Study on wear resistance and microstructure of nanosilica-iron based composites.

by

Abu Yazid Bin Zainuddin

Dissertation submitted in partial fulfillment of
the requirements for the
Bachelor of Engineering (Hons)
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Universiti Teknologi PETRONAS
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CERTIFICATION OF APPROVAL

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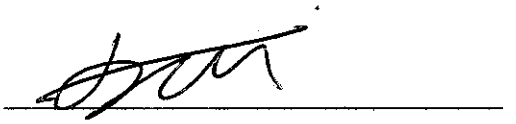
Approved by,

(AP Dr. Othman Bin Mamat)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
MAY 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.



ABU YAZID BIN ZAINUDDIN

ABSTRACT

When a machine is in operation, two moving surfaces interact to generate a large amount of wear particle. Abrasive wear of engineering machine components caused by the abrasive particle is the major industrial problem. In materials science, **wear** is the erosion of material from a solid surface by the action of another surface. It is related to surface interactions and more specifically the removal of material from a surface as a result of mechanical action. Wear caused by the presence of abrasive particles is influenced by their size, concentration, shape, hardness, and sliding velocities.

The objective of this report is to study on wear resistance of Nanosilica-iron based composites. The studies of this project are aim to research about the differential percentage of nanosilica addition to the pure iron with different sintered temperatures and focus on physical and mechanical properties of nanosilica-iron based composite, several tests will be conducted to the samples, which include density measurement, hardness test and wear resistance.

Currently, iron based silica sand nanoparticles composites 5, 10, 15 and 20 wt. % of nanoparticles silica and were developed through powder metallurgy technique and sintered at 900c, 1000c and 1100c. So to determined which composition are better regarding to the sintered temperature value will be test by using hardness test and wear resistance test.

The results show that the addition of silica sand nanoparticles to iron enhanced the hardness and wear resistance with increasing the sintered temperature and silica sandnanoparticles. An improvement in sintered densities was also observed with increasing trend of sintering temperatures. An optimum value of 20wt.% of silica sand nanoparticles in iron based composites was found to have best micro hardness values and wear resistance for all sintering temperatures.

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INTRODUCTION

1.1 Project Background

1.1.1 *Powder Metallurgy*

Powder technology is the science for the manufacture of parts from metal powders by compaction and heating that creates a homogeneous mass. Heating is executed in a furnace and is called sintering. The temperature at which sintering is performed is lower than the melting point of the powdered material. This is the basic principle of powder technology.

The processes involved in powder metallurgy are the following:

Blending and Mixing: This is carried out to achieve uniformity of the product manufactured. Distribution of properly sized particles is attained by mixing elementary powder with alloy powders to obtain a homogeneous mixture.

Pressing: The cavity of the die is filled with a specified quantity of blended powder, necessary pressure is applied, and then the compacted part is ejected. Pressing is performed at room temperature, while the pressure is dependent upon the material, properties of the powder used, and the density required of the compaction

Sintering: Changes occur during sintering, including changes in size, configuration, and the nature of pores. Sintering operation ensures that powder particles are bonded strongly and that better alloying is achieved.

1.1.2 *Wear Resistance*

In materials science, **wear** is the erosion of material from a solid surface by the action of another surface. It is related to surface interactions and more specifically the removal of material from a surface as a result of mechanical action. The need for mechanical action, in the form of contact due to relative motion, is an important distinction between mechanical wear and other processes with similar outcomes.

The definition of wear does not include loss of dimension from plastic deformation, although wear has occurred despite no material removal, because it may lack the action of another surface. This definition also fails to include impact wear, where there is no sliding motion, cavitations, where the counter body is a fluid, and corrosion, where the damage is due to chemical rather than mechanical action.

1.1.3 *Nanosilica-Iron Based Composite*

Metal and ceramics composites have been attracting researchers' interest in recent years, since it can provide many advantageous characteristic. Metal and ceramics composites are extremely used as materials for product which required high thermal and high wear resistance and hardness. In this experiment study the following composites were developed with 5%, 10%, 15% and 20% silica sand nanoparticles were prepare by using powder metallurgy method. Microstructure analysis, hardness and wear resistance were evaluated and discussed.

1.2 Problem Statement

Ceramics possess much higher specific strength and stiffness, improved high temperature properties, higher wear resistances and lower thermal expansion coefficients of metal. The study of wear resistance, particularly for Nanosilica-Iron Based Composite is not well established although it is necessary to understand the mechanism itself for further investigations and improvements in the synthesis of material industry.

1.3 Objective

Objective of this project is to determine the wear resistance of nanosilica-iron based composites.

1.4 Scope of Study

The studies of this project are aim to research about the differential percentage of nanosilica with different sintered temperatures and focus on physical and mechanical properties of the composite, several tests will be conducted to the samples, which include density measurement, hardness test and wear resistance test.

The scope of studies of this project includes:

Material Preparation

- Grinding
- Compaction

Material studies

- Density
- Microstructure

Physical and mechanical properties test

- Hardness
- Wear resistance

2.0 LITERATURE REVIEW

The effect of the hardness of particles on abrasive wear has received considerable interest as well. The influence of hard silica or quartz particles on wear process has been investigated by many researchers. The results have showed that the small titania particles have been found to reduce wear in hybrid rolling bearing and increase the polishing wear in all-steel bearing [1].Increase in friction is not proportional to the concentration but proportional to the amount of the actual interfering particles [1].In a two body abrasive system ,the wear rates increase with increase of particles size up to a critical size (around 100 um), and above this value , the particles size effect in wear rates become almost negligible [1].

Wear results in severe economics loss and it is estimated that the cost of abrasion is about 1-4%. Composite materials with steel matrix and ceramic particle reinforcements provide a scope of producing relatively inexpensive wear resistance material. Wear can be generally described as the removal of the material from a surface in relation motion by mechanical and or chemical process. The tribological behavior of the composites depended on the micro structural properties of the material and type of loading-contact situation (tribo-system) [5]. The sintering process of the powder metallurgy is mainly controlled by solid diffusion and the compaction load. Generally the compaction increase with increasing reinforcement volume fraction [3]. The compressive resistances increase with the increasing of the Al_2O_3 weight fraction and therefore the porosity of 15 wt % Al_2O_3 composites is the highest. A uniform distribution of reinforcement could be possible only when reinforcement size is not less than a critical value, which is the function of reinforcement size and volume fraction and reduction ratio of secondary process.

Though much less research work has been carried out on Cu based alloy composite materials, the milling time of the power has a remarkable influence on the internal oxidation of Al_2O_3 - Cu composites [3]. Aluminum based composites were produced with highly refined structure, strengthened by oxide and Carbide dispersion, homogeneous distribution reinforcement, full densification and better mechanical

properties [5]. The wear behavior of Cu alloy-Sic composite transforms from mild to severe wear, and then to seizure [3].

The properties of metal matrix particulate composites depend mainly upon the microstructure and properties of matrix materials, nature of particles, the distribution, size and shape of particles and the interfacial behavior between particles and matrix. The most developments in the of iron-based powder metallurgy based on principle of obtaining wear-resistance material by the creation of a psedoalloy with a clearly defined heterogeneous structure, in which the micro hardness of the base material and hard phrase are substansially different [4]. Had fabricated and characterized composites of iron –chromium reinforced with 5-25 wt. % of alumina particles using powder metallurgy method and investigated the XRD analysis as well as mechanical properties [4]. Prepared iron base powder metallurgy composites by using SiC and C as reinforcement and investigated that the tensile strength and hardness of the sintered Fe-C composites were inferior to those of the sintered Fe materials [4]. The reason for property differences in the sintered Fe-Carbide were Carbide decomposition and reaction between Fe and Carbide constituents. The green density, sintered density and hardness of the composites were measured. The polish samples were analyses by using FESEM and EDS analysis. Below the examples of journal that became references.

Table 1: Summary of the journal

No	Author(s) + Title	Findings	Remarks, comments
1	**The characterization of wear transitions in sliding wear process contaminated with silica and iron powder.** (C.Q.Yuan, Z.Peng, X.C.Zhou, X.P.Yan) *School of Engineering, James Cook University, Townsville, Old 4811, Australia* *Reliability Engineering Institute, Wuhan University of Technology, Wuhan 430063, PR CHINA*	**Materials** - N32 lubricant -Iron powder -Silica powder **Methods** -Pin-on disc test **Results** - Silica particles decreased after thus particles got involved in the wear process -The strong cutting effect of the silica powder was weakened by the presence of the iron powder -Iron powder effectively reduce the friction between the wear surfaces -by increase the experiment time, the silica was broken by wear effect to reproduce new sharp edges.	-In this experiment using pin on disc machine to measure wear resistance and it's sample are ball shape -It is different from project sample there is in pallet form

2	**Synthesis and characterization of zirconium carbide-reinforced iron-based composite.** (Karabi Das*, T.K. Bandyopadhyay) *Department of Metallurgical and Material Engineering, Indian Institute of Technology, Kharagpur 721 302, India*	**Materials** - Zircon sand -Blue dust -Aluminium powder **Methods** -Charge preparation -Microscopy, image analysis, and X-ray diffraction (XRD) study -Abrasive wear test **Results** - All the aluminium does not take part in the reduction reaction, some aluminium remains in solution with iron. -Keeping the amount of carbon in the charge material constant as the amount of aluminium in the charge composition decreases, the recovery of Zr decreases. -The hardness of the matrix is significantly higher than that of the plain carbon steel matrix, is due to solid solution strengthening of iron by aluminium and silicon.	-With this journal the author noticed that, to study about how silica sand are distributed in iron, are using XRD and SEM microscopy. -It's sample are 12mm square cross section and 10mm thickness, it's size are almost same with pallet size -Abrasive test using linear abraser are suitable method for wear resistance test using pallet.
3	**Development of copper: alumina metal matrix composite by powder metallurgy method.**	**Materials** - Copper powder -Alumina powder	-Higher hardness of the particle, better is the wear resistance

	R. Thirraviam* *Department of Mechanical Engineering,* *Sethu Institute of Technology* T. Sornakumar *Department of Mechanical Engineering,* *Thiagarajar College of Engineering* A.Senthil Kumar *Deparment of Production Egineering,* *Sethu Institute of Technology*	**Methods** -XRD analysis and microstructure -Hardness and wear testing **Results** -The sintering process mainly controlled by solid diffusion and the compaction load, the porosity deccrease with increasing reinforcement volume fraction. -The high energy ball milling process reduces the reinforcement size, and tends to eliminate reinforcement clustering as well as reinforcement. -A higher constraint to the localised matrix deformation during indentation as a result of the presence of Al_2O_3	-Measure the sample hardness first before wear resistance - Measure the wear resistance by using weight loss, and will convert the weight loss to volume lost.
4	**Characerization of iron-silica sand nanoparticles composites with different Sintering Tempearature** Tahir Ahmed, Othman Mamat. 2010, "*Characterization and Properties of Iron-Silica Sand Nanoparticles Composites*", Universiti Teknologi PETRONAS, Malaysia.	**Materials** - Iron powder -Silica Sand nanoparticles powder **Methods** -XRD analysis and microstructure -Hardness and wear testing -Powder metallurgy	-The author notice that, repare 5 sample from powder metallurgy method there are pure iron, Nano-silica mixed iron 5%,10%,15%,20% -Hardness measurements were made using micro Vickers hardness tester.

		Results -An increasing trend of hardness was observed with increasing trend of silica sand nano particles in iron as well as as increasing the sintered temperature. -Improved hardness may be resulted from solid solution strengthening of by Si atoms and formation of pearlite phase. - -From FESEM and EDS analysis it is observed that the silica sand nanoparticles diffuse in the porous sites of composites causing an improvement in mechanical properties as well as improved the microstructure	

3.0 METHODOLOGY

3.1 Materials

Nanosilica-Iron Based Composite sample are required to implement the research. The Iron powder (99.5%) with size 10um commercially available was used. For nanosilica we got from silica sand originated from Tronoh, Perak, Malaysia and grounded to nanoparticles by using ball mill with zirconium ball as grinding media.

Iron Powder

- Size: (10μm) commercially available was used
- Purity: 99.5%

Nano-Silica Sand

- Origin/Company: Tronoh (local source)
- Size: Average of 77.82nm (Intensity produced by Zetasizer Nano Analyzer)
- Purity: 95%

3.2Tools and Equipment

The following are major tools and equipments that will be utilized in the laboratory experiment for the research:

Equipments and tools use for producing nanosilica-iron composites

- US Stoneware Ball Milling Machine (for grinding and mixing materials)
- Carver Autopellet Press Machine, USA (for compaction samples)
- Metallic mould
- Sintering furnaces
- Ceramics balls

Equipments and tools use for study the microstructures of samples

- Scanning Electron Microscopic (SEM)

Equipments and tools use for study physical and mechanical properties

- Micro Vickers hardness tester.
- Mettler Toledo AX205 density measurement instrument
- DUCOM Multi Specimen Tester

3.3 Specific project flows

3.3.1 *Preparation samples with mixing of iron powder and Nano-Silica Sand*

The iron powder (99.5%) with size 10um commercially available was used mixed with silica sand nanoparticles (95%) average (78.82 nm) originated from Tronoh, Perak were ball mill for one hour. Then, 30 ceramic balls will be put inside the bottle with the iron powder and with silica sand nanoparticles mixture. The iron power and silica sand nanoparticles will be milling for one hour. The preparation of the iron powder and with silica sand nanoparticles mixture varies with the percentage of silica sand nanoparticles which are:

- 5% silica sand nano-particles
- 10% silica sand nano-particles
- 15% silica sand nano-particles
- 20% silica sand nano-particles

Every different percentage of silica sand nano-particles will be produce with three samples.

3.3.2 *Compaction the samples*

The autopalletiser 69MPa force by using a metallic mould of diameter of 13mm was use to make the compacted pellets. The following composites were developed: 5% silica nanoparticles, 10% silica nanoparticles, 15% silica nanoparticles, 20% silica nanoparticles. The compactions of samples to be produced are:

- 3 samples of pure iron with 5% silica nano-particles
- 3 samples of pure iron with 10% silica nano-particles
- 3 samples of pure iron with 15% silica nano-particles
- 3 samples of pure iron with 20% silica nano-particles

3.3.3 *Green Density Measurement*

All the samples will be measure their green density using Mettler Toledo AX205 Density Measurement, using Archimedes' Method. The green density are important because to compare it with sintered density.

3.3.4 *Sintering the all sample Composites*

The green compacts were sintered at 900 C, 1000 C, and 1100C for two hour in argon atmosphere. The heating and cooling rates of sintering process were 5C/min and 10C/min respectively.

3.3.5 *Sintered Density Measurement*

Both green and sintered densities of relevant samples were measured by using Mettler Toledo AX205 density measurement instrument following the Archimedes method and compare to green density to know the condition composition of composite. All samples were analyses by using FESEM and EDS to study about it microstructure.

3.3.6 *Hardness and Wear Testing*

The Vickers Hardness Value (HV) was determined using micro hardness tester. Wear resistance is determines by using DUCOM Multi Specimen Tester. It was performed according to ASTM Standard G99 (Eyre, 1991). This type of apparatus offers far better control of experimental conditions and become increasingly used in preference to other tribometers

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3.4 Project Planning using flowchart

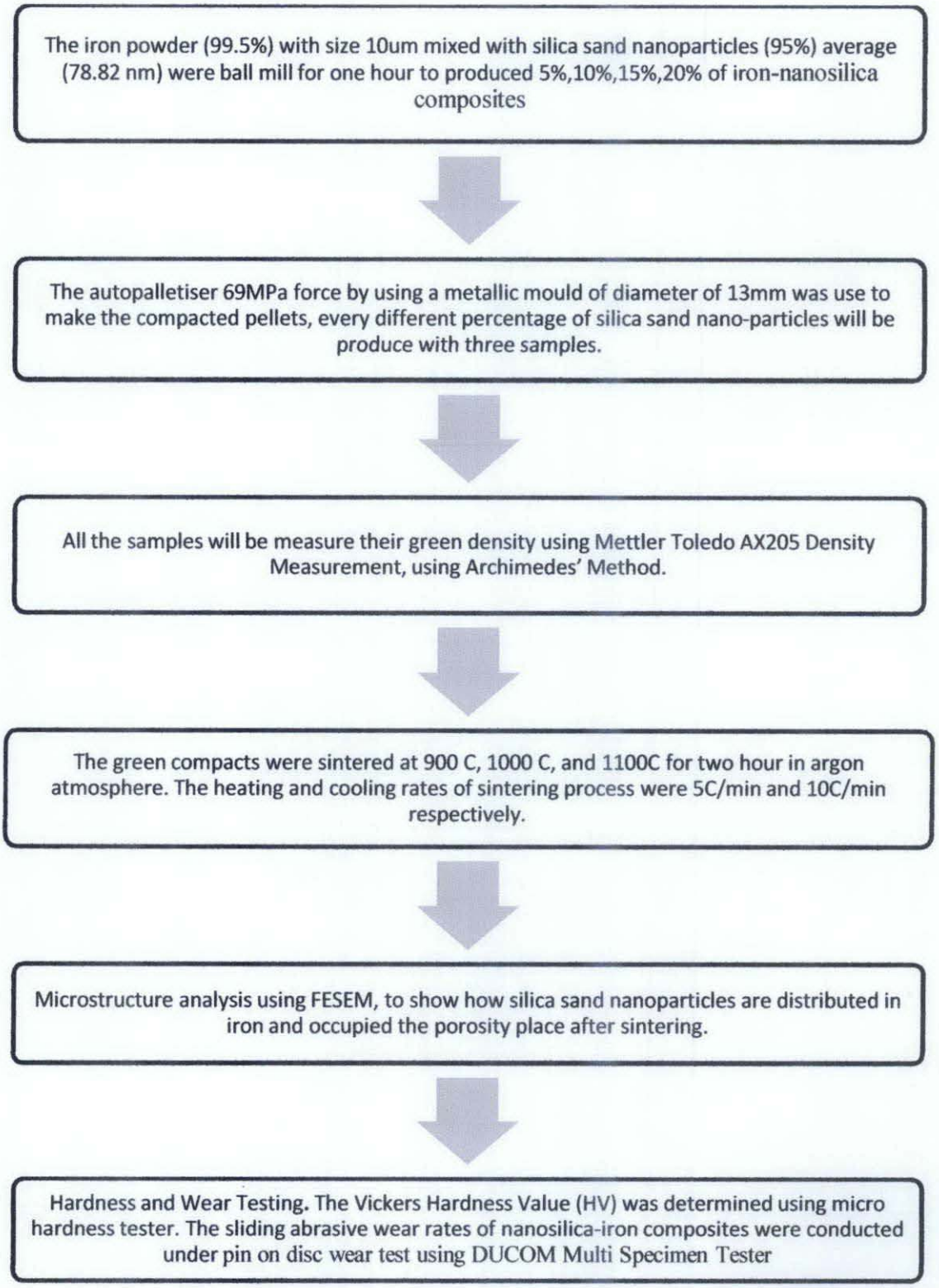

Figure 1: Project Flow Chart

3.5 Gantt Chart and Mile Stone

Table 2: Project Planning for July 2010 Semester

No	Detail/Week	1	2	3	4	5	6		7	8	9	10	11	12	13	14
1	**Selection of Project topic**															
2	**Preliminary research work**															
	• Literature Review															
	• Preliminary report preparation															
	• Preliminary report submission				▲											
3	**Start the project work**															
	• Ball mill nanosilica-iron mixture one hour															
	• Compact the mixture															
4	**Progress report and seminar**															
	• Data gathering analysis															
	• Preparation of progress report															
	• Progress report submission									▲						
	• Seminar									▲						
5	**Project work continuation**															
	• Take the green density of composites using Mettler Toledo AX205 Density Measurement															
	• Sintering furnace booking															
	• Sintering samples															
	• Take sintered density of composites using Mettler Toledo AX205 Density Measurement															
6	**Interim report**															
	• Data gathering analysis															
	• Submission of interim report															▲
7	**Final presentation**															

Mid-semester break

Study week

Process

▲ Key Milestone

Table 3: Project Planning for May 2011 Semester

No	Detail/Week	1	2	3	4	5	6		7	8	9	10	11	12	13	14
1	**Continuation project work**															
	• Literature riview															
	• Ball mill nanosilica-iron mixture one hour															
	• Compact the mixture															
	• Take the green density of composites using Mettler Toledo AX205 Density Measurement															
	• Sintering samples															
	• Designing and manufacture holder for pin on disc wear test.															
3	**Continuation project work**															
	• Study microstructure using FESEM machines															
	• Measurement of hardness of composites using Microvicker Hardness Tester machine															
	• Wear resistance test of the composites using pin on disc method															
4	**Progress Report**															
	• Data gathering and analysis															
	• Submission of progress report 2															
5	**Project Work Continues**															
	Pre-EDX															
6	**Pre-EDX**															
	• Poster, dissertation report, oral presentation and hard bound DR submission															
	• Submission of draft report															
	• Submission of dissertation report (soft bound)															
	• Submission of technical report															
	• Oral presentation	Study week														
	• Hard bound dissertation report submission	after oral presentation														

Mid-semester break

Process

Key Milestone

4. PROJECT ACTIVITIES

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4.1 Preparation of The Samples

Using the ratio wt%, 2 grams of the mixture of iron and silica nanoparticles has been made. The weight for the ratio is measured by using the Mettler Toledo AX205 density measurement instrument.

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Table 4: Composition weight of iron and silica sand nanoparticles.

Composition (total 2grams)	Iron powder (g)	Silica and nanoparticles (g)
Iron + 5%wt silica sand nanoparticles	1.9000g	0.1000g
Iron + 10%wt silica sand nanoparticles	1.8000g	0.2000g
Iron + 15%wt silica sand nanoparticles	1.7.000g	0.3000g
Iron + 20%wt silica sand nanoparticles	1.6000g	0.4000g

Figure 2 : Mettler Toledo AX205 density measurement instrument

Figure 3 : Silica sand nanoparticles

4.2 Mixing with ball mill

The pure iron was added with 5%wt silica sand nanoparticles, 10%wt silica sand nanoparticles, 15%wt silica sand nanoparticles, and 20%wt silica sand nanoparticles to produce mixture by using ball mill machine for 1 hour. Using 30 ceramics ball as a

grinding media to grind the mixture. From the ball mill, 3 samples from 5%, 10%, 15%, 20% iron-nanosilica is being produced. Weights of every sample are 2 grams.

Figure 4: Ball mill machine

4.3 Compacting

After the mixture was mixing using ball mill, the mixture will be weight 2 gram before compacting. This is because to make syncronize weight for every sample. The compaction is done by using the auto pallet press machine .The 69MPA are need as pressure to make 13mm diameter pallet.

Figure 5 : Autopallet press machine

Below are samples result produced after being compaction by using Autopallet press machine

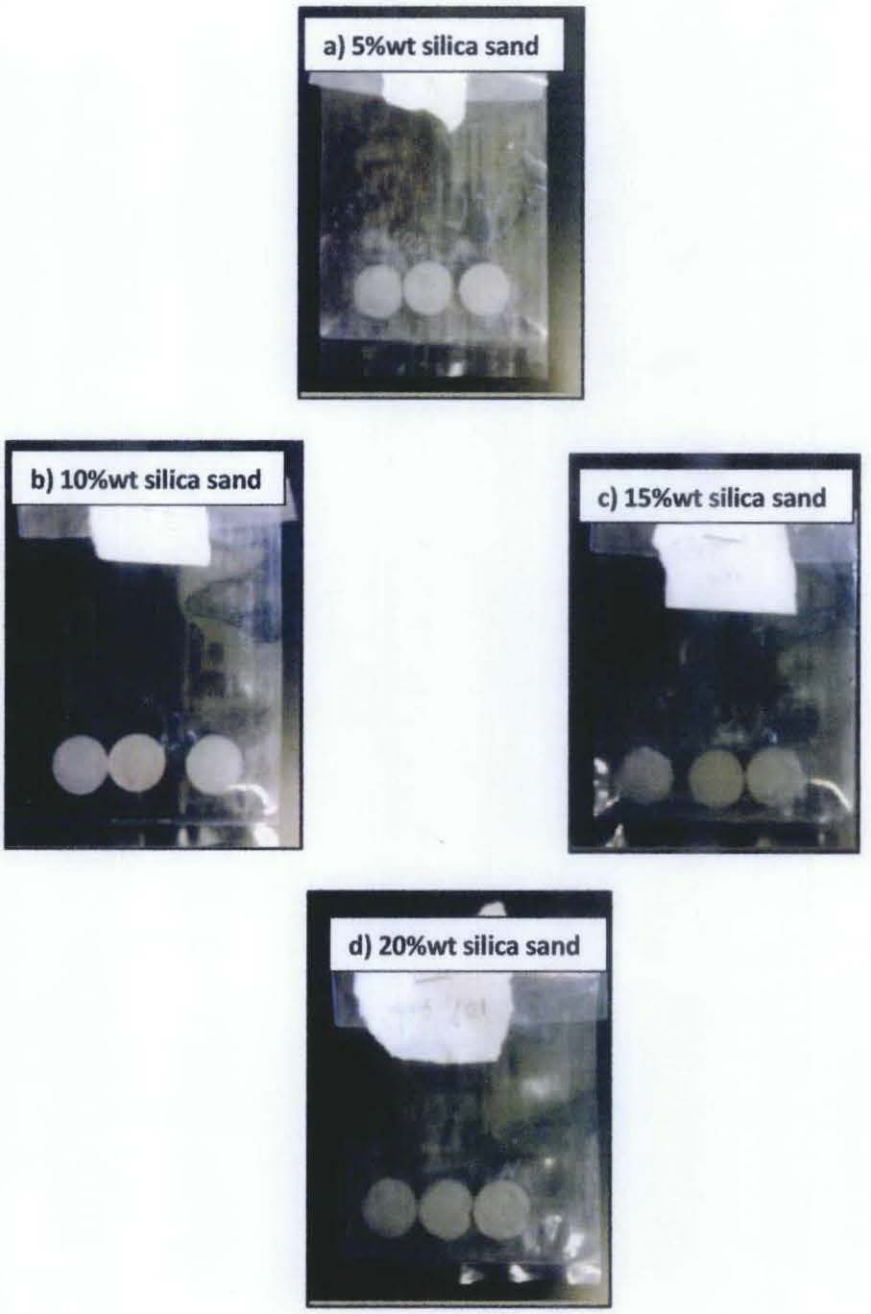


Figure 6 : a) Iron powder added 5%wt silica sand nanoparticles b) Iron added 10%wt silica sand nanoparticles c) Iron added 15%wt silica sand nanoparticles d) Iron added 20%wt silica sand nanoparticles

4.4 Sintering

Now after compaction, we need to get green density using Archimedes method, the density before sintered and after sintered will be comparing. Three batches samples were created, and each batch contains the pure iron, 5wt% silica sand nanoparticles, 10wt% silica sand nanoparticles, 15wt% silica sand nanoparticles and 20wt% silica sand nanoparticle developed and characterized at different sintering temperatures through powder metallurgy technique and sintered at 900 °C, 1000 °C and 1100 °C for two hour under argon atmosphere by using the sintering furnace These temperatures were decided to be as the parameter based on Iron – carbon phase diagram. The heating and cooling rates of sintering process were 5C/min and 10C/min respectively.

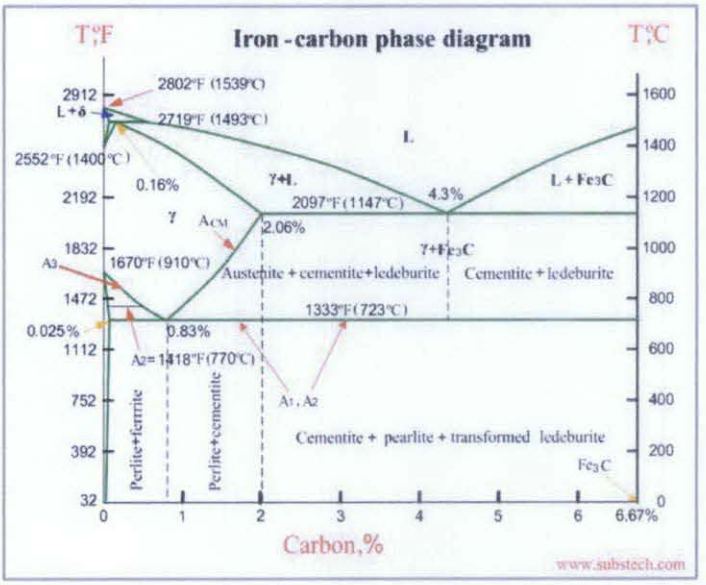


Figure 7 : The Iron - carbon phase diagram [15]

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

FESEM and EDX are needed to show that how silica sand nanoparticles are distributed in iron and occupied the porosity places after sintering. Increasing trend of silica sand nanoparticles indicates that more pores are filled with 20% silica sand nanoparticles as compared to 5% silica sand nanoparticle. The samples are observed at magnifications of 1000x with the resolution of 1nm.

Figure 8 : Field Emission Scanning Electron Microscopic (FESEM)

4.6 Microhardness

The Vickers Hardness Value (HV) was determined using micro hardness tester at constant load of 300gf and dwelling time of 15 seconds.

Figure 9 : Vickers Microhardness Tester

4.7 Setting up the pin on disc wear test using DUCOM Multi Specimen Tester

The main problem to do the wear test using DUCOM multi specimen tester is the holder for the shape using Carver Autopellet Press Machine because the holder are not available for the tablet for shape 13mm x 5mm. So the shape of the holder must be design and manufacture it to make sure the pin on disc wear test using DUCOM multi specimen tester can be implement. Drawing figure 6 show that the holder of the pin.

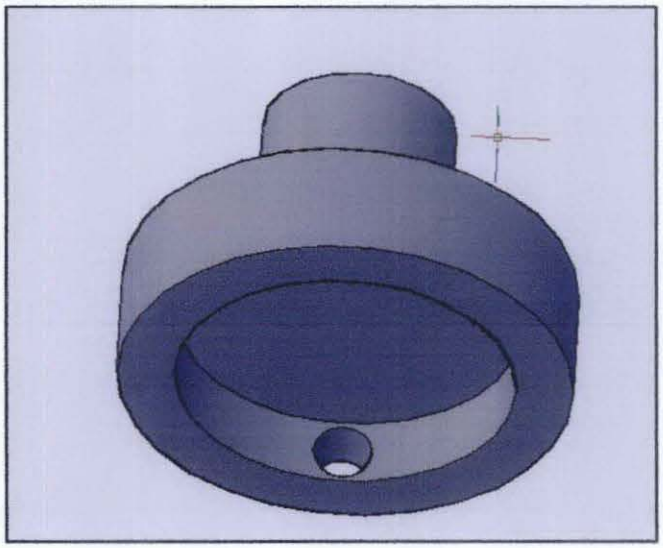


Figure 10: Holder for samples (pin)

The specimen and hardened steel disc were cleaned and dried using cotton dipping with acetone, the mass of each specimen was measured using 4 decimal point electronic digital balances. The specimen was placed and fitted in the bit slot and tightened by rotating chuck key into chuck cap. The chuck key rotated the clamp gear and by rotating the collar. Once the rotation reached exact frequency that matched with speed required, the stop watch was started to record the total time for rotation. When the time was up, the stop button was pushed on the frequency inverter to stop the rotating disc, specimen holder was jerked up. The final mass of specimen was recorded

Figure 11: DUCOM Multi Specimen Tester

The details regarding the pin-on-disk type wear testing apparatus can be referred to Figure 12. When using this machine, user can set the parameters by key in the values. The parameters for the DUCOM Multi Specimen Tester as per Table 5:

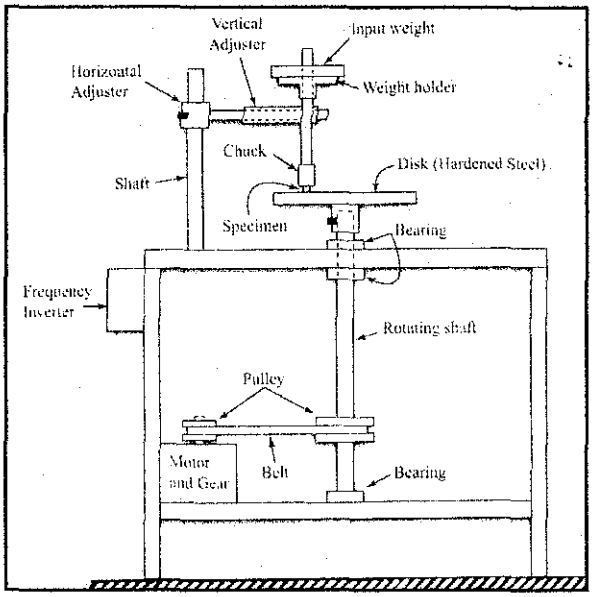


Figure 12: Schematic diagram of the pin-on-disk apparatus

Table 5: DUCOM Multi Specimen Tester Parameters

Variables	**Unit**	**Values**
Load	Kg	10
Rotating speed	RPM	360
Time	Minute	6,
Temperature	°C	Room temperature (24)
Geometry	-	Circular
Material used	-	1) Test specimens (Counterface-1) 2) Disk Material Hardened steel (Counterface-2)
Surface finish	-	Machined surface finish
Type of lubricant	-	Dry

5.0 RESULTS AND DISCUSSION

5.1 Sintered density

Now after compaction, we need to get green density using Archimedes method, the density before sintered and after sintered will be comparing.

Table 6: Comparison of green and sintered densities

Material	Green Density	Sintered@ 900 degree C	Sintered@ 1000 degree C	Sintered@ 1100 degree C
Fe (pure)	5.643	6.119	6.154	6.425
Fe + 5%Nanoparticles	5.125	4.739	5.144	5.401
Fe +10%Nanoparticles	4.576	4.625	4.745	4.869
Fe +15%Nanoparticles	4.087	3.521	4.249	4.286
Fe +20%Nanoparticles	3.929	3.954	4.053	4.132

Refer to the result; increasing trend of silica sand indicated that more pores are filled with 20% silica sand nanoparticles as compared to 5% silica sand nanoparticles. The green densities of the sample are reducing from the 5% iron-nanosilica to 20% iron - nanosilica. Same happen after the samples being sintered at 1000degree C and 1100degree C but for 900 degree C the sintered are not being consistent because density for 20% iron-nanosilica high than 15% iron-nanosilica. Some of sample those being sintered are high from green densities.. However after sintering an improvement in sintered density was observed. More improvement in sintered density was observed in case of 1100 °C sintering temperature due to appearance of liquid phase sintering. The formation of liquid phase is due to the melting of intermetallic compounds between iron

and silica sand nanoparticles which have lower melting point than the sintering temperature

5.2 FESEM and EDS Analysis of the Fe-SiO_2 nanoparticles composites

5.2.1 FESEM Analysis of 5, 10, 15 and 20 wt. % silica sand nanoparticles iron based composite. (900 °C sintering temperature)

Figures 13 (a, b, c and d) shows that how silica sand nanoparticles are distributed in iron and occupied the porosity places after sintering. Increasing trend of silica sand nanoparticles indicates that more pores are filled with 20% silica sand nanoparticles as compared to 5% silica sand nanoparticles. Diffusion welding between the iron and silica sand nanoparticles is started here in sintering temperature of 900 ^{o}C and it will increase as the sintering temperature increased.

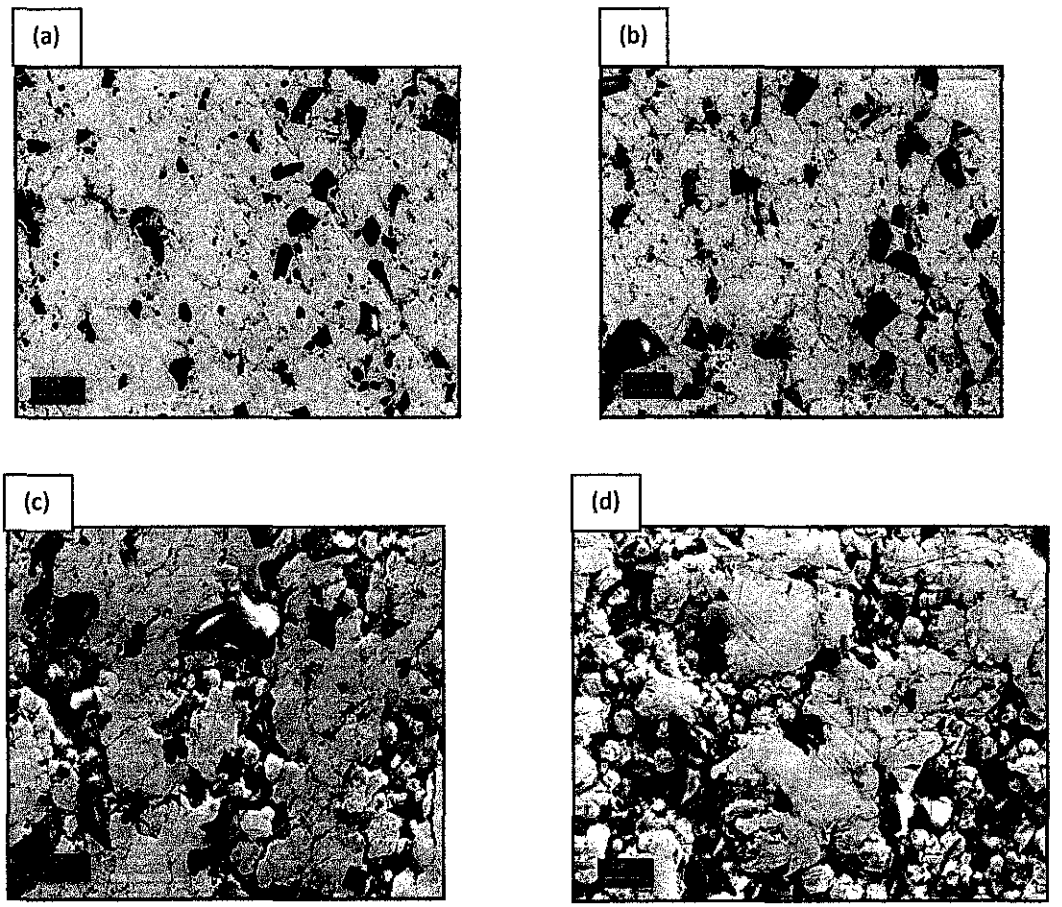


Figure 13 : FESEM analysis of Fe-SiO_2 nanoparticles composites with (a) 5wt.% SiO_2, (b) 10wt.% SiO_2, (c) 15wt.% SiO_2, (d) 20wt.% SiO_2. *(900 °C sintering temperature)*

5.2.2 FESEM Analysis of 5, 10, 15 and 20wt.% silica sand nanoparticles iron based composite. (1000 °C sintering temperature)

As the sintering temperature increased more diffusion take place and also most of the silica sand nanoparticles are connected with iron particles and eaten by them due to diffusion process. More clear and big void were observed in figure 14 (a, b, c and d) also ferritic iron phase and pealite iron phase are more clear and visible as compared to figure 13. These black voids are mixture of silica sand diffused in iron particles. More diffusion welding between the iron particle and silica sand nanoparticles is seen here due to homogeneous structure of the phases.

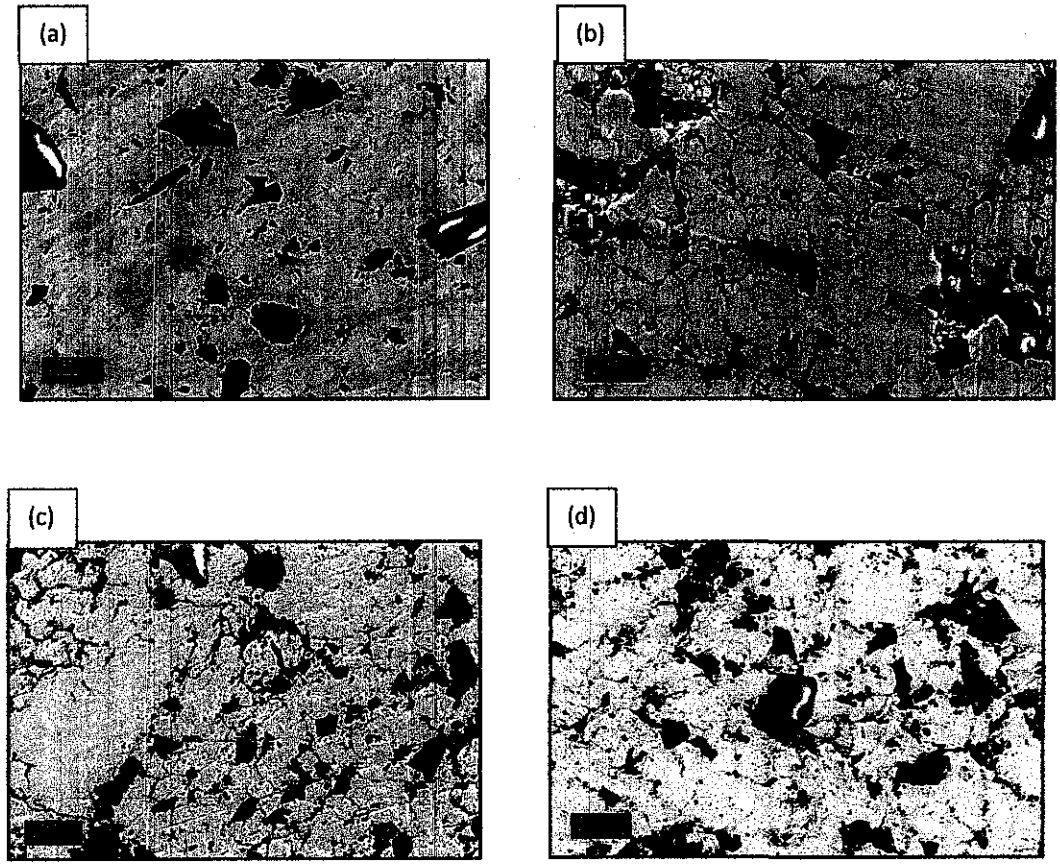


Figure 14 : FESEM analysis of Fe-SiO_2 nanoparticles composites with (a) 5wt.% SiO_2, (b) 10wt.% SiO_2, (c) 15wt.% SiO_2, (d) 20wt.% SiO_2. *(1000°C sintering temperature)*

5.2.3 FESEM Analysis of 5, 10, 15 and 20wt.% silica sand nanoparticles iron based composite. (1100 °C sintering temperature)

More clear and big void were observed in figure 15 (a, b, c and d) also ferritic iron phase and pealite iron phase are more clear and visible as compared to figure 14. The light zones represent ferritic iron. The light grey zones represent lamellar structure of pearlite phase. The dark zones represent voids surrounding the decomposed SiO_2 particles. During the sintering of Fe- SiO_2 compacts, some of SiO_2 particles decomposed into Si and O_2 atoms could diffuse into the Fe particles [9]. These black voids are mixture of silica sand diffused in iron particles.Also decomposition of SiO_2 particles resulted in growth of voids as observed by sintering the composites at 1100 °C, the voids are more big and clear.

The distribution of crystalline domain size for sintered sample: as predicated thermal analysis, the powder sintered at 800 °C is still nanostructure, where at 825 °C the material undergoes a strong grain growth and becomes ulta-fine with some grains even larger than 1 μm [11]. Also from classified metallurgy of steel it is well known that the grain growth can be limited by pinning the grain boundary with precipitate, such as carbides, nitrides, oxides and the intermetallics. Due to better diffusion welding between iron particles and silica sand nanoparticles a more clear and homogenous structure is seen in case of sintering temperature of 1100 °C

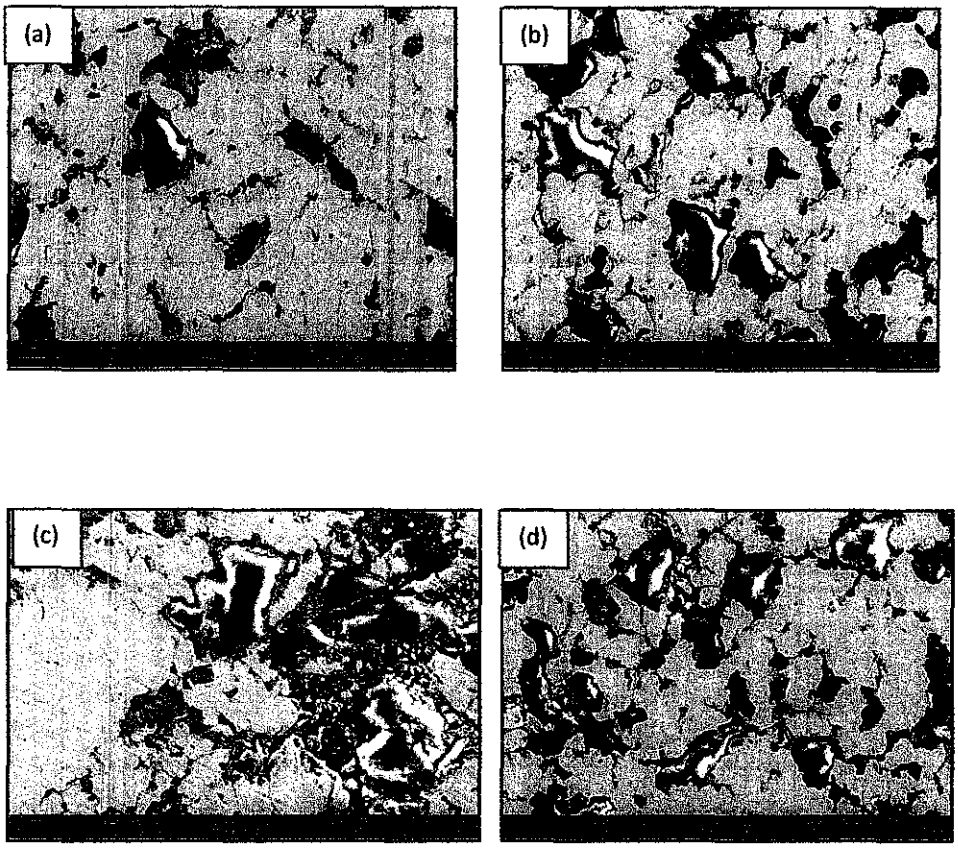
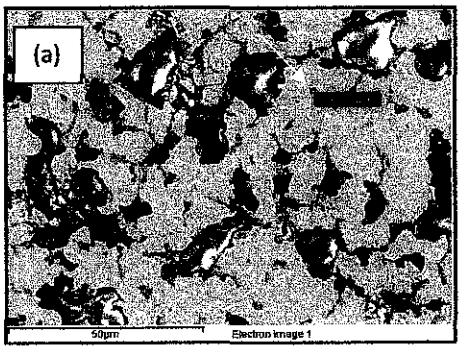


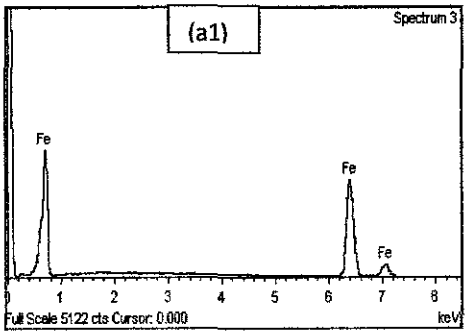
Figure 15 : FESEM analysis of Fe-SiO_2 nanoparticles composites with (a) 5wt.% SiO_2,(b)10wt.% SiO_2, (c) 15wt.% SiO_2, (d) 20wt.% SiO_2. *(1100 °C sintering temperature)*

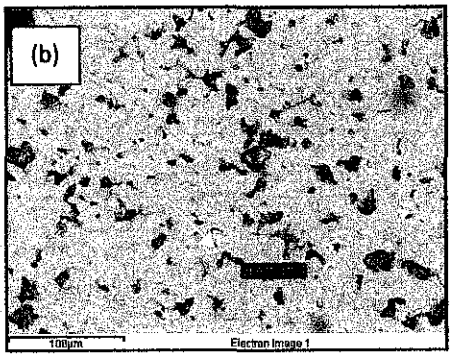
5.3 EDS (point) analysis of silica sand nanoparticles iron based composites (1100 °C sintering temperature)

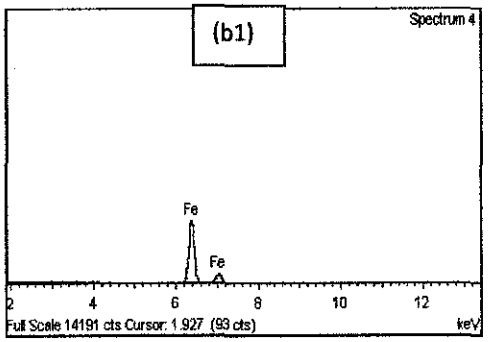
Figure 16 shows the point and EDS analysis where the different points have been taken to verify the description of figure 13 and figure 15: the light zones (figure 16a) represent ferritic iron. The light grey zones (figure 16b) represent lamellar structure of pearlite phase containing a little bit silicon content and make the surface harder. The dark zones (figure 16c, d) 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 EDS analysis of the silica sand nanoparticles iron based composites in figure 16(a1) only Fe peaks are present similarly

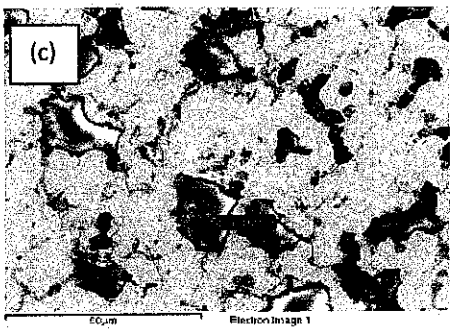
in figure 16(b1) the Fe peaks are present but in figure 16(c1) and figure 16 (d1) where the dark zones has been taken, the peaks of Fe, Si and O_2 are present.

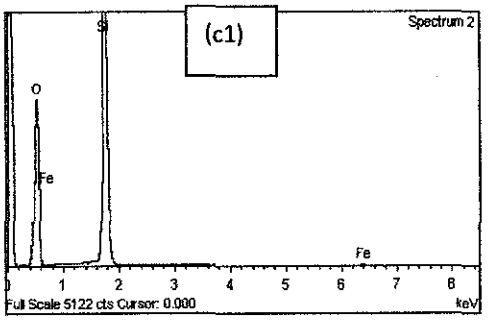


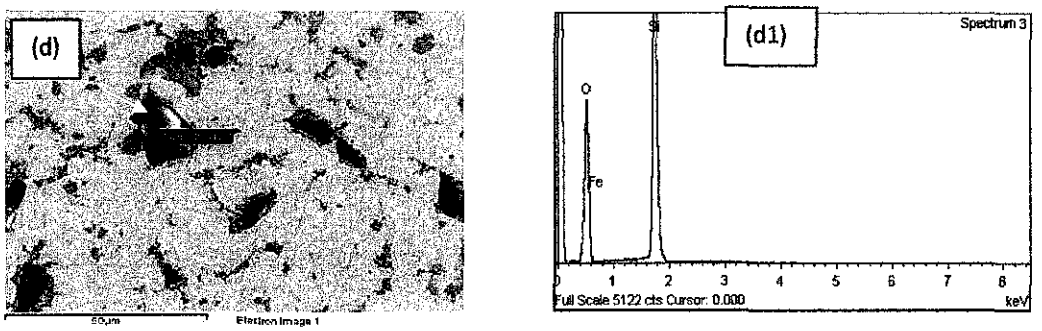












Figures 16: EDS (point) analysis of Fe-SiO_2 nanoparticles composites (*1100 °C sintering temperature)*

5.4 Hardness measurements of Fe-SiO_2 nanoparticles composites.

An increasing trend of hardness was observed with increasing trend of silica sand nanoparticles in iron as well as increasing the sintered temperature as shown in figure 9. The maximum hardness 153.5HVwas achieved in the composites with optimum value of 20 wt.% of silica sand nanoparticles and 1100 °C sintering temperature. Improved hardness may be resulted from solid solution strengthening of by Si atoms and the formation of pearlite phase. Such observation of solid solution strengthen by silicon diffusion was also observed [12]. The micro hardness tests also reveal that increasing trend of hardness is due to dispersion hardening of silica into iron matrix. Also good mechanical properties can be obtained due to good binding interface between the components. Composites that transient liquid phase sintering is only possible with proper sintering temperature which enhanced the diffusivity of alloying elements. The good binding interface provided the good mechanical properties of the composites.

During the micro hardness test, it was found that silicon rich phase have more hardness as compared to iron rich phase. It is due to diffusion of Si in Fe to make FeSi phase during sintering. This FeSi compound phase is main cause of increasing hardness of the composites. The results of micro hardness are verified by FESEM analysis by measuring the diagonal length of the indenter produced for both phases. Because the Si-rich phase does not allow indenter for deep indentation and resist more to indentation. But for Fe-rich phase from microstructure, it is easily observed some of surface is fracture due to indentation load. At 1100 °C there is some level of dissociation of SiO_2 into Si and O_2.

This temperature also approaches the melting temperature of Iron (1535 °C). There is therefore the possibility of a eutectic reaction between the dissociated Si and Fe to form the compound FeSi. This hard phase formed in the Si-rich region of the composite resist to the deep penetration of indenter.

Table 7: Comparison of Microhardness

Composition of nanosilica	900 °C/HV	1000°C/HV	1100 °C/HV
5% of nanosilica	105.3	53.8	87.1
10% of nanosilica	72.5	77.2	96.9
15% of nanosilica	53.9	86.6	138.3
20% of nanosilica	60.3	115.5	153.5

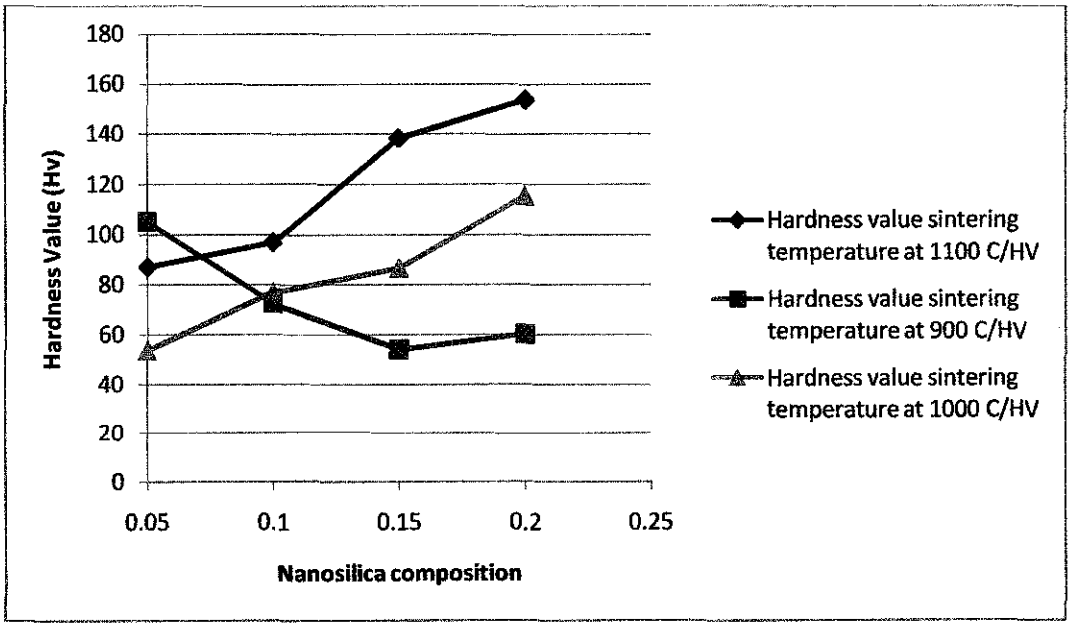


Figure 17: Hardness (Hv) analysis Vs Nanosilica composition

5.5 Wear resistance

Pin on disc type of testing apparatus has been widely to study wear properties and to classify the rank of the material. The test was known as a general test that can determine the sliding wear behavior of materials pairs and its correlation. It was performed according to ASTM Standard G99 (Eyre, 1991) .

The wear mechanisms operative plastic ploughing and grooving of the ironbase matrix phase. At higher loads, removal of the iron from the surface is very high. The material loss is considered to result from removal of material chips from the specimen, due to the microcutting of the abrasive particles. Residual porosity plays an important role when the wear is performed under a high wear load so, the porosity effect must be counted. Porosity in the wear surface of the composite effectively reduces the contacting surface area and thus increases the net wear load. Other factors that increase the wear loss due to porosity are the notch effect and the shape of the porosity

5.5.1 Patterning holder for sample

The main problem to do the wear test using DUCOM multi specimen tester is the holder for the shape using Carver Autopellet Press Machine because the holder are not available for the tablet for shape 13mm x 5mm. So the shape of the holder must be design and manufacture it to make sure the pin on disc wear test using DUCOM multi specimen tester can be implement. Drawing figure 6 show that the holder of the pin.

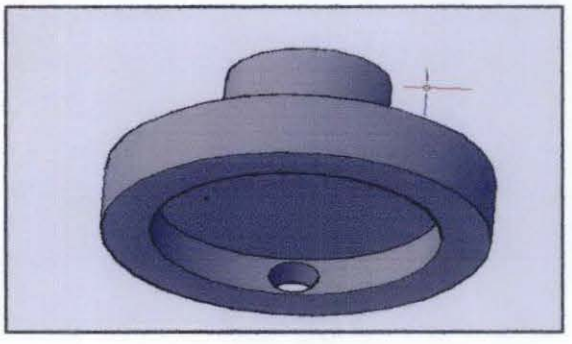


Figure 18: Front view holder for samples (pin)

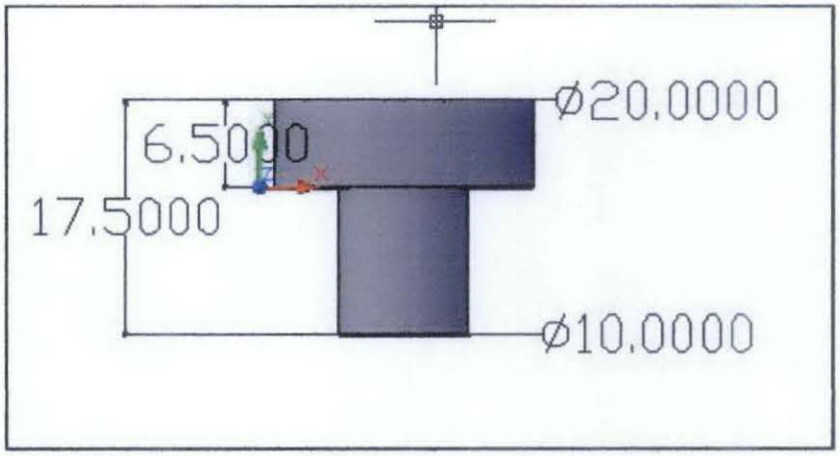


Figure 19: Side view with dimension holder for samples (pin)

5.5.3 Wear test at 100Newton load

Before start the experiments, a surface treatment, e.g. grinding and polishing are done to prevent the surface roughness effect to the materials. Hence, the material's wear decreases as its surface hardness increases, which is consistent with the outcomes of [13]. However, these authors [13] recommend care while interpreting their results, given that some materials may not behave in the same way. This is the new area of research because still no researchers had studied the behavior of the iron based silica sand nanoparticles composites. The test was known as a general test that can determine the sliding wear behavior of materials pairs and its correlation. It was performed according to ASTM Standard G99 (Eyre, 1991) but following parameters should be counted for this research:

1. Different in surface roughness of materials.

   Refer to the sample clearly see that, adding more reinforcement to the iron which is ceramics more surface roughness is produced. An increase in the antagonistic surface roughness significantly increased the wear of composite[8].The friction was found to depend on surface roughness where the rougher surfaces gave higher friction coefficients [14].The wear rate was found to be independent of the roughness, whereas the roughness had a strong influence on the wear rate. surface roughness itself can have a major impact on experimental results, where a

smoother surface usually is the better [14]. The influence of roughness on the friction and the wear rate was stronger than the influence of coating material.

Table 10 shows the weight loss composite reinforced with 5, 10, 15 and 20wt% of silica sand nanoparticles. The iron 5wt% 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 8: Weight loss with 100 Newton load

Compositionof SiO_2	Before(g)	After (g)	Weight loss (g)
5wt%	2.52103	2.48573	0.0353
10wt%	2.19914	2.18604	0.0131
15wt%	2.41111	2.40031	0.0108
20wt%	2.96427	2.96177	0.0025

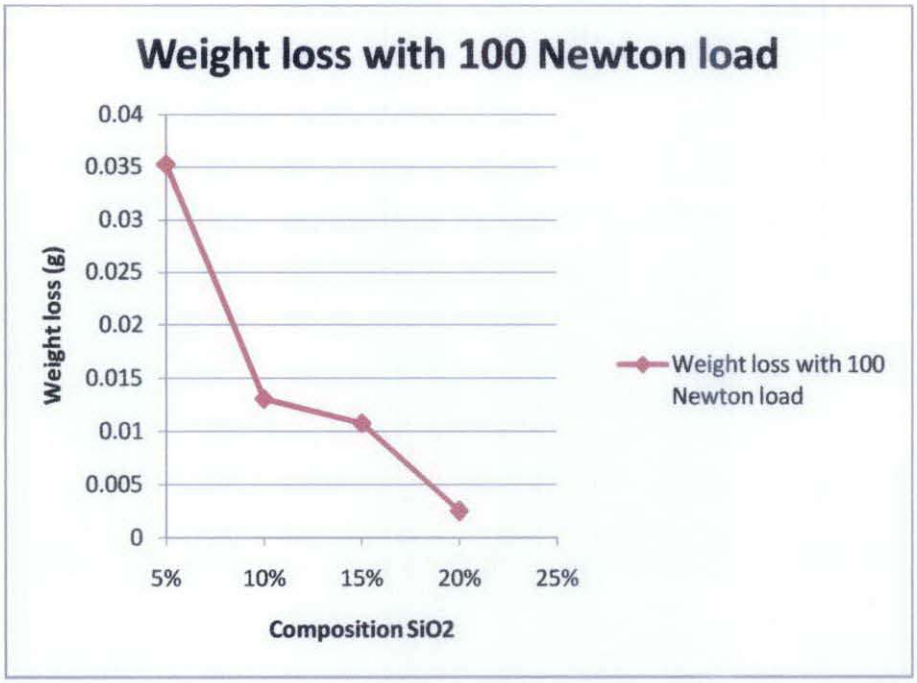
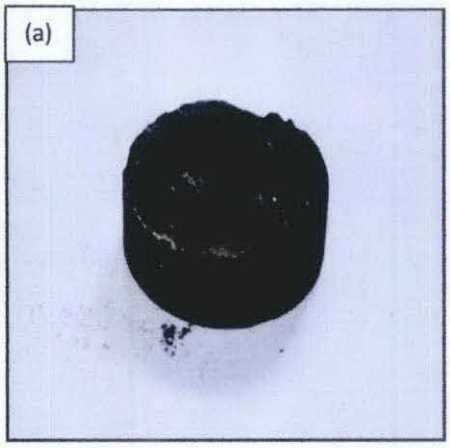
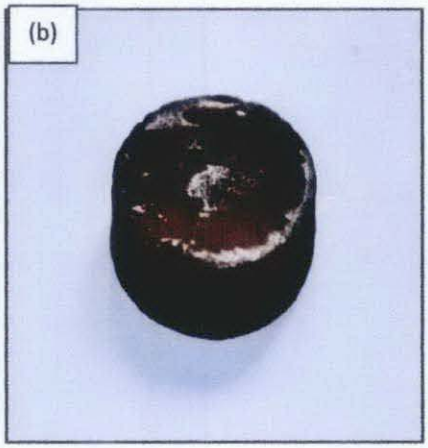


Figure 20: Weigh loss (g) 100 Newton Vs Nanosilica composition





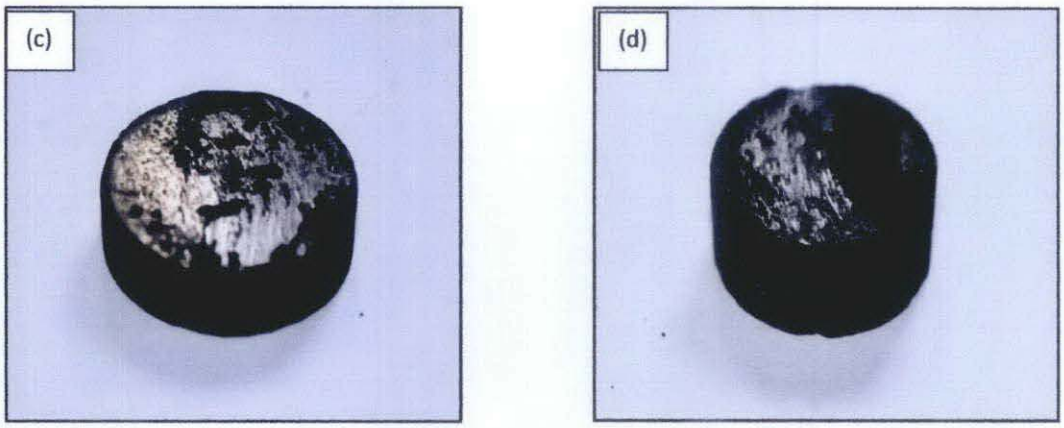


Figure 21 : Sample of being wear of Fe-SiO_2 nanoparticles composites with (a) 5wt.% SiO_2,(b)10wt.% SiO_2, (c) 15wt.% SiO_2, (d) 20wt.% SiO_2. *(100 Newton load)*

5.5.4 SAMPLE CALCULATION FOR WEAR RATE

By using densitimeter mass in air of the sample, m_a and in water, m_w were measured and recorded. To find the density of the sample, the following formula was used;

$$\rho = \frac{m_a}{m_a - m_w} X \rho_w$$

Where ρ_w is the density of water.

Volume loss, W was calculated using the following formula;

$$W = \frac{\Delta m}{\rho}$$

Where $_w$ is the density of water.

Volume loss, W was calculated using the following formula;

Where Δm is the mass loss of specimen due to wear.

The wear rate was calculated using the following formula:

$$W(t) = \frac{W}{t}$$

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

To calculate the wear rate, the mass in air of the sample, m_a and in water, m_w were measured and recorded.

Table 9: summaries the calculation and shows the wear rate of each samples.

Composition of SiO_2	m_a (g)	m_w (g)	*Density* (g/mm^3)	W (mm^3)
5wt%	2.51023	2.09320	0.006019	5.8648
10wt%	2.19664	1.75563	0.004981	2.6299
15wt%	2.41068	1.90107	0.004730	2.2833
20wt%	2.92896	2.29860	0.004646	0.5381

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

Composition of SiO_2	Wear rate, W(t) (10^{-2} mm^3/s)
5wt%	1.6291
10wt%	0.73052
15wt%	0.63425
20wt%	0.14947

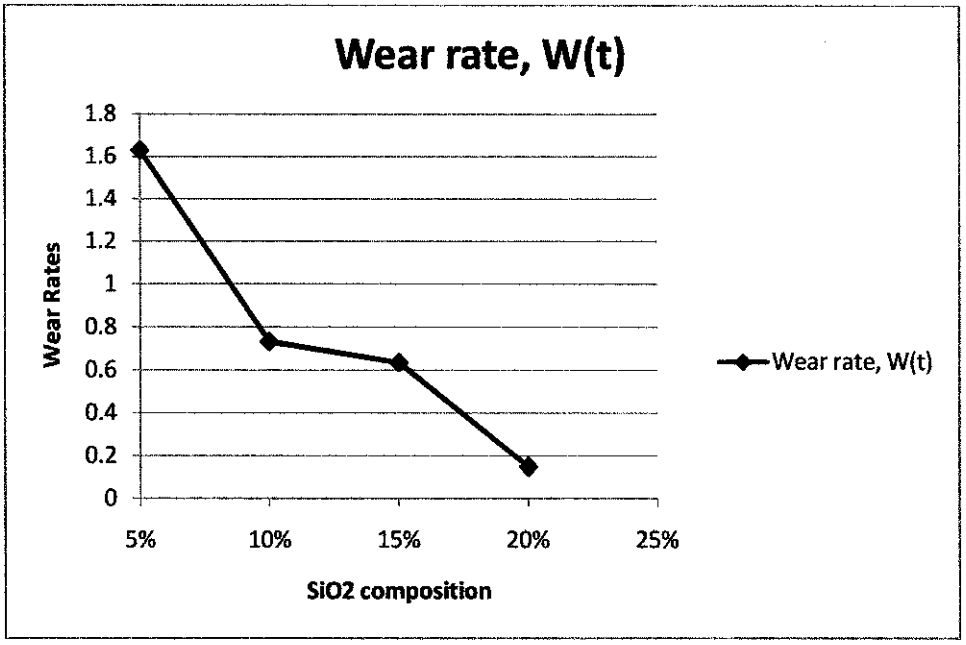


Figure 22: Wear rate mm3/s 100 Newton Vs Nanosilica composition

The composite with 20wt% of silica sand nanoparticles has higher wear resistance compared to pure iron and composite with 5wt% of silica sand nanoparticles. The wear rates of composite decreases as the silica sand nanoparticles contents increases. As the conclusion, the hardness of the composite influences the wear rate of the composite .Increased the hardness of iron silica sand nanoparticles slow down the wear rates of this composites.

6.0 CONCLUSION

The results show that the addition of silica sand nanoparticles to iron enhanced the hardness and wear resistance with increasing the sintered temperature and silica sandnanoparticles. An improvement in sintered densities was also observed with increasing trend of sintering temperatures. An optimum value of 20wt.% of silica sand nanoparticles in iron based composites was found to have best micro hardness values and wear resistance for all sintering temperatures. From FESEM and EDS analysis it is observed that the silica sand nanoparticles diffuse in the porous sites of composites causing an improvement in mechanical properties as well as improved the microstructure. The temperature for sintering must be well selected in order to get the optimum results. This study was done with 3 various temperatures; 900°C, 1000°C, and 1100°C. 1100°C was found to be the suitable temperature because the sintered densities at this temperature were higher than the green densities and this temperature also approaches the melting temperature of Iron (1535 °C). The hardness and the surface roughness of the composites affect the weight losses and the wear rate of the composites. The hardness of iron matrix increases as the silica sand nanoparticles contents increases. The wear results showed that the wear resistance of composites increased with increase of the reinforcement weight fraction due to the strong particulate matrix bonding and high hardness of the silica sand nanoparticles. So from this investigated the iron mixed with 20wt% nanosilica sand particles show the best result on hardness and wear resistance.

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