. Fe-Si. During microharness test, Si-rich phase do not allow indenter to make a deep indentation. However, it was different from Fe-rich phase which easily being indent on the surface. SiO_2 may dissociate into Si and O_2 at the temperature of 1100^0C.

In addition, sintering environment also can affect the hardness value of composites. For this research, nitrogen atmosphere is used as the sintering environment. Therefore, there is possibility of nitriding occur towards composites which can as well increase the hardness.

Table 4.5 : Microhardness Testing Result of Silica Sand Nanoparticles-Iron Based Composites at 1100^0C

Composition (total 5grams)	Hardness (HV)
Pure Iron	82.9
Iron + 5%wt silica sand nanoparticles	99.3
Iron + 10%wt silica sand nanoparticles	113.4
Iron + 15%wt silica sand nanoparticles	133.9
Iron + 20%wt silica sand nanoparticles	152.1

CHAPTER 5

CONCLUSION & RECOMMENDATION

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5.1 Conclusion

This main purpose of this research to study the effect silica sand nanoparticles reinforcement on the mechanical properties of iron based composites have been investigate. The reinforcement of silica sand nanoparticles into iron powder has given some significant impact on its properties.

Firstly, as the percentages of silica sand increased, the density will decrease which may be good in term of weight saving or higher strength-to-weight ratio. In the meantime, there was improvement in density after being sintered. This were due to changes exhibits in porosity, pore size also pore shape and with the high rate of atomic motion progressively (diffusion) leads to growth of bonds between particles in the microstructure. It was proven by FESEM AND EDX analysis which observed that the silica sand nanoparticles diffused in the porous sites of composites causing improvement in mechanical properties such as hardness as well as improved the microstructure.

In conclusion, sintering temperature and weight percentage of silica sand being reinforced into iron play a big role for the improvement in the Silica Sand Nanoparticles-Iron Based Composites properties. From the investigation, a composite with the highest weight percentage of silica sand nanoparticles (20%) reinforced offered the best characteristic and properties.

5.2 Recommendation

There were some findings and challenges faced throughout conducting the research project Therefore, some recommendations can be implemented so that there will be improvement for further study.

In order to investigate more mechanical properties, samples can be produced in dog-bone shape. Therefore, tensile and flexural testing can conducted to obtain the tensile and flexural properties of the Silica Sand Nanoparticles-Iron Based Composites.

Silica Sand Nanoparticles-Iron Based Composites can also be sintered at higher temperature in other suitable environment to compare the distribution of silica sand nanoparticles into pure iron.

If possible, the mixing and compaction step have to be done in vacuum space since pure iron can easily be oxidized in normal atmosphere. Besides, the variety of speed during the mixing can also be studied to see the homogeneities of mixture.

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APPENDICES

Appendix I

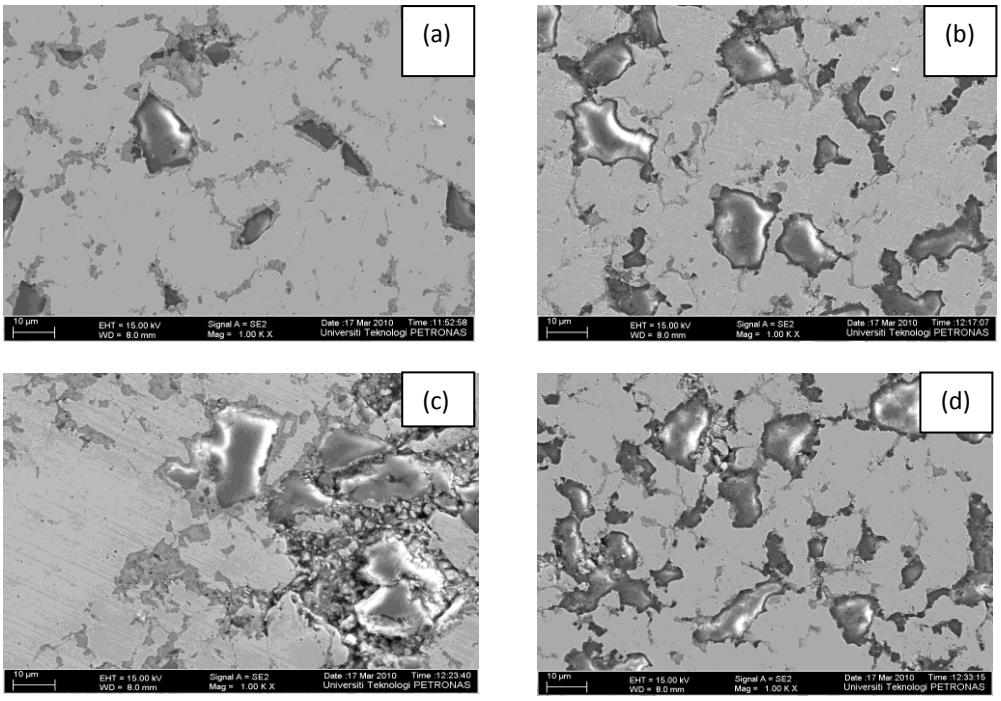

Figure 1 : FESEM analysis of Fe-SiO_2 nanoparticles composites with; (a) 5wt% of SiO_2 (b) 10wt% of SiO_2 (c) 15wt% of SiO_2 (d) 20wt% of SiO_2 at 1100^0C sintering temperature in Nitrogen atmosphere [Tahir, 2010]

Table 1 : Density of Fe-SiO_2 nanoparticles composites at different sintering temperature [Tahir, 2010]

Material	Sintered @ 900 ^{o}C	Sintered @ 1000 ^{o}C	Sintered @ 1100 ^{o}C
Fe (99%)	6.638	6.782	7.347
Fe + 5% Nanoparticles	5.408	5.427	5.509
Fe + 10% Nanoparticles	4.768	4.838	5.109
Fe + 15% Nanoparticles	4.509	4.608	4.717
Fe + 20% Nanoparticles	4.256	4.349	5.109

Table 2 : Hardness value of Fe-SiO_2 nanoparticles composites at different sintering temperature [Tahir, 2010]

Sintering temperature/Hardness	900 ^oC/HV	1000 ^oC/HV	1100 ^oC/HV
Pure Iron	51.7	57.6	82.7
5%	61.3	71.2	84.9
10%	77.5	83.7	120
15%	90.3	87.9	137.9
20%	110.1	112.1	167.4

Appendix II

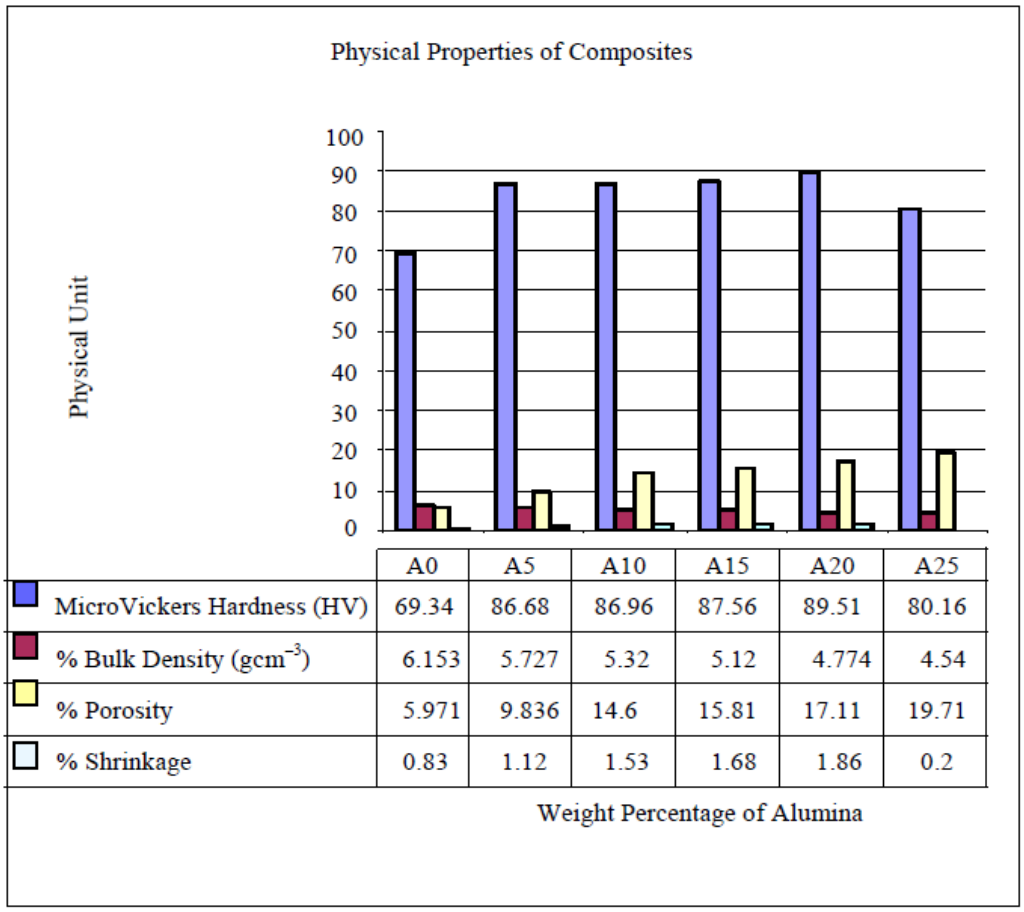

Figure 2 : Experimental results of Fe-Cr-Al_2O_3 composites properties [Saidatulakmar, 2008]

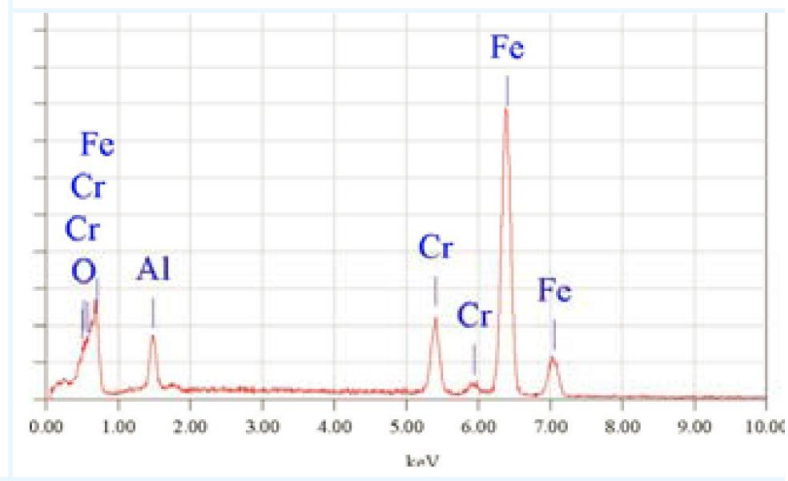

Figure 3 : EDX results of Fe-Cr-Al_2O_3 composites characterization [Saidatulakmar, 2008]

Appendix III

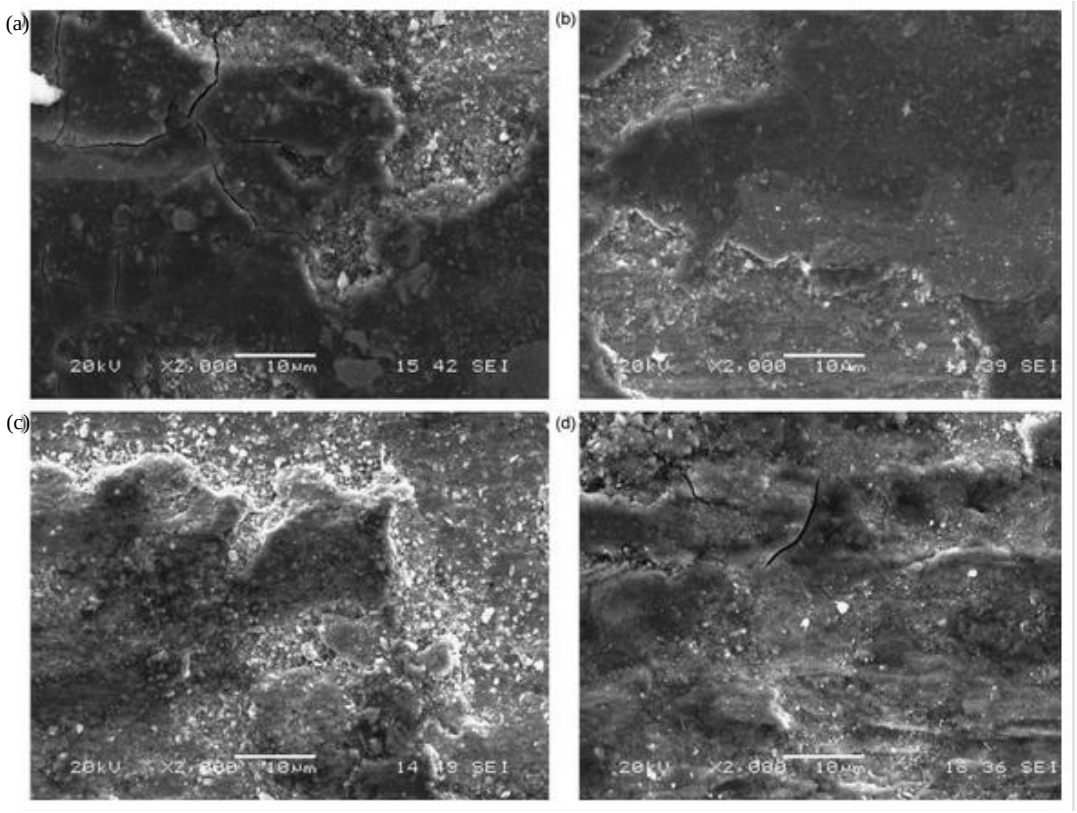

Figure 4 : SEM morphology of the wear tracks of composite coatings prepared with different current densities: (a) 1 A/dm^2(vol. 3.98% Al_2O_3), (b) 3 A/dm^2 (vol. 8.49% Al_2O_3), (c) 6 A/dm^2 (vol. 8.46% Al_2O_3) and (d) 9 A/dm^2 (vol. 8.81% Al_2O_3) [Gul, 2008]

Appendix IV

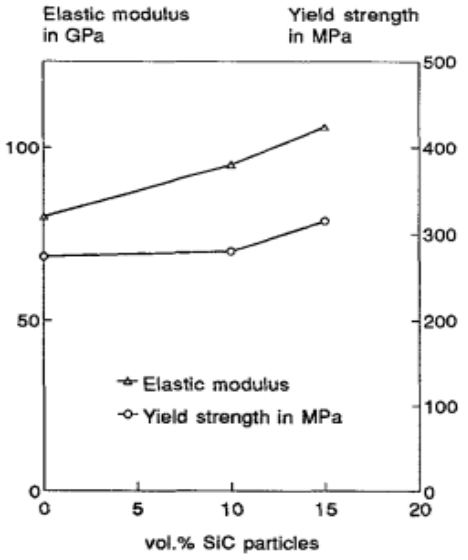

Figure 5 : Effect of particle content on strength of F800 SiC particle reinforced Al-6wt% Fe powder extrusions [Staniek, 1993]

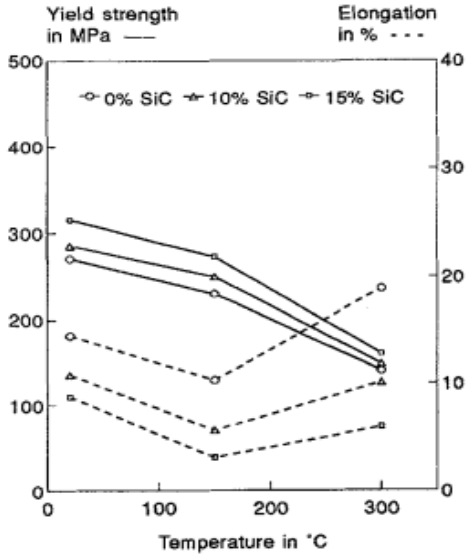

Figure 6 : Temperature effects of tensile properties of F800 SiC particle reinforced Al-6wt% Fe powder extrusions [Staniek, 1993]

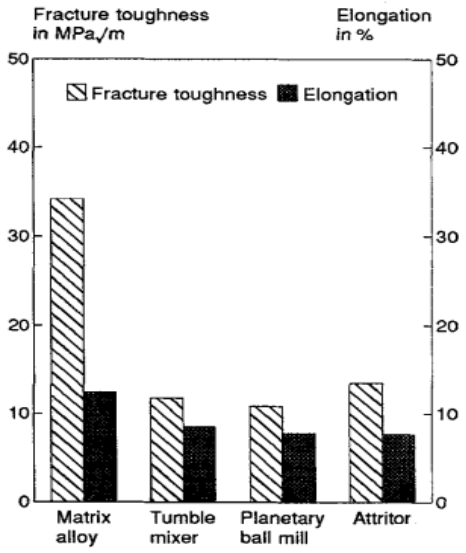

Figure 7 : Effect of mixing techniques on fracture toughness and elongation of Al-6wt% Fe powder extrusions reinforced with 10vol% SiC particles [Staniek, 1993]

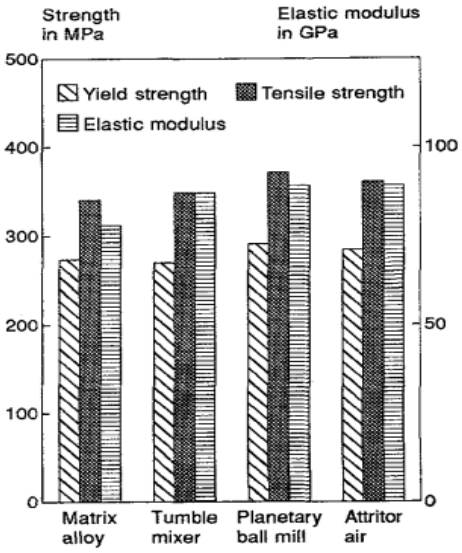

Figure 8 : Effect of particle content on strength of F800 SiC particle reinforced Al-6wt% Fe powder extrusions [Staniek, 1993]

Appendix V

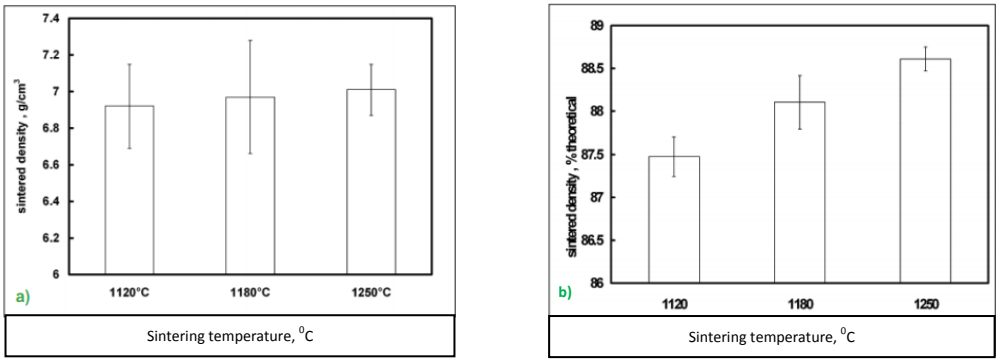

Figure 9 : Effect of sintering temperature on densification [Anand, 2006]

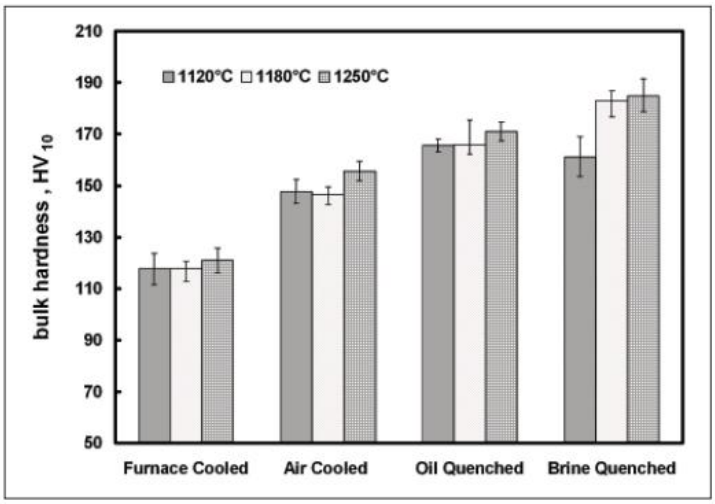

Figure 10 : Bulk hardness of SH737-2Cu-0.9C samples sintered at different temperatures and cooled by different methods [Anand, 2006]