SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING

UNIVERSITI SAINS MALAYSIA

SYNTHESIS OF POLYCRYSTALLINE SILICON CARBIDE USING ALUMINUM CARBIDE AND CALCIUM CARBIDE WITH SILICON TETRACHLORIDE

By

MUHAMMAD HAKIMI BIN RUZLAN

Supervisor: Dr. Sheikh Abdul Rezan bin Sheikh Abdul Hamid

Co-Supervisor: Prof. Dr. Ir. Cheong Kuan Yew and Dr. Arjuna Marzuki

Dissertation submitted in partial fulfilment

of the requirements for the degree of Bachelor of Engineering with Honours

(Materials Engineering)

Universiti Sains Malaysia

JUNE 2018

DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitled "Synthesis of Polycrystalline Silicon Carbide Using Aluminium Carbide and Calcium Carbide with Silicon Tetrachloride'. I also declare that it has not been previously submitted for the award of any degree or Diploma or other similar title of this for any other examining body or University.

Name of Student: Muhammad Hakimi bin Ruzlan

Signature:

Date:

Witnessed by

Supervisor: Dr. Sheikh Abdul Rezan bin Sheikh Abdul Hamid   **Signature:**

Co-supervisor: Prof. Dr. Ir. Cheong Kuan Yew and Dr. Arjuna Marzuki

Date:

ACKNOWLEDGEMENTS

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LIST OF ABBREVIATION

XRD	X-ray Diffraction
FESEM-EDX	Field Emission Scanning Electron Microscopy-Energy Dispersive X-ray
ICP-OES	Inductive Coupled Plasma-Optical Emission Spectrometry
FTIR	Fourier Transform Infrared Spectroscopy
I-V	Current-Voltage
PVT	Physical Vapour Transport
NPs	Nanoparticles
CVD	Chemical Vapour Deposition
p-Si	p-type Silicon
DOE	Design of Experiment
UV	Ultraviolet
MW	Microwave
MMC	multimode cavity
Eqn.	Equation
UT	Ultrasonic
1D	one dimensional
CCE	charge collection efficiency
γ-ray	gamma ray
e-h	electron-hole
e	electron
IR	Infrared
RF	radio wave frequency
RT	room temperature

DI	deionised water
e-beam	electron beam
α-radiation	alpha-radiation
3D	Three dimensional
VLS	Vapour-liquid-solid
Co-60	Cobalt-60
CTE	Coefficient of Thermal Expansion

LIST OF SYMBOLS

C	degree Celsius
°C/min	degree Celsius per minute
≥	more than or equals to
eV	electron Volt
K	Kelvin
Eg	band gap energy
MV/cm	Mega Volt per centimetre
W/mK	Watt per meter Kelvin
ml/min	millilitre per minute
Ea	activation energy
kJ/mol	kilo joule per mol
kPa	kilo Pascal
cm3/min	cubic centimetre per minute
ɣ	gamma
keV	kilo electron Volt
nm	nanometre
g	gram
ml	millilitre
Hz	Hertz
Rpm	revolution per minute
Mm	millimetre
Kgf	kilogram-force
mA	milli Ampere
Å/s	Armstrong per second

kV	kilo Volt
α	alpha
cm	centimetre
kJ	kilo Joule
wt.%	weight percentage
mg/L	milligram per liter
MeV	mega electron volt

SINTESIS POLIKRISTAL KARBIDA SILIKON MENGGUNAKAN KARBIDA ALUMINIUM DAN KALSIUM KARBIDA DENGAN SILIKON TETRAKLORIDE

ABSTRAK

Dalam kajian ini, sintesis karbida silikon polykristal yang tinggi (SiC) pada suhu $400\,°\,C$ hingga $600\,°\,C$ dan keberkesanannya dalam sistem dosimetri sinaran adalah dua kebimbangan utama dan disiasat. Sintesis SiC dimulakan dari bahan mentah aluminium karbida (Al_4C_3), Kalsium Karbide, CaC_2 dan silikon tetrakloride ($SiCl_4$). Al_4C_3 disintesis daripada serbuk aluminium (Al) serbuk dan grafit (C) dengan kaedah pencampuran basah. Al_4C_3 didepositkan pada wafer Si jenis-p menggunakan teknik tampal karbon. Si wafer didepositkan bertindak balas dengan $SiCl_4$ yang mengalir dalam tiub kuarza dengan kombinasi $400\,°\,C$ hingga $600\,°\,C$ selama 1 jam hingga 3 jam dengan kadar aliran gas dari 100 cc / min hingga 300cc / min. Kesan parameter ini pada pembentukan SiC dicirikan oleh pembiasan sinar-X (XRD), Mikroscop Pembiasan Sinar-X Mengesan-Tenaga Elektron (FESEM-EDX), Induktif Spektroskopi Pelepasan Plasma-Optik (ICP-OES) , dan Spectroskopi Penukaran Fourier Inframerah (FTIR). Puncak SiO_2 pada $70\,°$ XRD yang boleh dilihat dalam reka bentuk eksperimen (DOE) Run 4 (kadar reaksi: $500\,°\,C$, masa reaksi: 2 jam, kadar aliran gas: 200 cc / min), manakala regangan ikatan Si-C diperhatikan sekitar 800cm-1 dalam semua run DOE, regangan maksimum Si-C dalam Run 7 (kadar tindak balas: $600\,°\,C$, masa tindak balas: 3 jam, kadar aliran gas: 300 cc / min) berkaitan dengan data FTIR. FESEM-EDX menunjukkan komposisi unsur Si, C, Al dan O, mencadangkan bahawa pengoksidaan berlaku sama sekali dengan proses klorin. Si yang mempersembahkan ICP-OES telah membuktikan proses pengurangan karbohidrat berlaku tetapi prosesnya kurang bereaksi dengan $SiCl_4$. Kesimpulannya, SiC tidak boleh dibuat.

SYNTHESIS OF POLYCRYSTALLINE SILICON CARBIDE USING ALUMINUM CARBIDE AND CALCIUM CARBIDE WITH SILICON TETRACHLORIDE

ABSTRACT

In this research, the synthesis of high purity polycrystalline silicon carbide (SiC) at low temperatures of 400, 500 and 600 °C was investigated, and its efficiency in formation of SiC used in radiation dosimetry system which is main concerns nowadays and was investigated. The synthesis of SiC was initiated from raw material of aluminium carbide (Al_4C_3), Calcium Carbide, CaC_2 and Silicon Tetrachloride ($SiCl_4$). Al_4C_3 was synthesized from aluminium (Al) powder and graphite (C) powder by wet mixing method. Al_4C_3 was deposited on p-type Si wafer using carbon paste technique. The deposited Si wafer was reacted with flowing $SiCl_4$ in quartz tube with combinations of 400 °C to 600 °C for 1 hour to 3 hours with gas flow rate from 100 cc/min to 300 cc/min. The effects of these parameters on the formation of SiC were characterized by X-ray Diffraction (XRD), Field Emission Scanning Electron Microscopy-Energy Dispersive X-ray (FESEM-EDX), Inductive Coupled Plasma-Optical Emission Spectroscopy (ICP-OES), and Fourier Transform Infrared Spectroscopy (FTIR). SiO_2 peak at 70° in XRD which can be observed in design of experiment (DOE) at reaction rate of 500° C, reaction time of 2 hours and gas flow rate of 200 cc/min , whereas Si-C bond stretching was observed at around $800cm^{-1}$ in all DOE runs, the maximum stretching Si-C in Run 7 (reaction rate: 600° C, reaction time: 3 hours, gas flow rate: 300 cc/min) with regards to FTIR data. FESEM-EDX showed elemental composition of Si, C, Al and O, suggested that oxidation happened altogether with chlorination process. Si presented ICP-OES has proved the carbothermal reduction process took place but the process was less reacted with $SiCl_4$. Therefore, SiC not successfully formed, however, partially formation of SiC was observed in SEM/EDX analysis.

CHAPTER 1

INTRODUCTION

1.1   Background

In this project, the synthesis of SiC at low temperatures was studied by using two-step process; (a) preparation of thin layers of Aluminium Carbide (Al_4C_3) on the Silicon (Si) wafer and the (b) chlorination of the Aluminium Carbide using $SiCl_4$ to form SiC thin layer. For the preparation of thin layers, this thesis use two step, one was use electron beam evaporator (e-beam) and another step is using carbon paste, which adding and mix together with Al_4C_3 with some powder of Calcium Carbide (CaC_2) as a catalyst. But the main process in this thesis is using carbon paste. For the chlorination, the main solution in this chlorination is Silicon tetrachloride ($SiCl_4$) which in liquid form. This $SiCl_4$ will change to gas and flow along quartz tube and react with carbon paste with Al_4C_3 to form SiC layer. The synthesis methods of Silicon Carbide layer was reviewed from the literature and they were carbothermal reduction, magnesiothermic, pyrolysis of polymeric precursors, and synthesis of solid- gas reaction

The silicon carbide powder made from chemical vapour deposition and sol–gel method has high purity and narrow particle size distribution. But the reactants are unstable and toxic and the production cost is high (Lin and Chuang, 2007). Direct carbonization method is simple and cheap, but it will leave significant amounts of unreacted silicon and impurities. So far, the carbothermal reduction method is the most prevailing method for SiC powder synthesis (Lin and Chuang, 2007). . It not only gives high purity b-SiC powder but also involves inexpensive starting materials (e.g., silicon dioxide, rice husk, graphite and charcoal)

Although the carbothermal reduction method only requires cheap reactants and could have good products, it is usually performed at 1600 ºC or above in order to have high yield of b- SiC (Lin and Chuang, 2007) . Because of low temperatures, the yield of SiC is usually low (Lin and Chuang, 2007).

According to Gubernat et al. (2017) feasibility of the SiC synthesis using shungite which is a type of silicon rock has been proven. Advantages of the synthesis must mainly refer to the low temperature – 1000 °C – as the Acheson method used on the industrial scale requires temperatures exceeding 2500°C (Gubernat et al., 2017). It is worth noticing that in case of powder and pellets the products are also in powder form or need small forces to disintegrate samples. Obtained products can be simply applied as abrasive or polishing powders. However, one of the disadvantages of the synthesis, there is variability of the chemical composition in the shungite. Therefore, it is necessary to provide uniformity of the bed batch before synthesis and – if required – the correction of composition is easy to perform by using additives e.g. carbon(Gubernat et al., 2017).

Silicon carbide (SiC) has a number of advantages over silicon, among them a wider energy bandgap that enables devices to run at temperatures as high as 600 °C; a breakdown voltage about 10 times that of silicon, making the material robust enough to withstand very high voltages; and high thermal conductivity, allowing it to handle enormous amounts of power. All those properties make SiC especially attractive when engineers need to design systems that efficiently convert AC into DC, regulate voltages, and control motors. (Boyd, 2013).

Rail systems are not the only application for which Mitsubishi is using silicon carbide. It has introduced an air-conditioner model with a SiC inverter that reduces power consumption, and also several motors for industrial use that employ SiC controllers. In addition, it has begun testing an inverter for elevators and is developing inverters for electric vehicles. Mitsubishi announced a prototype for large-capacity all-SiC power switch rated at 1200 volts and 1200 amperes; the company claims it provides the highest performance in the industry. The switch achieves a 75 percent reduction in power loss, which means it needs only half the cooling equipment usually required, Mitsubishi says. The target applications are factory automation equipment, elevators and escalators, and solar-farm and wind-farm control systems (Boyd, 2013).

Because of the potential advancement of SiC in electronic application, particularly in unforgiving condition, the synthesis technique is has been widely researched on and enhanced from the parts of temperate and huge clump generation. Ordinary amalgamation of SiC includes high temperature and costly crude materials which expands the vitality of utilization. The work to examine a low temperature creation of SiC is still being developed and accordingly, synthesis of SiC at low temperature was the fundamental approach in this project.

The synthesis of SiC is possible in relatively in range of 1500–1600 ∘C (Gubernat et al., 2017). It is worth emphasising that compared to the most popular method of SiC synthesis for example, the Acheson method where the temperature of synthesis is about 2500∘C the proposed method is much more effective (Gubernat et al., 2017). The process of silicon carbide formation was proposed and discussed. In the case of synthesis SiC from powder of raw materials the product is also in powder form and not requires any additional process such as crushing and milling (Gubernat et al., 2017). Obtained products

are pure and after grain classification may be used as abrasive and polishing powders (Gubernat et al., 2017).

In this research, low-temperature synthesis of high immaculateness polycrystalline SiC utilizing $SiCl_4$ and Al_4C_3 with impetus saved on p-type Si (p-Si) wafer was contemplated utilizing outline of trial (DOE) in which Al_4C_3 was incorporated for benefit in this investigation. The parameters to be considered in this exploration were the chlorination temperature between $SiCl_4$ and Al_4C_3, and soaking time for the reaction between $SiCl_4$ and Al_4C_3 and flow rate of Argon (Ar) as carrier gas of $SiCl_4(g)$. It was trusted the orchestrated SiC can be utilized as a semiconductor detector for atomic radiation.

In the present study, the authors focused on silicon carbide supports for the following reasons. Silicon carbide supports exhibit good thermal shock tolerance, high-temperature catalytic activity, good anti-fouling properties resulting from high hydrophilicity, and good abrasion resistance (Ha et al., 2017). Moreover, silicon carbide supports can be prepared from inexpensive raw materials which are silica and carbon as starting materials (Jamil N. et al., 2014). The average pore size of an uncoated silicon carbide support is limited by the large particle sizes and irregular particle shapes of silicon carbide (Ha et al., 2017).

Wide band gap materials such as diamond like carbon, III–nitride, and silicon carbide have been widely applied in numerous electronic devices, where hostile conditions including high power, high temperature exist (Phan et al., 2017). These materials have also been employed in MEMS applications which aim at high frequency and high temperature sensing transducers (Phan et al., 2017). Among these materials, silicon carbide is a preferable choice owing to its excellent physical properties along with the availability of wafers (Phan et al., 2017). Nevertheless, the main obstacles which

hinder the wide applications of SiC are the high cost of wafer and low etching rate of SiC in comparison to Si (Phan et al., 2017).

Silicon carbide (SiC) is a wide band gap semiconductor (E_g for α phase: 3.2 eV, for β phase: 2.2 eV) that has drawn active attention of recent workers for its improved functionalization property over carbon (C) due to existence of multiple-bilayer wall structure on surface which allows surface Si atoms to functionalize more readily with molecules (Nayak et al., 2018). The compound can withstand high temperatures, up to 1000 °C, in air because SiC intrinsically is a wide band gap semiconductor (Nayak et al., 2018).

Silicon carbide combines a number of unique characteristics, namely high mechanic properties within a broad scope of temperatures, resistance to the effects of acids and alkalis, as well as distinctive electrical properties(Petrus et al., 2018). These properties may be ascribed to strong and stiff Si–C covalent bonds, which cause tremendous problems in obtaining dense sinters due to a low self-diffusion coefficient (Petrus et al., 2018). Therefore, sintering of SiC requires applying additions facilitating the process. Dense sinters may be obtained by liquid-phase sintering with the use of oxide additions (alumina, zirconia, yttria) and by solid state sintering, with the additions of boron and carbon(Petrus et al., 2018). The use of oxide additions allows to significantly lower the temperature of sintering and increase the relative density of the sinters(Petrus et al., 2018).

Nevertheless, it also contributes to a decrease of hardness and thermal stability due to the presence of glassy phase on the grain boundaries(Petrus et al., 2018). The use of boron and carbon requires higher temperatures of sintering, yet it allows to acquire sinters characterized with high hardness and relative density similar to theoretical (Petrus

et al., 2018). Numerous polymorphic structures constitute another difficulty in obtaining a dense SiC sinter. These structures may be categorized into two phases: β phase and α phase (Petrus et al., 2018). The β phase, in spite of being a primal phase, forming during SiC synthesis, is metastable at any temperature, as it undergoes a transition into the α phase. A number of polytypes may be distinguished in the α phase, i.e. 6H, 4H and 2H (Petrus et al., 2018). These varieties differ only in the sequence of composition of the double atomic layers. The degree and the pace of transition are subjects to numerous factors (Petrus et al., 2018). By means of example, the transformation rate increases with the increase of annealing time and decreases in the presence of nitrogen (Petrus et al., 2018).

1.2   Problem Statement

In this study, SiC needs to be produced in low temperature instead of high temperature processing methods.. The high temperature will cause high costs. SiC as radiation dosimeter can detect radiation such as radiation that from alpha-ray and betta-ray can be detected by using SiC in harsh environment. Some precaution and steps were taken in order to make SiC production more feasible. Some of the techniques were low temperature production using appropriate raw materials and methods such as physical vapour deposition and subsequent low-temperature chlorination in the range of 400-600 °C. $SiCl_4$ reacts with aluminium carbide (Al_4C_3) pellet on silicon wafer in the presence of CaC_2 as catalyst to form SiC which was the main purpose in this project. This make the synthesis of radiation dosimeter will become more applicable.

SiC synthesis, is stable at any temperature, as it undergoes a transition into the α phase. A number of polytypes may be distinguished in the α phase, i.e. 6H, 4H and 2H (Yi et al., 2017). These varieties differ only in the sequence of composition of the double atomic layers. The degree and the pace of transition are subjects to numerous factors (Yi et al., 2017). By means of example, the transformation rate increases with the increase of annealing time and decreases in the presence of nitrogen (Yi et al., 2017). The 6H type is stabilized by boron, whereas the presence of aluminium favours the creation of the 2H variety (Yi et al., 2017).

Detectors based on Silicon and Silicon Carbide are employed to monitor and characterize different sources of ionizing radiations, such as laser-generated plasmas, UV and radioactive sources (Cannavò et al., 2017). Surface and depth active depletion zones are employed to detect low and high energetic particles and soft and hard X-ray emission, depending on the detection efficiency of each device (Cannavò et al., 2017). The different gap, geometry and leakage currents characterize the response of the detectors (Cannavò et al., 2017).

Silicon carbide (SiC) detectors are widely used in radiation detection of harsh environment and high temperature due to the superior resistance and stability of SiC material at high temperature and intense radiation (Liu et al., 2017). In recent years, many epitaxial SiC-based Schottky and p-i-n junction detectors have been developed for alpha particles detection, X-ray monitoring and neutron detection, and their potential applications in high energy physics experiments, nuclear reactors and deep space exploration have been found for their favourable properties such as a thickness up to 100 μm, a charge collection efficiency nearly 100% , an energy resolution about 0.3%–0.5% to alpha particles, a radiation resistance nearly three orders of magnitude higher than

silicon detectors, (Liu et al., 2017). Most of the researches related to the detection of charged particles were focused on alpha particles and protons, few reports were on heave ions (Liu et al., 2017).

1.3   Objectives

There are one objectives in this research as listed in the following:

a. To synthesize high purity polycrystalline SiC using Aluminum Carbide, Al_4C_3 and Calcium Carbide, CaC_2 as catalyst with silicon tetrachloride $SiCl_4$ at lower temperatures below 600 ℃

1.4   Research Approach

This research can be divided into two steps. The first step was the synthesis of thin layer of Aluminium Carbide (Al_4C_3) and in this project, Al_4C_3 powder was used in the EBM and coating method. The as-received powder and synthesized layers were characterized by using XRD, XRF, and SEM/EDX. The deposition of Al_4C_3 on the p-type silicon (p- Si) wafer was performed by applying electron beam evaporation method and also with carbon paste. The interaction between Al_4C_3 and p-Si wafer, thickness of as-coated Al_4C_3 layer was determined by using XRD and FESEM-EDX.

The next step of this research was the reaction between Silicon Tetrachloride ($SiCl_4$) and Al_4C_3 being studied. The parameters to be controlled and evaluated in this work were the reaction temperature and time between $SiCl_4$ and Al_4C_3 and the flow rate of Ar gas as carrier gas in the process.

The reaction temperature was controlled at 400 °C, 500 °C and 600 °C whereas the reaction time was set at 1 hour, 2 hours and 3 hours. The flow rate was also between 200-400 cc/min and it was controlled by flow meter during the process. The interaction in between parameters was evaluated by using Design of Expert (DOE) software. The fabricated SiC thin layers were characterized with XRD, FESEM-EDX, ICP-OES and FTIR while the commercial SiC Schottky diode was characterized for its electrical property before and after α- and ɣ-radiation in the aspect of I-V performance.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter mostly focuses on synthesis of silicon carbide (SiC) layer on Silicon wafer. The synthesis methods of Silicon Carbide layer were reviewed from the literature and they were carbothermal reduction, magnesiothermic, pyrolysis of polymeric precursors, and synthesis of solid- gas reaction. Some of the applications of Silicon Carbide also were explained in this chapter to give on idea of how the SiC is useful for industrial applications. The HSC software was used for thermodynamic evaluation of the formation reactions of SiC on silicon wafer by estimation of the Gibbs free energy and also the stability diagrams for the studied system was addressed in this chapter.

2.2 Properties of SiC

2.2.1 Physical and mechanical properties

The low-density ceramic of SiC has a variety of properties including as an abrasive material, wide bandgap semiconductors in high-power electronics, and structural ceramics in the automotive and aerospace industries. The most widely used approach for powder synthesis is known as the Acheson process: namely, a reduction of silica by carbon at an elevated temperature. This method allows the production of powders with different grades of purity and particle sizes >5 μm. Pure sub-micrometer SiC particles can be synthesized by CVD methods in silane/hydrocarbons systems, sol-gel processes, plasma, and microwave radiation route (Mukasyan, 2017).

Silicon carbide (SiC) is a wide gap semiconductor considered to be one of the major enabling materials for advanced high power and high temperature electronics applications. Silicon carbide has more than 250 polytypes, but only two polytypes with the maximal width of forbidden gap have real industrial applications (Parshin et al., 2017). They are 6H-SiC and 4H-SiC polytypes with alternating layers of cubic and hexagonal crystal structures and forbidden gaps of 3.03 eV and 3.26 eV, respectively (Parshin et al., 2017). A great amount of publications deals with electrical parameters investigation of different polytypes of silicon carbide and its application in high-temperature microelectronics. But up to now, the shortwave part of the millimetre (MM) range and Terahertz (THz) band of electromagnetic waves (100–3000 GHz) is still the "white spot" in the research of its dielectric parameters (Parshin et al., 2017). There are only a small number of publications in this field.

2.2.2 Chemical properties

For molecular weight SiC which is 40.096 g/mol, the hydrogen bond Acceptor Count and Complexity are 1 and 10 respectively. The monoisotopic mass and exact mass are 39.77 g/mol respectively. It has 2 heavy atom count and 1 covalently-Bonded Unit Count (www.pubchem..gov).

2.2.3 Electrical properties

For 4H-SiC, the bandgap energy, E_g is 3.26 eV. The relative dielectric constant and thermal conductivity are 9.7 and 4.9 W/cmK respectively. The saturated electron velocity of SiC is 2×10^7 cm/s (www.iue.tuwien.ac.at).

2.3 Applications of Silicon carbide (SiC)

Silicon carbide (SiC) and silicon carbide fiber-reinforced, silicon carbide matrix (SiC-SiC) composites exhibit desirable properties for use as structural components in nuclear reactors. The Department of Energy's Accident Tolerant Fuels (ATF) and Advanced Reactor Technologies (ART) programs aim to utilize materials like SiC-SiC composite to improve reactor safety, performance and fuel utilization. Enhanced reactor performance and fuel utilization can be achieved by increasing burn-up and fuel cycle length. However, achieving these goals with SiC-SiC necessitates development of suitable joining materials that can withstand the higher fluences anticipated in advanced light water reactors (ATF) and high temperature fast reactors like the Energy Multiplier Module (EM2). Furthermore, SiC joining technology is expected to enable the fabrication of more complex internal core components such as fuel rod supports structures (Khalifa et al., 2017).Based on figure 2.1, it show SiC can spread into various application which system integration such as energy, e-mobility, transport, industry and Information and Communication Technology (ICT) .
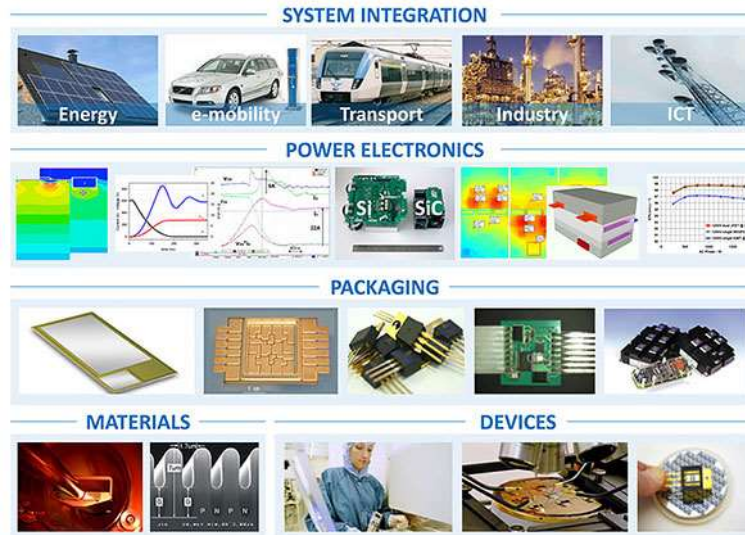
Figure 2.1   Applications of SiC

SiC is discussed as a future high performance replacement of the silicon power components. The new technology enables products with almost ideal behaviour. Currently, SiC Schottky rectifiers are already available as qualified standard products (www.vincotech.com). Silicon carbide (SiC) detectors are widely used in radiation detection of harsh environment and high temperature due to the superior resistance and stability of Sic material at high temperature and intense radiation (Liu et. al., 2017). Detectors based on Silicon and Silicon Carbide are employed to monitor and characterize different sources of ionizing radiations, such as laser-generated plasmas, UV and radioactive sources (Liu et. al., 2017).

The utilization of silicon carbide (SiC) semiconductor material has been on the expansion since the 1990's after a drive for hardware which could with-stand high temperatures and radiation measurements. In that capacity amazing, low imperfection material is presently monetarily accessible for applications, for example, high-voltage hardware, ultra-violet photodiodes and light producing diodes. In spite of the fact that the

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utilization of semi-protecting silicon carbide material for radiation recognition purposes has been beforehand illustrated, its utilization in pragmatic applications has been restrained by space charge security issues caused be imperfection fixations inside the material, the purported polarization impact, by which the check rate and resultant range changes with light time (Hodgson et al., 2017). In their paper, Hamilton et. al. (2017) stated that the first two generations of SiC MOSFET devices have been evaluated to obtain their electrical and thermo-mechanical performances at temperatures up to 350 °C. The target application for this is a high performance hybrid electric vehicle inverter whose junction temperature is allowed to increase up to a peak of approximately 300 °C for occasional short distances, such that the space and weight consuming cooling system does not have to be scaled up to meet this occasional demand (Hamilton et al., 2017). Although 350 °C is approximately 200 °C above the rated operating temperatures of current SiC MOSFET devices, initial static measurements showed good performance up to 350 °C, beyond which device failures were noted (Hamilton et al., 2017).

Silicon carbide has been attracted much attention in recent years because of its potential application in many kinds of optoelectronic devices, such as solar cells, images sensors, gas sensors and photodiodes , applications in optics (earth observation), micro-machined SiC optic fiber as pressure sensors for high-temperature aerospace applications and SiC-SiC composites optics for UV applications (Ouadfel et al., 2017). The silicon carbide (SiC) has become the focus of considerable attention due to its excellent material properties, promising it for different applications. Several reports on p-type SiC were published, but not much effort has been focused on a hot-pressed 6H-SiC material with granular structure or on different structure of crystalline or amorphous thin SiC films (Ouadfel et al., 2017). To date no works have been performed on low-doped

polycrystalline 6H-SiC and no chemical polishing solution has been developed on the material (Ouadfel et al., 2017).

Silicon carbide (SiC) is a wide gap semiconductor considered to be one of the major enabling materials for advanced high power and high temperature electronics applications. Silicon carbide has more than 250 polytypes, but only two polytypes with the maximal width of forbidden gap have real industrial applications (Parshin et al., 2017). They are 6H-SiC and 4H-SiC polytypes with alternating layers of cubic and hexagonal crystal structures and forbidden gaps of 3.03 eV and 3.26 eV, respectively (Parshin et al., 2017). A great amount of publications deals with electrical parameters investigation of different polytypes of silicon carbide and its application in high-temperature microelectronics. But up to now, the shortwave part of the millimetre (MM) range and Terahertz (THz) band of electromagnetic waves (100–3000 GHz) is still the "white spot" in the research of its dielectric parameters (Parshin et al., 2017).

There are only a small number of publications in this field. Silicon carbide materials, and solid state-sintered silicon carbide materials (SiC) in particular, have found widespread use as seals, bearings and valves in a variety of media in industrial wear applications(Striegler et al., 2018). SiC materials are used due to their good tribological properties and high corrosion resistance. Silicon-infiltrated SiC (Si-SiC) ceramics are also used for wear applications(Striegler et al., 2018). Especially large wear parts are produced from Si-SiC because these components can be produced without shrinkage during the reaction sintering. The materials are produced by infiltrating a SiC preform, which also contains a small amount of free carbon, with liquid silicon at a temperature of 1500–1700 °C (Striegler et al., 2018).

Infiltration occurs by capillary action, with the Si partially reacting with the free carbon. Due to this reaction, the silicon content can be reduced, but Si-SiC ceramics usually contain 5–20 vol.% of free silicon, which is less stable than SiC is at high temperatures and in solutions at high pH values (Striegler et al., 2018).This is mainly explained by inaccessibility of high-quality single-crystal samples, but more importantly, by the absence of real problems in this frequency range, for which silicon carbide would be a key material. CVD diamond was in a similar situation until it found its unique application place due to the complex of its unique properties, i.e., in barrier windows for the megawatt power microwaves in the International Thermonuclear Experimental Reactor (ITER) project (Parshin et al., 2017).

The situation with silicon carbide is somewhat similar. For SiC, there is a possible unique field of application as the material for input-output windows of the 50-500 kW power level (Parshin et al., 2017). Certainly, diamond due to its tremendous thermal conductivity (~ 2000 W/(m.K) and very low losses (tan δ ~ 10-5) will easily cope with this task, but diamond is ten times more expensive (Parshin et al., 2017).Researchers gradually turned their attention to wide bandgap semiconductor components, such as GaN, SiC and so forth. GaN is more suitable for high-voltage, high-power and high-temperature applications in high-frequency applications in low and medium voltage ranges compared to other major wide bandgap technologies such as GaN (Jie et al., 2017). The voltage limit for GaN and SiC is typically 600 V or 900 V (Jie et al., 2017).SiC is better than GaN in term of maturity and robustness (Jie et al., 2017) .

There are many interesting devices that can be produced by both materials (RFPD, 2018). Currently see GaN being used for lower power/voltage, high frequency

applications and SiC for high power and high voltage switching power applications (Figure 2.2).
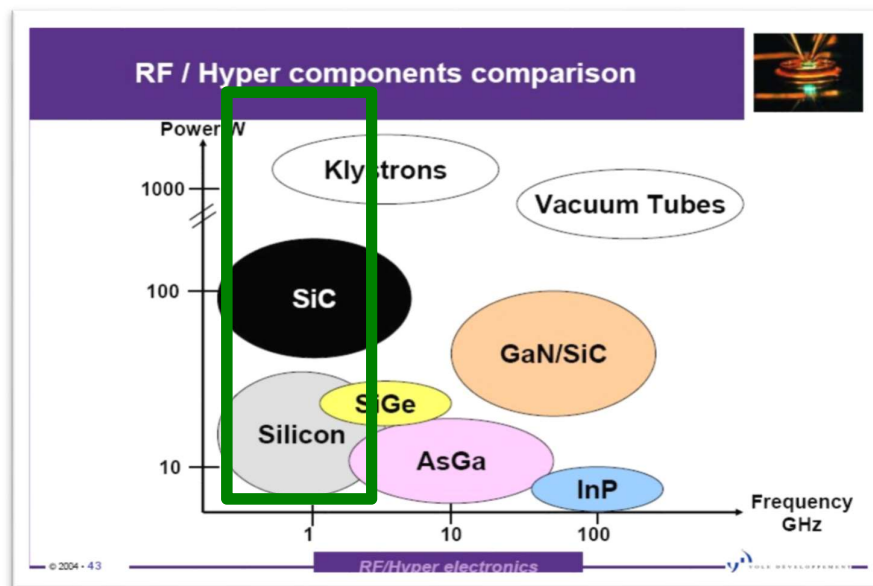
Figure 2.2   Semiconductor material power and frequency regions (RFPD, 2018).

SiC power device technology (devices and materials) is more mature than GaN; SiC required a lower upfront investment which was more commensurate with the size company we are while allowing us to utilize our existing infrastructure more easily ; path to profitable growth is shorter with SiC than GaN; SiC business can be used to fund a GaN effort downstream; SiC offers the possibility of producing MOSFETs, GaN does not(RFPD, 2018).

At present, SiC has been used in terrestrial photovoltaic inverter, power factor correction(PFC), hybrid electric vehicles, fans, rail locomotives, uninterruptible power

supply (UPS) and industrial motor drive (Jie et al., 2017).It is hard to compete with the superior performance of SiC for Si and GaAs in harsh environments, once its key technology is tackled, it will be more widely used in artificial satellites, rockets, missiles, radar, fighter, communications, marine exploration, oil drilling and other military and civilian systems (Jie et al., 2017). As a result, SiC has become a hot topic in the field of new materials, microelectronics, optoelectronics and energy research. The National Aeronautics and Space Administration (NASA), the European Space Agency (ESA), and the Japan Aerospace Research and Development Agency (JAXA) Power device space performance and power supply design and other series of research (Jie et al., 2017).Silicon carbide has a potential for sun oriented UV radiation observing: amazingly impervious to UV radiation harm, almost heedless to unmistakable and infrared radiation and less delicate to temperature varieties than standard radiometric frameworks. Two photodiodes are outfitted with filters in the UVB (280– 315 nm) and UVA (315– 400 nm) ranges while a third is filtered to coordinate the thermal activity range. UVA, UVB segments of the sun oriented radiation and UV record (UVI) at the world's surface have been resolved in two site positions in Tuscany, Italy(Borchi et al., 2011). Silicon carbide is characterized by having a high hardness, thermal conductivity, chemical resistance and thermal strength at elevated temperatures (Kaźmierczak-Bałata and Mazur, 2018). These properties are related to the strong directional chemical bonds in this material. However, low self-diffusion values for carbon and silicon, throughout the SiC structure, are observed during sintering process (Kaźmierczak-Bałata and Mazur, 2018).

Porous ceramic filters are an effective way to separate the dusts from the polluted air at high temperature. The filters can be made of Mullite, cordierite or SiC because of their excellent chemical stability and thermal shock resistance (Yuan et al., 2016). There are many methods to prepare porous SiC ceramics, such as foaming and ceramic precursor

(Yuan et al., 2016). However, bonding technique is usually adopted for the preparation of porous SiC ceramic filter (Yuan et al., 2016). Because ceramic filter should have high compressive strength, high porosity, large pore size and permeability. For bonding technique, Mullite, cordierite, zirconia and glass are commonly used as inorganic binders (Yuan et al., 2016). According to Yuan et al. (2016), bentonite, a mineral with the main composition of montmorillonite, is commonly used successfully in the preparation of reticulated porous SiC ceramics.

2.4   Synthesizing methods of SiC

2.4.1   Carbothermal reduction process

Recently, Al_4SiC_4 powder with a grain size of 100~200 μm were successfully prepared by carbothermal reduction method in previous works (Xing et al., 2018). In addition, it was reported that natural minerals such as Aluminum Oxide Al_2O_3 can be adopted as raw materials for carbothermal reduction method, which makes it draw more and more researchers' attention for further study and research(Xing et al., 2018). According to Fukushima et al. (2017) the   research conducted by (Ha et al., 2017) will focus on silicon carbide supports for the following reasons: (i)Silicon carbide supports exhibit good thermal shock tolerance, (ii) high-temperature catalytic activity , (iii) good anti-fouling properties resulting from high hydrophilicity, and (iv) good abrasion resistance .   However, only a few ordered mesoporous

SiC structures have been successfully fabricated because the growth and porous structure of SiC crystals are very difficult to be controlled during the high temperature synthesis process.. One way based on the carbothermal reduction of silica: the

carbonaceous materials like CaC_2 are filled into the channels of the templates to form SiO_2/C composites, and the composites are transformed to SiC via carbothermal reduction process. The SiC powders were rapidly synthesized by spark plasma-assisted carbothermal reduction (SPCR) at low temperature using quartz sand, carbon black and phenolic resin as raw materials. These carbothermal reductions process are always performed at very high temperature (~ 1400 °C) (Zhao et al., 2011).

Another is based on high temperature pyrolysis reaction, in this method, the pre-ceramic polymers, such as polycarbosilane, dimethyldichlorosilane, $Si(CH_3)_2Cl_2$ are infiltrated in mesoporous silica and then pyrolyzed to produce mesoporous SiC (Zhao et al., 2011). However, most of these methods still suffer from various drawbacks, but the findings obtained so far have provided important clues despite the advantages. The used approach depends on the desired product properties, such as purity, particle size distribution, morphology and yield, as well as cost considerations. The Acheson process is primary industrial method for producing SiC powder; this approach involves carbothermal reduction (CR) of quartz sand by coke at more than 2000 °C(Wang et al.,2017).

SiC products prepared by conventional carbothermal reduction reaction at high temperature present larger diameter size; thus milling for a long time and purifying by acid washing are the required steps for producing small SiC powders (Wang et al., 2017b). The post-milling process leads not only to contamination by impurities but also low-quality powders. For instance, milled SiC powders possess wide particle size distribution and irregular polygon particle morphology. Owing to several technical difficulties, which involve high energy consumption, considerable time consumption and inferior purity of the final powders, these substantial drawbacks remained despite significant works having

been carried out. Therefore, synthesis SiC powders with fine particle size, narrow size distribution and spherical shape by reducing reaction temperature and shortened holding time during CR reaction is of considerable importance (Wang et al.,2017).
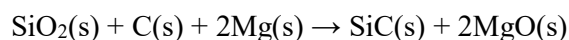
2.4.2 Magnesiothermic reduction method

Additional, magnesiothermal reduction of a mixture of silica, carbon and magnesium can be used to synthesize SiC (Mojarad et al., 2015). This method has the advantage of reducing silica at a lower temperature and providing greater control of the structural ordering. In addition, it is more cost-effective and saves energy because it does not require a high-temperature furnace (Mojarad et al., 2015). Mesoporous SiC has been synthesized by magnesiothermal reduction of mesoporous carbon silica nanocomposites. According to Mojarad et al. (2015), the structure of the resulting mesoporous SiC was less ordered than the mesoporous silicate starting material. The main disadvantage of using mesoporous silicate is its structural instability during firing suggesting that a more reliable starting material to synthesize porous SiC with a high specific surface area might be ordered microporous silicate with a narrow pore size distribution of 0.1–0.8 nm and a high specific surface area (Mojarad et al., 2015).

In the present study, nanostructured SiC was synthesized by magnesiothermal reduction of microporous silica derived from ZSM-5 with carbon from several different sources, to investigate the role of the carbon in the magnesiothermal reaction (Mojarad et al., 2015). Molten-salt-mediated magnesiothermic reduction method was used to prepare 2H-SiC ultrafine powders by using silicon dioxide, active carbon and magnesium

powders as raw materials. Such a process for the production of SiC would never be cost-effective for the bulk production of this commodity and it is not the aim of this work to develop an alternative for bulk SiC production, but to investigate methods for the production of high value–low volume nanostructured SiC for specialized applications such as sintering additives (Mojarad et al., 2015).

The magnesiothermal synthesis of SiC is described by the following equation:

$$SiO_2(s) + C(s) + 2Mg(s) \rightarrow SiC(s) + 2MgO(s)$$

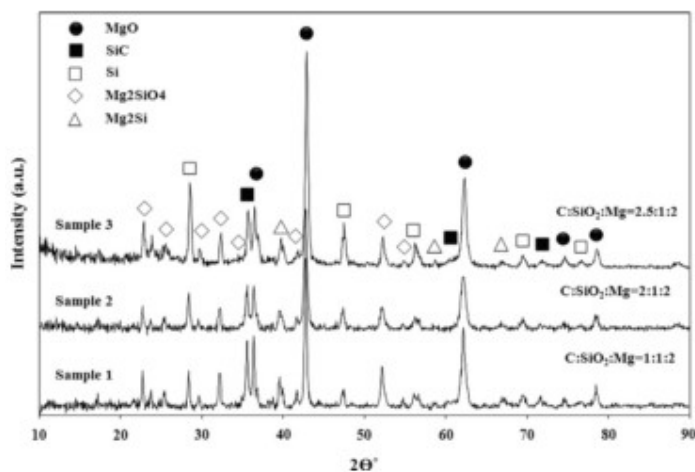$$\Delta G^O = -352.771 + 0.04158T \ (kJ/mol) \hspace{3cm} Eq. \ 2.1$$

Figure 2.3   XRD patterns of as-synthesized SiC samples 1–3 before acid leaching (Mojarad et al., 2015)

According to studies by Tsuboi et al. (2017) they performed the magnesiothermic reduction with two methods, one is the solid state or solid-liquid reaction of silica glass with magnesium, and the other is the reaction with magnesium vapor. These reaction setups are illustrated in Fig. 1. In the solid state or solid-liquid reaction, Tsuboi et al. (2017) tried two ways, in which synthetic silica glass substrates with approximately 5 ×

30 mm2 in size and 2 mm in thickness were used. In one way as illustrated in Fig. 1(a), the silica glass substrates embedded in Mg grains were heat-treated (Tsuboi et al., 2017). The silica substrate with Mg grains was put in a stainless tube of 3/8 in. in outer diameter under Ar atmosphere; the one side of the tube was sealed with a Swagelok® stainless cap (Tsuboi et al., 2017). Then the other side of the tube was sealed with another stainless cap and the tube was heated at 600°C, 650°C, or 700°C for three hours (Tsuboi et al., 2017).
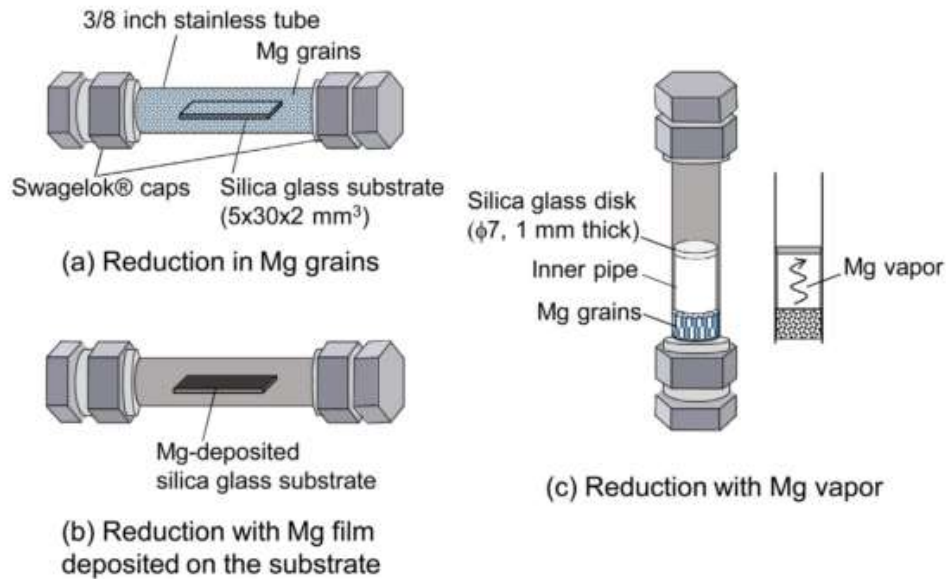

Figure 2.4   Experimental setups for magnesiothermic reduction at various conditions.(a) Reduction in Mg grains, (b) reduction with Mg film deposited on silica glass substrate, (c)reduction with Mg vapour. (a) and (b) are solid state or solid-liquid reaction, and (c) is the solid-vapour reaction (Tsuboi et. al., 2017)

2.4.3   Pyrolysis of polymeric precursors

According to Bao et al. (2000), a simple method was developed to produce silicon carbide foams using polysilane polymeric precursors. Polyurethane foams were immersed in polysilane precursor solutions to prepare pre-foams (Bao et al., 2000). Subsequently, these were heated in nitrogen at different temperatures in the range of 900◦C to 1300◦C

(Bao et al., 2000). The silicon carbide foams produced in this manner showed well-defined open-cell structures and the struts in the foams were free of voids (Bao et al., 2000). The shrinkage which accompanies pyrolysis of the pre-foams was reduced by increasing the concentration of the polymeric precursor in the solutions (Bao et al., 2000).According to Yu et al. (2017), a novel synthetic method to prepare N-doped TiO_2 catalysts was studied, by which a stable and homogeneous liquid polymeric precursor was produced, thus the catalysts can be loaded on suitable substrates, making catalysts facilely separated from waste water. The doping ratios and annealing temperatures were optimized by testing photo degradation of the products to methyl orange (Yu et al., 2017). According to Yu et al. (2017) to improve the Brunauer–Emmett–Teller surface areas of the catalyst, PEG was grafted to the structure of precursor polymer, by which an increase of 55% in BET surface areas and 20% in the photo degradation efficiencies was achieved.

Refer to He et al. (2017) the precursor for ZrB_2 (denoted as ZP) was provided by the Institute of Process Engineering, Chinese Academy of Sciences, Beijing, China. The ZP can be converted to stoichiometric ZrB_2 when pyrolyzed at temperatures above 1500 °C, with a ceramic yield of ~45 wt. % (He et al., 2017). The precursor can be converted to SiC when pyrolyzed at temperatures higher than 1300 °C, with a ceramic yield of ~50 wt. % (He et al., 2017). The precursors were used as-received without further purification. The ZP and PCS, with a desired ratio to form a ceramic composite of ZrB_2-to-SiC volume ratio being 1:4, were dissolved in the mixture of xylene and ethanol (He et al., 2017). Dilute ammonia was then added to the solution under constant stirring to adjust the pH to 4 (He et al., 2017). Addition of ammonia led to the transformation of the liquid solution into a gel (He et al., 2017). After the completion of the gel formation, the solvent was evaporated by vacuum drying at 80 °C to obtain the solid precursor, such solid precursor