The Microstructure, Physical and Mechanical Properties of Silicon Carbide for High Temperature Wear Application: An Experimental Observation

by

Muhammad Ismeth Amir bin Johari
15019

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

JANUARY 2015

Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

The Microstructure, Physical and Mechanical Properties of Silicon Carbide for High Temperature Wear Application: An Experimental Observation

By

Muhammad Ismeth Amir bin Johari
15019

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

Approved by,

_____

(Dr. Mazli Mustapha)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
January 2015

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.

_____

MUHAMMAD ISMETH AMIR BIN JOHARI

ABSTRACT

Silicon carbide has gain much attention in recent years as the best material for the application in harsh environment condition. This is due to their excellent properties such as good wear resistance with high hardness and strength at elevated temperature. In this study, the sintered specimen of silicon carbide with sintering additives of four different composition were fabricated using powder metallurgy method. Physical properties were measured by means of bulk density and apparent porosity whereas the mechanical property was measured in term of hardness. From this study, it can be observed that the silicon carbide was sintered to about 92% of its theoretical density by using alumina and yttria as sintering additives. This finding was due to eutectic liquid formed between alumina and yttria at sintering temperature of 1930°C. The specimen with 25% wt. of sintering additives gave the maximum value of hardness of 25.5 GPa.

ACKNOWLDEGEMENT

In the name of Allah, the Most Gracious and Merciful, all the praises and thanks goes to the Almighty whom we ultimately depend for sustenance and guidance. Without His blessing, it would be almost impossible for the author to complete this final year project. The author would like to express his deepest gratitude and appreciation to all those who have played their part in assisting him during the completion of the project.

The author would like to express his special thanks toward Universiti Teknologi PETRONAS and Mechanical Engineering department for providing an opportunity for the project to be properly planned and conducted. This opportunity has managed to help the author by having a better understanding on how a proper research project should be done.

Besides that, the author would like to take this chance to express his utmost gratitude and appreciation towards his personal project supervisor, Dr. Mazli Mustapha whom has provide a significant role in the project. His generous guidance and continuous support made it possible for the author to overcome any obstacles faced. The blessing, assistance and advice given by him can never be repaid and shall always be remembered.

Last but not least, the author would like to thank his family and friends for their continuous support and encouragement which has made it possible for the author to produce this report. Thank you very much.

TABLE OF CONTENTS

CERTIFICATION OF APPROVAL……II

CERTIFICATION OF ORIGINALITY……...III

ABSTRACT……IV

ACKNOWLEDGEMENT……V

TABLE OF CONTENTS……VI

LIST OF FIGURES……VII

LIST OF TABLES……....IX

CHAPTER 1: INTRODUCTION

1.1 Background of Study……..1

1.2 Problem Statement……..2

1.3 Objective and Scope of Study ……...….2

CHAPTER 2: LITERATURE REVIEW

2.1 Overview of Silicon Carbide……...4

2.2 Powder Metallurgy……..5

2.3 Experimental Works……6

CHAPTER 3: METHODOLOGY

3.1 Research Methodology……7

3.2 Activities and Key Milestone (FYP I)……8

3.3 Activities and Key Milestone (FYP II)……9

3.4 Powder Metallurgy……..10

3.5 Sample Preparation……..11

3.6 Hardness……....……14

3.7 Density Measurement……...15

3.8 Microstructure Analysis……...17

CHAPTER 4: RESULTS AND DISCUSSION

4.1 Linear Shrinkage…….18

4.2 Hardness……..19

4.3 Density Measurement……..20

4.4 Comparison of Density and Hardness…….21

4.5 Microstructure Analysis…….....22

CHAPTER 5: CONCLUSION AND RECOMMENDATION...……..26

REFERENCES…….27

LIST OF FIGURES

Figure 1.1: A flow chart of powder metallurgy method and sample analysis……...3

Figure 3.1: Research Methodology flow chart……..7

Figure 3.2: Buehler Delta Abrasimet 2145 Manual Chop Cutter……..11

Figure 3.3: Buehler SimpliMet 1000 Automatic Mounting Presses…….....11

Figure 3.4: Buehler Metaserv 2000……...12

Figure 3.5: Vickers Hardness indentation and measurement…….14

Figure 3.6: LECO LM 247 AT micro hardness tester……....15

Figure 3.7: Mettler Toledo Density Measurement Kit……...16

Figure 3.8: PHENOM Pro X desktop scanning electron microscope……....17

Figure 4.1: Vickers hardness result of samples with different additives composition……19

Figure 4.2: Archimedes' density results of samples with different additives composition……...…….20

Figure 4.3: Graph of density and percentage of porosity of all four samples…….21

Figure 4.4: Graph of density and hardness for all samples showing the correlation between microstructure and mechanical property……...21

Figure 4.5: Microstructures of sample 4…….....22

Figure 4.6: Selected area of specimen 4 for spot elemental analysis denoted as spot 1 and 2 respectively.……..23

Figure 4.7: Elemental weight percent of spot 1 for sample 4……..……...24

Figure 4.8: Elemental weight percent of spot 2 for sample 4..……...24

Figure 4.9: (a) Mapping analysis of selected area of specimen 4 and distribution of element of (b) silicon (c) aluminium (d) yttrium…….….25

LIST OF TABLES

Table 3.1: Final Year Project I Gantt chart……..8

Table 3.2: Final Year Project II Gantt chart…….9

Table 3.3: Starting composition of the samples……..10

Table 3.4: Etchant preparation……13

Table 4.1: Linear Shrinkage at temperature 1930˚C……...18

Table 4.2: Vickers hardness result of samples with different additives composition……19

Table 4.3: Archimedes' density of samples with different additives composition……20

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Silicon carbide (SiC) was discovered in year 1890 by the assistant to Thomas Edison, Edward G. Acheson. It was when during an experiment on the synthesis of diamonds, he actually discovered the new material by accident. Acheson firstly thought the new material was a compound made of carbon and alumina, which then leading him to name it carborundum. Silicon carbide occurs naturally in meteorites, although very seldom and in very small amounts. The material is composed of atoms from carbon and silicon which is bonded with strong chemical bonds in the crystal lattice. This results in a very hard and strong material.

During the early days, the ceramics used as conventional industrial materials consisted of mainly alumina and other oxides. However, there have been strong demands in recent years for the use of ceramics to replace materials such as metals and alloys in application for harsh environments. Consequently, new ceramics such as carbides and other covalently bonded materials have received high attention due to their unique characteristics.

It is known that silicon carbide has outstanding mechanical properties including high strength, stiffness, good resistance to wear and corrosion. Besides that, its high thermal conductivity and low thermal expansion gives this material exceptional thermal shock resistant qualities. All these characteristics mentioned makes this material as excellent candidate in harsh environment applications.

1.2 Problem Statement

SiC have become the first choice for many high temperature applications due to their outstanding mechanical and physical properties. Compared with other compound, SiC is provided with the characteristics like high hardness and strength at elevated temperature. However, the material can be very difficult to produce without the use of sintering aids. This is mainly due to the strong bonds which exists between carbon and silicon atoms in the crystal lattice. Without the use of any aids, the sintering process need to achieve high sintering temperature around 2000 to 2300 ˚C for the process to be completed. This is not very convenience as it is very difficult to reach the required temperature. With the use of sintering aids such as boron oxides and aluminium oxides, sintering temperature can be lowered down to below 2000 ˚C point. The purpose of this study is therefore to investigate the influence of aluminium oxide, Al_2O_3 and yttrium oxide, Y_2O_3 at eutectic composition on the microstructure characteristics and also the physical and mechanical properties of sintered silicon carbide.

1.3 Objective and Scope of Study

The main objectives of this project are:

1. To investigate the physical and mechanical properties of sintered silicon carbide specimen.
2. To analyse the microstructure of the sintered silicon carbide specimen.

SiC particles can be synthesized by using powder metallurgy method which consist the steps such as blending, compacting and sintering. Parameters such as compact pressure, sintering time and temperature used were constant for every specimen produced. In this experiment, specimen are produced through the combination of SiC powders, aluminium oxide, Al_2O_3 and yttrium oxide, Y_2O_3. The effect of four different sintering additives composition on the specimen were then investigated through density measurement, hardness testing and microstructure analysis. Standardized testing methods which has been developed acted as the main reference when experiments was conducted. Figure 1.1 summarized the project methodology that were involved during this experimental study.

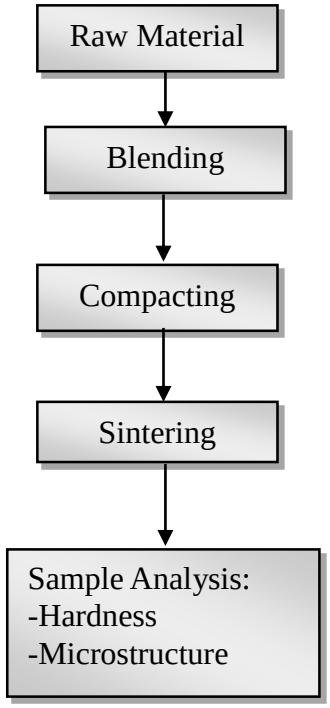


FIGURE 1.1: A flow chart of powder metallurgy method and sample analysis.

CHAPTER 2

LITERATURE REVIEW

2.1 Overview of Silicon Carbide

Silicon Carbide, is a compound which composed of silicon and carbon with chemical formula of Si-C. According to Huebner (2009), SiC consists of one atom of silicon bonded to another one atom of carbon which combined together and produces strong bond that is extremely stable in various range of temperatures and chemical environments. It also has other desirable properties such as a high hardness and high modulus of elasticity. Prochazka et. al (1978) suggests that chemical and physical properties found in silicon carbide make the compound as excellent material for high temperature applications. These properties include good oxidation and corrosion resistance, good heat transfer coefficients, low expansion coefficient, high thermal shock resistance and high strength at elevated temperature. However, as a ceramic material, SiC can be classified as quite brittle and has less mechanical strength than that of other metal carbide. Besides that, it is also can be susceptible to chipping and fracture when provided with large mechanical stress or shock (Fluid Sealing Association, 2006). Among other disadvantages is that the material is quite difficult to be manufactured in any required shapes for component design (Huebner, 2009).

In terms of high temperature wear application, silicon carbide has been available for more than 30 years ago. One of the example is the use of SiC as the main material for mechanical seal. Mechanical seal, as defined by Huebner (2009), are a type of device which are used to prevent and eliminate leakage of process fluid in equipment such as pumps and mixers. Overall, the use of a good mechanical seal can helped in improving equipment reliability, reduced emissions and can subsequently help to improve safety factor.

2.2 Powder Metallurgy

Huang et. al. (2002) emphasize that SiC is very difficult to be densified without the use of suitable additives and external pressure. This is mainly due to the covalent nature of silicon-carbon (SI-C) bonding and its low self-diffusion coefficient. There are many types of sintering aids available, and each of them has their own advantages and disadvantages. The main reasons for using such aids is to help reducing sintering time and temperature (Carlson, 2013). Among popular sintering aids to obtain a dense SiC is oxides such as Al_2O_3 and Y_2O_3. Liquid-phase sintering (LPS), with oxide additives, was first experimented in 1982 by Omori and Takei. They showed that β-silicon carbide could be densified by sintering at the temperature of 2100˚C, using yttrium and aluminium hydroxide. It was observed that the additives in later stage reacted with each other and formed oxides subsequently facilitated the densification process via liquid-phase sintering. The liquid phase which is formed between the reactants enable low temperature sintering, and can also help to improve the microstructure of the material during the sintering or heat treatment (Jong-Kook et. al., 2002).

In addition to that, Prochazka et. al. (1978) suggests that besides the use of sintering aids, very fine SiC powders can also help to allow densification occurred at the lowest possible temperature. The grain type of silicon powders used also can help to refine specimen's microstructures in order to obtain desirable mechanical properties. They also conclude that sintering process usually takes about 10 to 60 minutes before completed. They pointed out that sintering time however could not provide any significant impact to the outcome. It is known that during hot pressing process, the atmosphere use must be inert to the mixture. Hence, any oxidizing atmospheres such as air cannot be used since they tend to convert the compound into silica by oxidation process. Such examples of inert atmospheres include argon, helium, and nitrogen gaseous.

From an experiment done by Ihle et. al. (2004), they produced SiC with a composition of 50 % by weight of α-Si-C and 50 % by weight of additives. The additives use consisted of 64.4 % by weight of Al_2O_3 and 35.6 % by weight of Y_2O_3. The samples were then formed into discs with a thickness of 5 millimetres and a diameter of 25 millimetres. The actual molar composition recorded of the sintered SiC from the

experiment were 15.8 mol of Si-C, 4 mol of Al_2O_3, 1 mol Y_2O_3 and 1 mol of argon. On the other hand, the composition of additives use was 4:1 mol ratio of Al_2O_3 and Y_2O_3 respectively.

2.3 Experimental Works

For the result part, indentation tests were conducted to calculate the hardness of the sintered silicon carbide. Hardness can be define by the resistance of metal to plastic deformation. Besides determining the hardness of the material, the indentation results can used to calculate fracture toughness (Carlson, 2013). Vickers test are mentioned in literatures as simple to use when compared with other alternative hardness tests. This is because the required calculations are independent of the size of the indenter in Vickers. Besides that, the indenter can also be used for all materials regardless of its hardness.

The density of the final specimen were measured using Archimedes' method. This method of measuring density is consider by many as more accurate than any other density measurement. Water is use as the main medium, where it disperse to fill the open porosity available in the material. The final measured density therefore only include the solid material and any closed porosity. However, the degree of water penetration into the open pores can be limited by the size of the water molecule itself and.

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

To achieve the objective of this study, every steps taken were checked carefully and thoroughly in order to ensure the smoothness of flow of this project. It is very important to minimize any errors which could later provide obstacle to achieve the proposed objectives. For this study, journals from scholars and paper works from academician were the main reference to understand the features and characteristics of SiC. Then, experimentations are carried out to investigate the physical and mechanical properties of the sintered material as well as its microstructure feature. Figure 2 below, shows the workflow for research methodology**:**

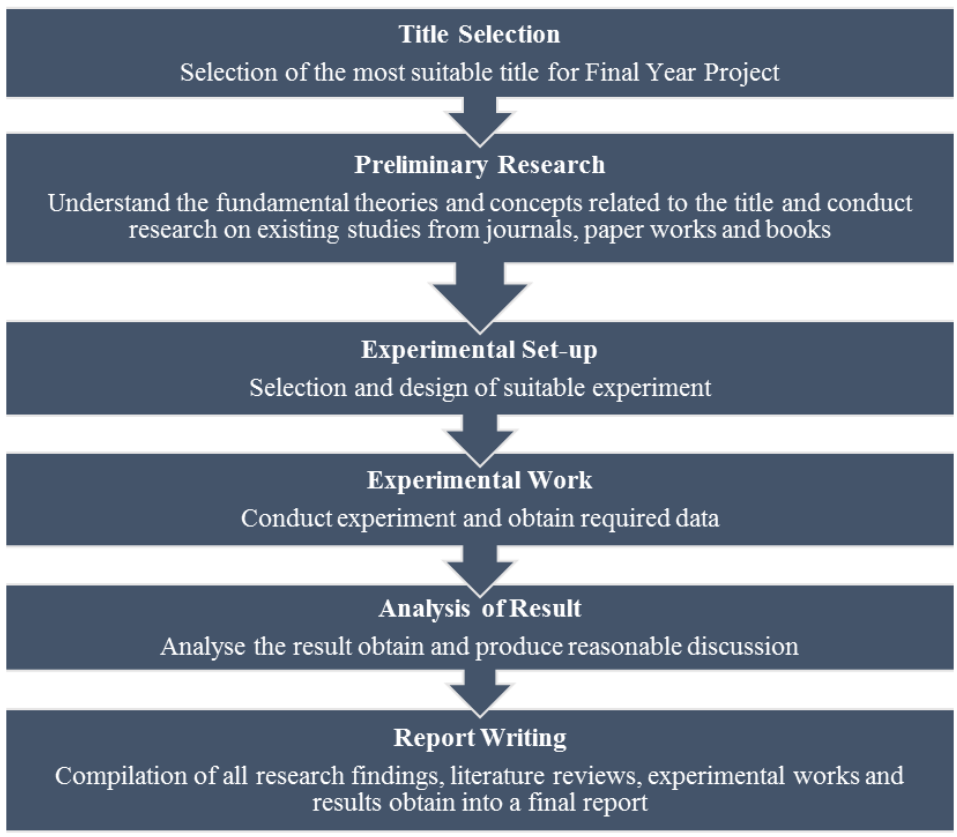


FIGURE 3.1: Research Methodology flow chart.

3.2 Activities and Key Milestones (FYP I)

TABLE 3.1: Final Year Project I Gantt Chart.

No	Details	Week													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of Project Topic	●													
2	First meeting with Supervisor		●												
3	Preliminary Research Work					●									
4	Preparation of Extended Proposal -Background of study -Problem Statement -Objectives and Scope of Study -Literature Review -Methodology -Conclusion						●								
5	Submission of Extended Proposal						●								
6	Proposal Defence									●					
7	Project work continues														
8	Preparation of Interim Report -Abstract - Background of study -Problem Statement -Objectives and Scope of Study -Literature Review -Methodology -Conclusion and Recommendation													●	
9	Submission of Interim Draft Report													●	
10	Submission of Interim Report														●

● Key Milestone Process

3.3 Activities and Key Milestones (FYP II)

TABLE 3.2: Final Year Project II Gantt chart.

No	Details	Week														
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Project work continues - Develop specimen through powder metallurgy	■	■	■	■	■	■	●								
2	Submission of Progress Report							●								
3	Project work continues - Microstructure Analysis - Density Measurement - Vickers Hardness								■	■	■	■	■	■	●	
4	Pre-SEDEX										●					
5	Submission of Draft Final Report											●				
6	Submission of Dissertation (Soft-bound)												●			
7	Submission of Technical Paper												●			
8	Viva Presentation													●		
9	Submission of Project Dissertation (Hard-bound)															●

● Key/Suggested Milestone ■ Process

3.4 Powder Metallurgy

Test specimen were produced from starting powder of original powder of β-SiC. The powder were then blended with sintering additives of Al_2O_3 and Y_2O_3. Four different composition of SiC and sintering additives were sintered. Table 3.3 below summarized the starting composition of the samples produced.

TABLE 3.3: Starting composition of the samples.

Sample	Composition (wt. %)	
	SiC	Additives
1	95	5
2	90	10
3	85	15
4	75	25

The samples formed into discs shape with a thickness of 10 millimetres and a diameter of 20 millimetres. Maximum compact pressure were used in compaction process with force of 147 kN. Conventional solid-state sintering were completed in high temperature furnace in flowing argon at 1 bar. All samples were sintered at 1930˚C with dwell time of 1 hour.

3.5 Sample Preparation

3.5.1 Sectioning

The most common device used in sectioning is by using the abrasive cut-off machine. Abrasive sectioning was performed in wet condition where sufficient flow of coolant was directed into the cutting section. Wet cutting can help to produce a smooth surface finish as well as preventing excessive surface damage on the sample. The sectioning of the sample was done using Buehler Delta Abrasimet 2145 Manual Chop Cutter as shown in Figure 3.2 below. It was ensured that the samples were clamped tightly during cutting and pressure were applied carefully.

FIGURE 3.2: Buehler Delta Abrasimet 2145 Manual Chop Cutter.

3.5.2 Mounting

The primary purpose of mounting specimens is for convenience in handling of handling of difficult shapes and sizes of samples. Besides that, mounting can also provide protection and preservation of edges or surface defects during materials preparation. The samples were mounted using hot mounting method where thermosetting polymeric powder was used. The powder was placed in a mold with the specimen and suitable heat and pressure was applied. The mounting process is done was using Buehler SimpliMet 1000 Automatic Mounting Presses as shown in Figure 3.3.

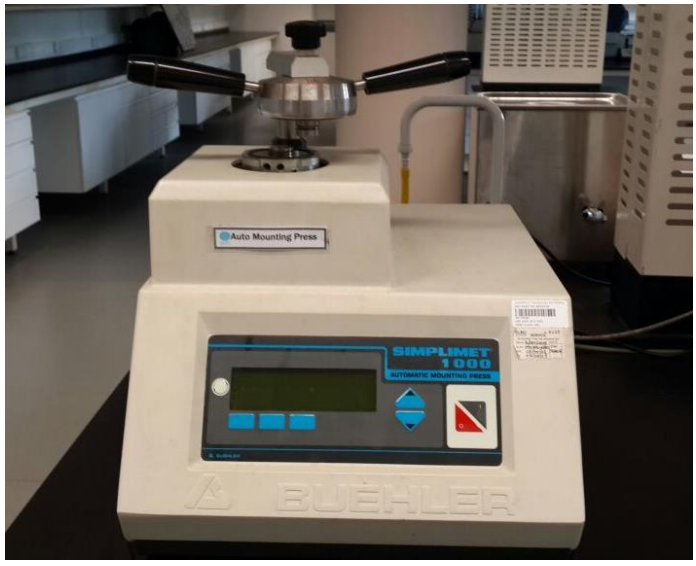


FIGURE 3.3: Buehler SimpliMet 1000 Automatic Mounting Presses.

3.5.3 Grinding

Grinding process was completed in order to minimize thickness of damaged layer from the sectioning process earlier. Similar with sectioning process, all grinding steps should be performed wet with water as lubricant in order to minimize sample heating. The initial abrasive size use was to establish a flat sample surface and to remove the damaged layer from cutting. Each following abrasive sizes then help to remove the damage from the previous step. The grades of grinding abrasives used is 180, 320, and 600 grit (grains per square inch). The flatness of the surface of the samples must be ensured throughout the grinding steps.

3.5.4 Polishing

Polishing produced a deformation free surface that is flat, scratch free and also have mirror-like appearance. Such surfaces are very necessary in order to have the right interpretation of microstructure of the specimen. Both polishing and grinding were completed using Buehler Metaserv 2000 in Figure 3.4.

FIGURE 3.4: Buehler Metaserv 2000

3.5.4 Etching

The main purpose of etching is to remove the final thin layer of deformation created from the earlier sample preparation processes. Each particular etchant will preferentially attack particular sites on the sample surface with the 'highest energy', leading to various features able to be distinguished for microstructure analysis later. Etching commonly refers to chemical etching, where a dilute chemical solution is used to selectively corrode certain microstructural feature on the samples. For SiC, the etchant used was Murakami's Reagent which is suitable for carbides based material. The following Table 4 describes the preparation of Murakami's Reagent.

TABLE 3.4: Etchant preparation.

Etchant	Composition	Concentration	Conditions	Comments
Murakami's Reagent	Potassium Ferricyanide, $K_3Fe(CN)_6$	10 g	Pre-mix KOH and water before adding $K_3Fe(CN)_6$	Cr and alloys (use fresh and immerse); iron and steels reveals carbides; Mo and alloys uses fresh and immerse; Ni-Cu alloys for alpha phases use at 75 Celcius; W and alloys use fresh and immerse; WC-Co and complex sintered carbides.
	Potassium Hydroxide, KOH	10 g		
	Water	100 ml		

3.6 Hardness

Micro indentation of the specimen was conducted through a series of five different measurements in compliance with ASTM C1327 in which stated that at least five acceptable indentations shall be made. The indentation and measurement of Vickers Hardness are displayed in Figure 6. All samples were indented with a load value of 1 kgf (9.81 N) with a dwell time of 15 seconds. A series of indentations then must be made with increment of load until radial cracks are optically visible. At least two different crack length are needed before the fracture toughness of the material can be determined. The equation needed to find both hardness and fracture toughness are shown below. As silicon carbide is classified as among ceramics material, the hardness indentation test will be conducted by following the procedure given in ASTM C1327. Figure 3.5 shows the Vickers hardness indentation and measurement. Hardness test was conducted on the specimen using LECO LM 247 AT micro hardness tester as shown in Figure 3.6.

$$Vicker's\ Hardness\ (HV) = \frac{F}{A} = \frac{1.8544F}{d^2} \qquad \textbf{Eq. 1}$$

$$Fracture\ Toughness\ (\ K_{Ic}\ ) = Y\sigma\sqrt{\Pi c} \qquad \textbf{Eq. 2}$$

Where **Y** is geometric factor and **c** is the surface crack length.

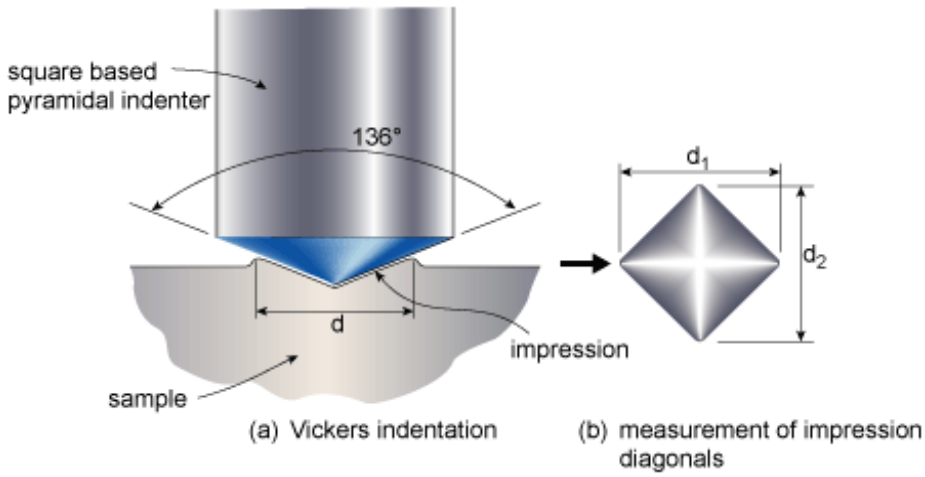


FIGURE 3.5: Vickers Hardness indentation and measurement.

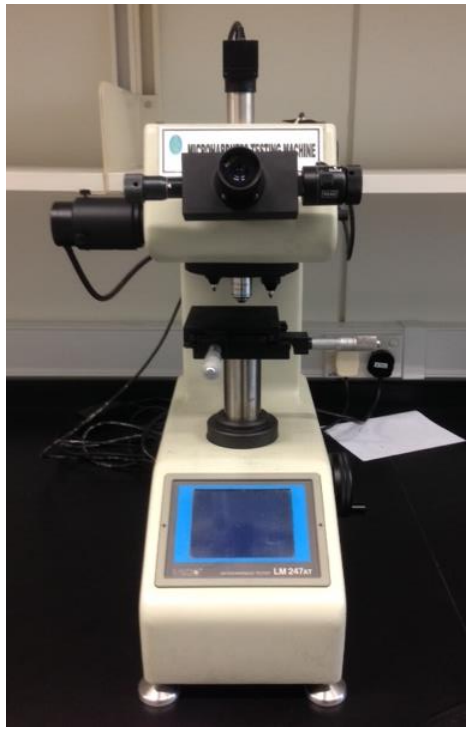


FIGURE 3.6: LECO LM 247 AT micro hardness tester.

3.7 Density Measurement

Density measurement of a material although it is not very significant, however, the correct measurement of density can help to provide the understanding of the relation between microstructure and mechanical properties. The density of the silicon carbide samples were assessed by using Archimedes method based on ASTM B962. The Archimedes method will be performed by drying the sample followed by the measurement of the dry weight. After that, the sample will submerged in deionized water and will be again weighed. Finally, the saturated sample will be removed from water and brushed with a cloth to remove surface water, and the saturation weight will be measured again. The pore volume, the geometric volume, and the percentage of porosity will be then calculated using equations which are as follows:

$$Volume\ of\ pores = \frac{weight_{saturated} - weight_{dry}}{density\ of\ fluid} \quad \textbf{Eq. 3}$$

$$Volume\ of\ geometric = \frac{weight_{saturated} - weight_{submerged}}{density\ of\ fluid} \quad \textbf{Eq. 4}$$

$$Percentage\ of\ porosity = \frac{volume_{pores}}{volume_{geometric}} \quad \textbf{Eq. 5}$$

Derivation of the Archimedes' Method,

$$Dry\ Weight = \rho_A V_A \quad \textbf{Eq. 6}$$

$$Saturated\ weight = \rho_A V_A + \rho_{water} V_{pores} \quad \textbf{Eq. 7}$$

$$Submerged\ Weight = \rho_A V_A - \rho_{water} V_A \quad \textbf{Eq. 8}$$

Where ρ_A and V_A are the density and volume of the bulk material (excluding porosity).

$$\frac{Saturated - Submerged}{\rho_{water}} = \frac{(\rho_A V_A + \rho_{water} V_{pores}) - V_A(\rho_A - \rho_{water})}{\rho_{water}} = V_{geometric} \quad \textbf{Eq. 9}$$

$$\frac{Saturated - Dry}{\rho_{water}} = \frac{(\rho_A V_A + \rho_{water} V_{pores}) - \rho_A V_A}{\rho_{water}} = V_{pores} \quad \textbf{Eq. 10}$$

The density measurements through Archimedes' Method were completed using Mettler Toledo density measurement kit as shown in Figure 3.7 below.

FIGURE 3.7: Mettler Toledo Density Measurement Kit

3.8 Microstructure Analysis

After the samples has been sintered, the final form of the densified SiC were examined through its microstructure characteristics. Phenom Pro X desktop scanning electron microscope (Figure 3.8) were used to obtained the microstructure images of sample 4. Apart from that, energy dispersive X-Ray Spectroscopy (EDS) were utilised to estimate element weight percent in the sample. Mapping analysis also were done by using EDS to observe the distribution of element of silicon, alumina, yttrium and oxides. In order to reveal the grain characteristic of the samples, etching were first carried out by immersing the sample with Murakami's Reagent.

FIGURE 3.8: PHENOM Pro X desktop scanning electron microscope

CHAPTER 4
RESULTS AND DISCUSSIONS

4.1 Linear Shrinkage

Table 4.1 shows the linear shrinkage percentage of all samples after sintering process have been completed at 1930˚C.

TABLE 4.1: Linear Shrinkage at temperature 1930˚C

Sample	Additives (wt. %)	Shrinkage (%)
1	5	18.2
2	10	18.8
3	15	18.9
4	25	19.0

The results indicate that shrinkage for all samples at the same sintering temperature are within 18.2 to 19.2 %. Since the type and ratio of additives, sintering temperature, time and atmosphere were held constant during the sintering of the samples, hence, the major factor that affect the rate of shrinkage is the starting composition of additives in the mixture. The result obtained shows an increasing pattern of linear shrinkage with the starting composition of sintering additives in the mixture. The additives is inferred to diffused into the available pores in the sample and hence promote shrinkage.

4.2 Hardness

The hardness values of each samples are listed in the following Table 4.2 and Figure 4.1. All samples were able to be indented and produced acceptable diagonal impressions as describe in the ASTM C1327. However, all samples did not produce sufficient cracks with the maximum load value of 1 kgf force and thus, the fracture toughness values were unable to be calculated.

TABLE 4.2: Vickers hardness result of samples with different additives composition.

Sample	Additives (wt.%)	Hardness (GPa)
1	5	24.7
2	10	24.9
3	15	25.1
4	25	25.5

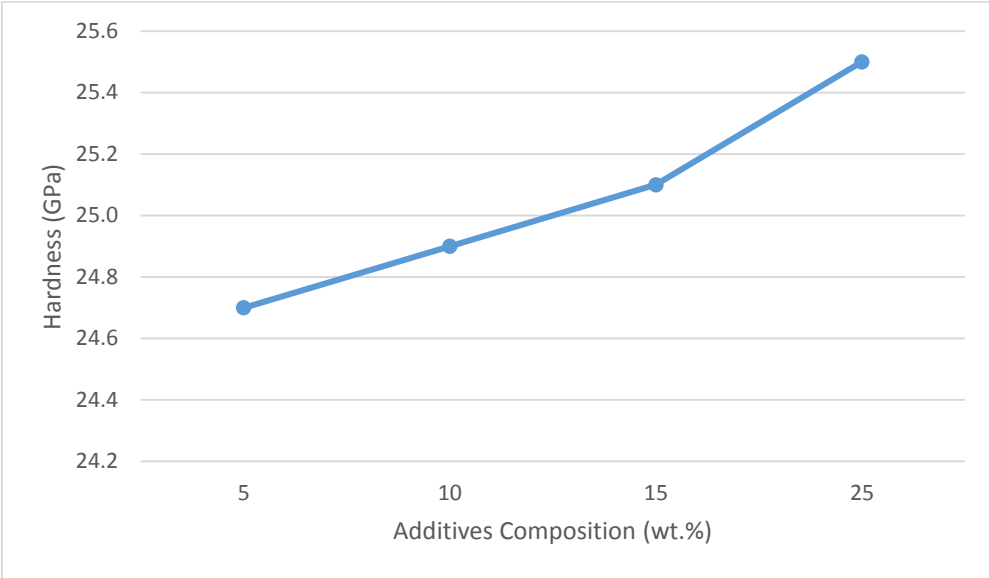


FIGURE 4.1: Vickers hardness result of samples with different additives composition.

Micro indentation results of Vickers Hardness for all samples with the oxide additives are quite similar as the value ranged from 24.7 to 25.5 GPa. Sample 4 with the highest content of additives of 25 wt. % has the highest hardness value of 25.5 GPa. The measured values were all lower than the published hardness of commercial SiC which is 27.5 GPa (2800 kg/mm^2). This lack of hardness can be attributed to the factor such as the formation of porosity in the sintered samples.

4.3 Density Measurement

The Archimedes' density and percent theoretical density for all four samples are listed in Table 4.3 and Figure 4.2 below.

TABLE 4.3: Archimedes' density of samples with different additives composition.

Sample	Additives (wt. %)	Density (g/cm^3)	Theoretical Density (%)
1	5	2.95	90.2
2	10	3.01	90.6
3	15	3.08	91.1
4	25	3.19	91.7

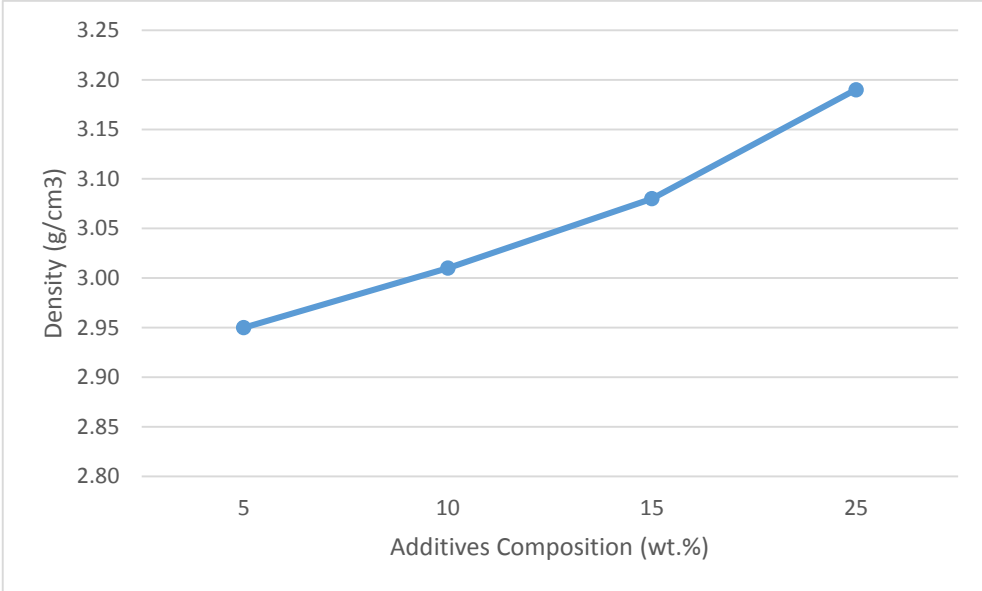


FIGURE 4.2: Archimedes' density results of samples with different additives composition.

As been expected, sample 4 with the highest additives content has the highest density value of 3.19 g/cm^3 with 91.7 % TD while sample 1 with the lowest additives content yielded a density value of 2.95 g/cm^3 with 90.2 % TD. The results can be explained through Figure 4.3 where it is observed that final density of the samples are very associated with the percentage of porosity formatted after sintering process has been completed. The higher the porosity percentage, hence can resulted in a lower density measurement. However, all density of the samples measured are slightly less than the published value of SiC density which is 3.21 g/cm^3. It can be assumed that all samples have achieved high densification as all theoretical density percentage calculated are higher than 90 % TD.

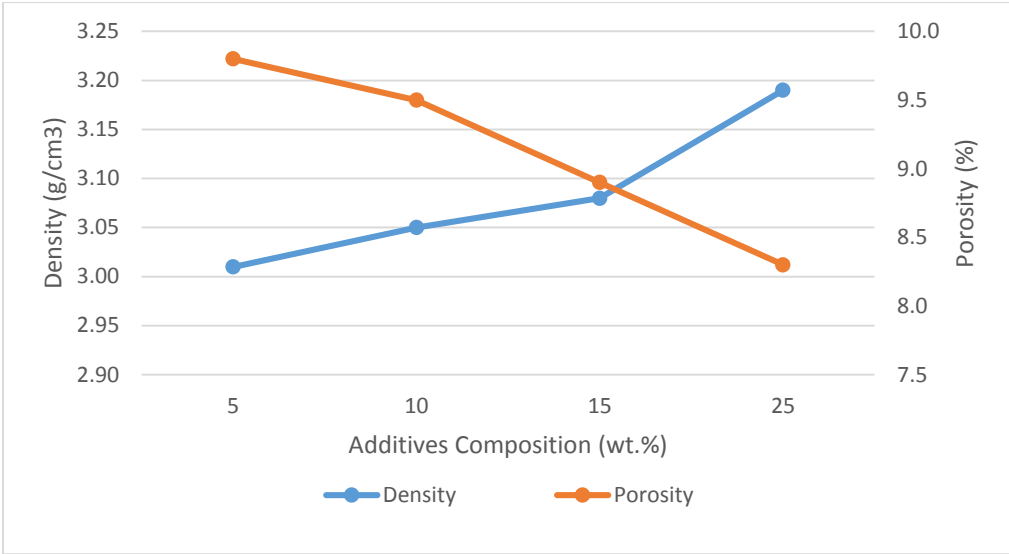


FIGURE 4.3: Graph of density and percentage of porosity of all four samples

4.5 Comparison of Density and Hardness

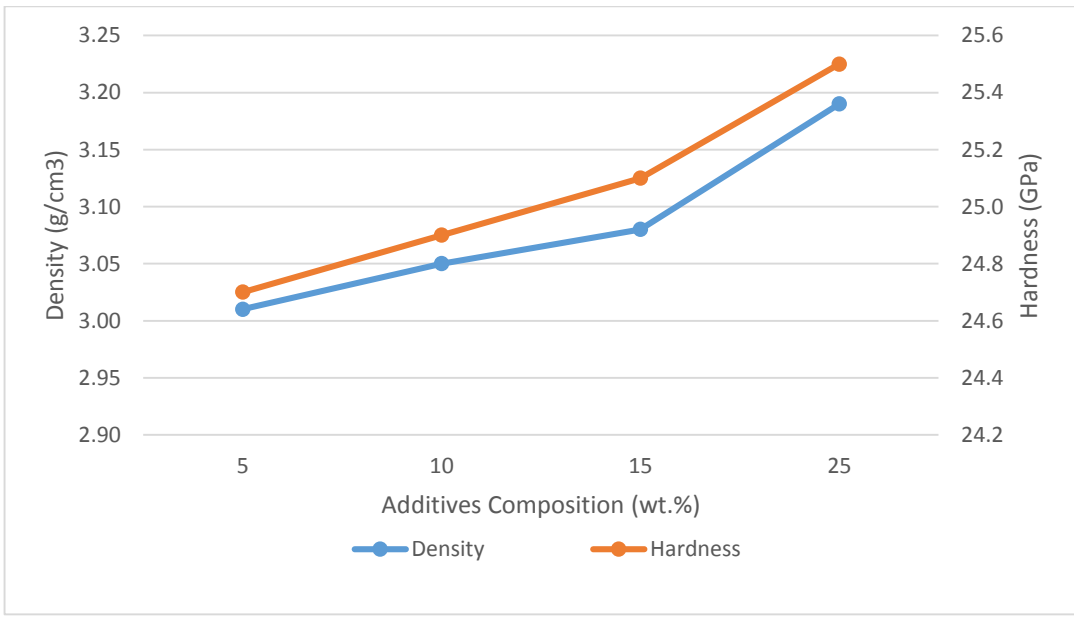
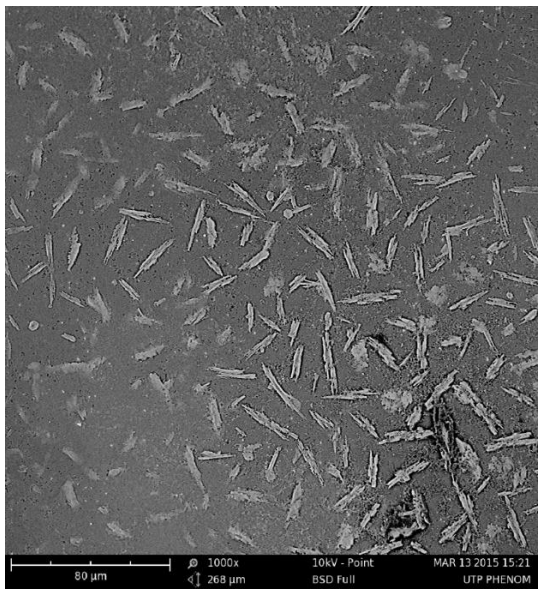


FIGURE 4.4: Graph of density and hardness for all samples showing the correlation between microstructure and mechanical property.

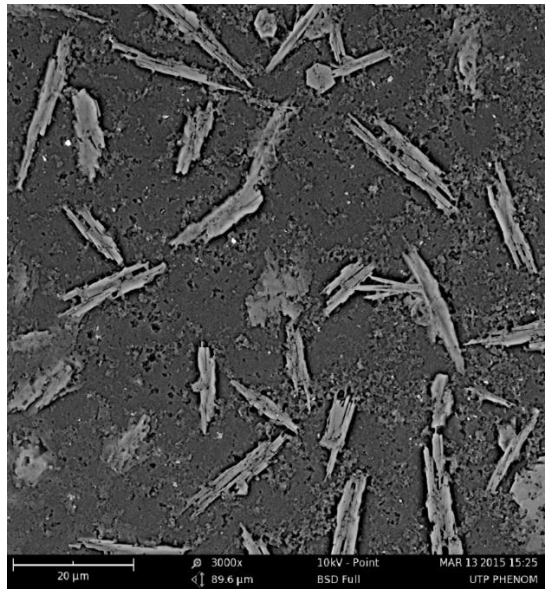
The graph in Figure 4.4 indicated that the lowest density sample also has the lowest hardness and vice versa. The composition of sintering additives used may have influenced the overall hardness of the sample together with its density. This can be explained as a more dense sample is usually more difficult to be indented and hence, resulting in higher hardness value.

4.5 Microstructure Analysis

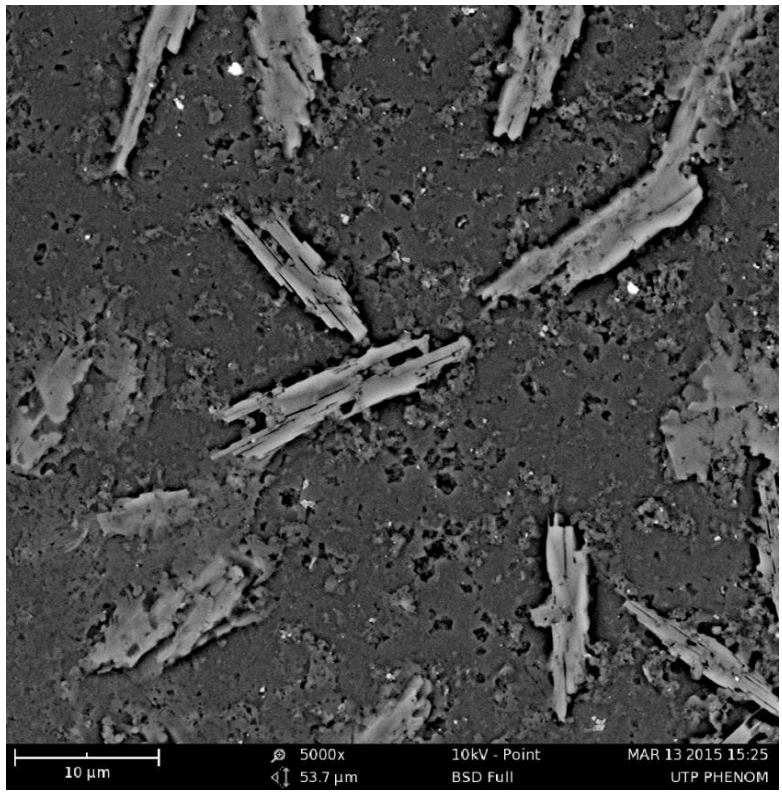
Electron micrographs for sample 4 were produced by using Scanning Electron Microscopy on the surface of the polished specimen.



(a)



(b)



(c)

FIGURE 4.5: Microstructures of sample 4 containing SiC particles with aluminium oxides and yttrium oxides as additives at sintering temperature of 1930˚C. (a) 1000x resolution. (b) 3000x resolution. (c) 5000x resolution

Figure 4.5 shows SEM micrographs of polished surface of sample 4 after sintering in various resolution. From the micrograph Figure 4.5 (c), the arrangement and formation of pores on the sample surface can be clearly visible. The pores are seen as dark regions as there is no backscattering of electron occurred. The phase in grey are classified as the SiC while the light regions are consisted of the sintering additives, which are alumina and yttrium. Sintering additives are visible as almost white because it contains heavier atoms than SiC and hence, backscattering more electrons. From the microstructure obtained, it reveals that the specimen contain very dense SiC with evenly distributed sintering additives. EDS analysis were also conducted on the specimen, in which the two spots chosen are illustrated in the Figure 4.6 below. From the analysis, it confirmed that vital element such as silicon, aluminium, yttrium and oxygen are presence in the sample and its concentration are plotted in the following Figure 4.7 and 4.8. The mapping analysis of specimen 4 are presented in Figure 4.9 where the distribution of elements in the sample can be observed.

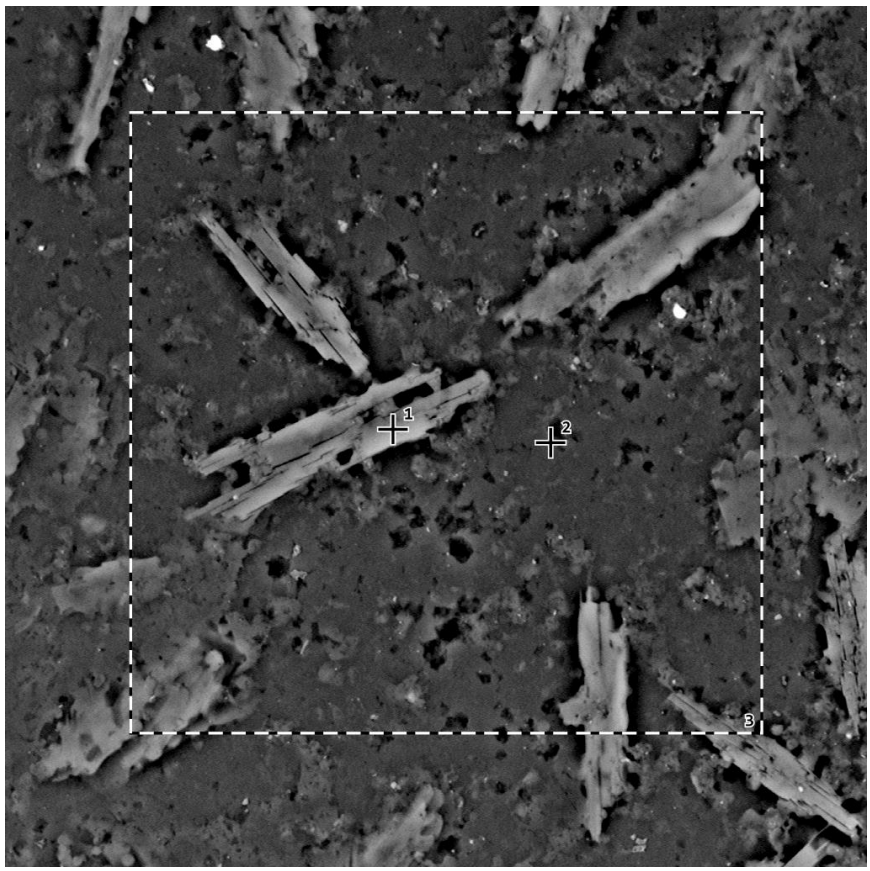


FIGURE 4.6: Selected area of specimen 4 for spot elemental analysis denoted as spot 1 and 2 respectively.

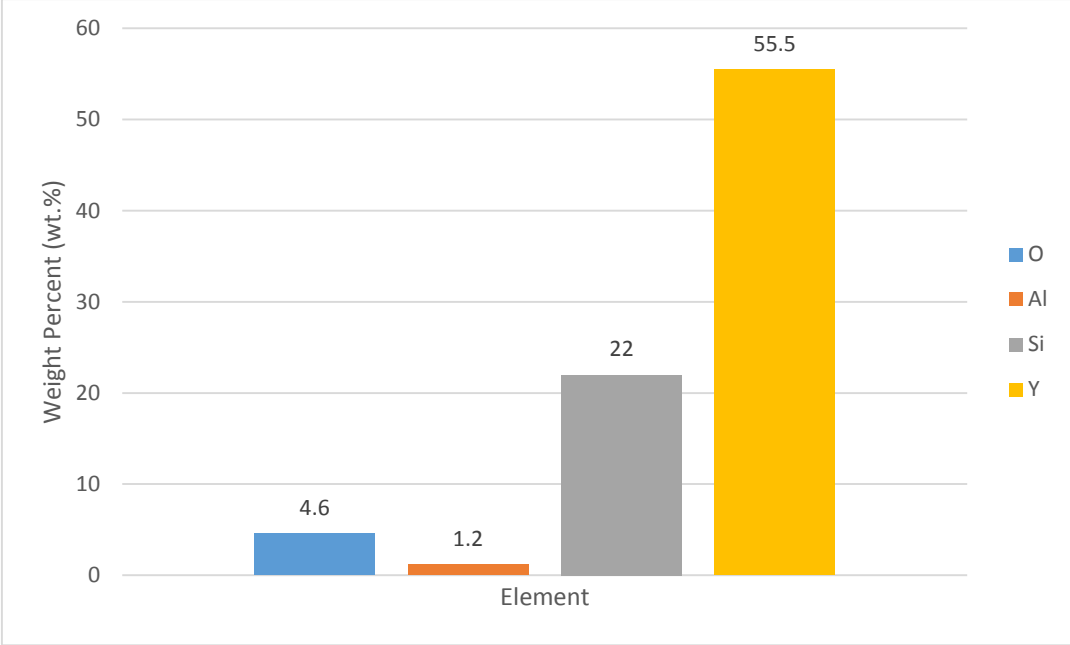


FIGURE 4.7: Elemental weight percent of spot 1 for specimen 4

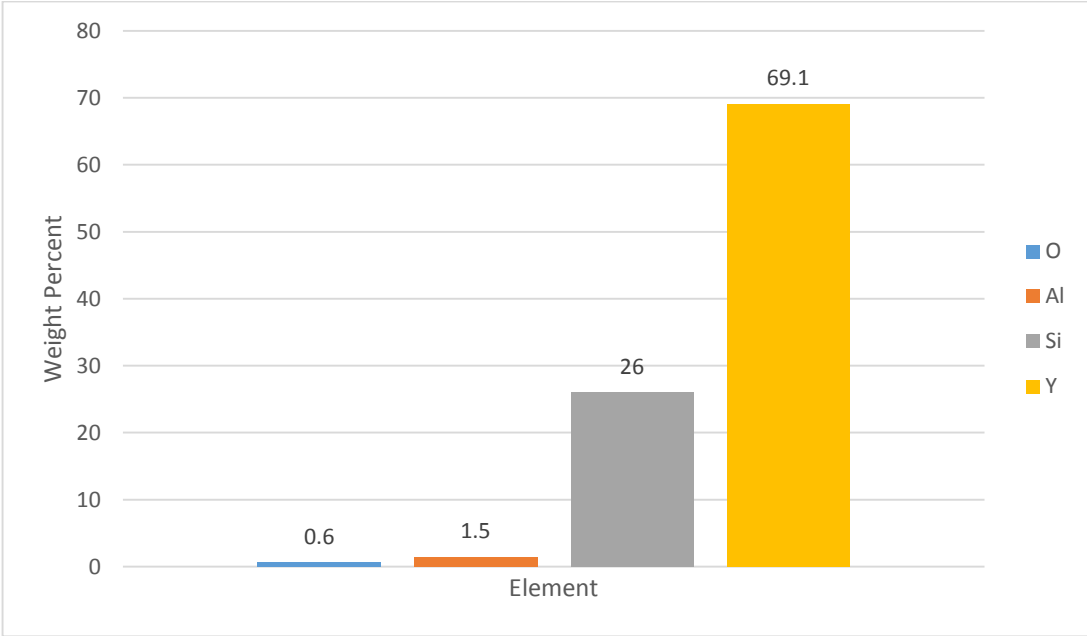


FIGURE 4.8: Elemental weight percent of spot2 for specimen 4

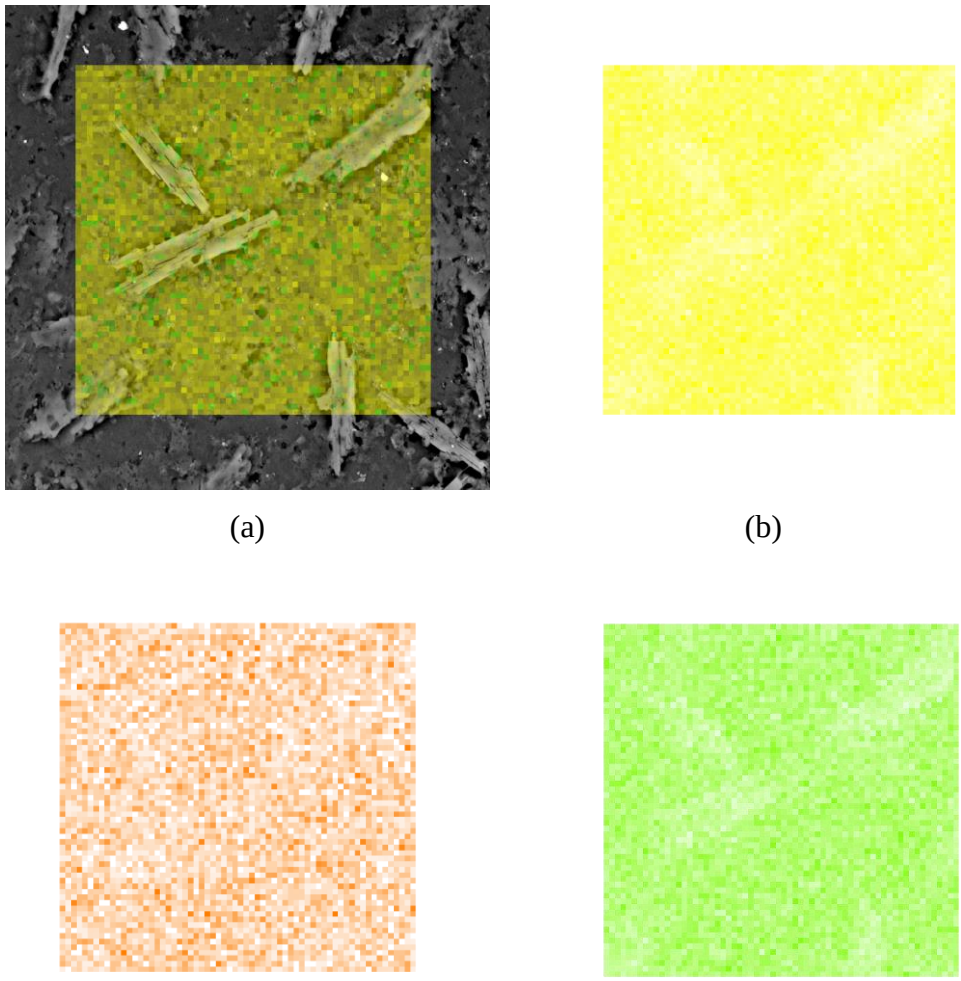


FIGURE 4.9: (a) Mapping analysis of selected area of specimen 4 and distribution of element of (b) silicon (c) aluminium (d) yttrium

CHAPTER 5

CONCLUSION AND RECOMMENDATION

It was shown that SiC with combination of sintering additives were successfully densified through solid-state sintering at the temperature of 1930˚C. From the results of experimental works, it is observed that the sample with the highest additives content exhibited the highest density and hardness value compared to others. The highest density and hardness value obtained were 3.19 g/cm^3 and 25.5 GPa respectively. It can also be concluded higher additives content could facilitate the densification rate during sintering process which will lead to higher mechanical and physical properties value of the produced sample.

For future works, the author would like to suggest few recommendations in order to improve the study that has been conducted. The recommendations are as the following:

1. The densification of specimen can be further improved by considering parameters such as starting powder size, sintering temperature, and type of sintering additives.
2. The effect of different type of sintering additives on the physical and mechanical properties of the final specimen should be investigated.
3. Include additional mechanical testing such as three point bending test.

REFERENCES

A.H. Rashed. (2002). “Properties and Characteristics of Silicon Carbide.” Poco Graphite Inc. Retrieved from http://www.poco.com/Portals/0/Properties.and.Characteristics.of.Silicon.Carbide.pdf

ASTM B962-14, Standard Test Methods for Density of Compacted or Sintered Powder Metallurgy (PM) Products Using Archimedes’ Principle, ASTM International, West Conshohocken, PA, 2014, www.astm.org

ASTM C1327-08, Standard Test Method for Vickers Indentation Hardness of Advanced Ceramics, ASTM International, West Conshohocken, PA, 2008, www.astm.org

Carlson, T. A. (2013). “Processing, Microstructure, and Properties of Carbon Nanotube and Silicon Carbide Composites.” Retrieved from https://www.ideals.illinois.edu/bitstream/handle/2142/44142/Thomas_Carlson.pdf?sequence=1

Fluid Sealing Association (FSA). (January, 2006). “Sealing Sense.” Pumps and Systems: 64-66. Retrieved from http://www.fluidsealing.com/sealingsense/jan06.pdf

Fuentes, R. I. (1986). “Sintering of Silicon Carbide.” Massachusetts Institute of Technology. Dept. of Materials Science and Engineering. Retrieved from http://hdl.handle.net/1721.1/14208

Z. H. Huang, D. C. Jia, Y. Zhou, Y. J. Wang. (2002). "Effect of a new additive on mechanical properties of hot-pressed silicon carbide ceramics." Materials Research Bulletin 37(5): 933-940.

Huebner, M. (2005). “Material Selection for Mechanical Seals.” Proceedings of the Twenty-second International Pump Users Symposium: 127-135.

Ihle, J., Hermann, M., Adler. J. (2005). "Phase formation in porous liquid phase sintered silicon carbide: Part III: Interaction between Al2O3–Y2O3 and SiC." Journal of the European Ceramic Society **25**(7): 1005-1013.

J. K. Lee, J. G. Park, E. G. Lee, D. S. Seo, Y. Hwang. (2002). "Effect of starting phase on microstructure and fracture toughness of hot-pressed silicon carbide." Materials Letters 57(1): 203-208.

Omori, M. & Takei, H. (1982). J. Am. Ceram. Soc., **65** c-92.

Prochazka, S. & Dondalski, W. J. (1978). U.S. Patent No. 4,108, 929. New York: U.S. Patent and Trademark Office.