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SYNTHESIS AND CHARACTERIZATION OF a-SILICON CARBIDE NANOSTRUCTURES

Enagnon Thymour Legba
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Cubic-phase silicon carbide (β-SiC) nanostructures were successfully synthesized by the reaction of silicon monoxide (SiO) powder with multi-walled carbon nanotubes (MWCNTs) at high temperatures. Experiments were conducted under vacuum or in the presence of argon gas in a high-temperature furnace and the fabrication parameters of temperature (1300 -1500°C), time, and reactant material mass were varied to optimize the material. The resulting samples were then physically characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). XRD analysis revealed the presence of dominant β-silicon carbide phases. SEM images depicted morphologies similar to the starting MWCNTs, having relatively larger diameter sizes, shorter lengths and reduced curvature. TEM observations showed the presence of solid and hollow nanostructures with both crystalline and amorphous regions.

Additional experiments were performed to investigate de-aggregation and dispersion procedures for the β-SiC nanostructures fabricated. Optimum results for these experiments were achieved by ultrasonication of 0.01 wt.% β-SiC in N,N dimethyl formamide (DMF) and dispersion using a spin coater.

A methodology for electrical testing of β-SiC nanostructures was developed using the de-aggregation and dispersion process established. SEM observations revealed that the random nature of the dispersion procedure used was not efficient in forming contacts regions that would allow electrical measurements of β-SiC nanostructures on the pre-patterned silicon substrate.

KEYWORDS: β-SiC silicon carbide nanostructures, Carbon nanotubes, De-aggregation, Dispersion, Shape memory synthesis.

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SYNTHESIS AND CHARACTERIZATION OF β-SILICON CARBIDE NANOSTRUCTURES

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THESIS

Enagnon Thymour Legba

The Graduate School
University of Kentucky
2007
SYNTHESIS AND CHARACTERIZATION OF β-SILICON CARBIDE NANOSTRUCTURES

THESIS

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in Electrical Engineering in the College of Engineering at the University of Kentucky

By

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2007

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I am very thankful to my advisor, Dr. Ingrid St Omer, for her continuous support and guidance in my graduate study and thesis work. I also thank her for giving me the opportunity to work with her on the summer Research Experience for Undergraduates (REU) program.

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CHAPTER 1: INTRODUCTION

1.1 Background

The development of bulk silicon carbide (SiC), as an alternative semiconductor material, has been the focus of several research applications because of its various excellent mechanical, electrical, optical and chemical properties. In fact, bulk silicon carbide’s wide-bandgap, high thermal conductivity, mechanical hardness as well as inertness to exposure in corrosive environments give it the ability to function in high temperature and harsh environmental applications [1, 2]. Electronic devices and microelectromechanical systems (MEMS) based on SiC are being developed, in particular, blue light-emitting devices [3]. In addition, bulk SiC is regarded as a promising substitute for Si or GaAs-based electronic devices, especially in high temperature, high power/high frequency and high radiation device applications [1, 3, 4]. Recent advances in growing ultra-high quality bulk SiC crystal have allowed the development and improvement of SiC-based solar inverters, industrial motor drivers, output rectifiers [5], wireless devices, broadband amplifiers, cellular infrastructure, test instrumentation [6].

However, there is growing interest among the materials and device community regarding the use of semiconductor materials at the nanometer scale for enhanced functionality. This interest is because of the variations in electrical and optical properties that occur when electrons are strongly confined in one-, two-, or three-dimension(s). In fact, since the discovery of carbon nanotubes (CNTs) in 1991 [7], the development of low-dimensional semiconductor nanostructures has been the focus of intensive research for potential application in nanoscience, nanotechnology, and biotechnology because of their
promise for innovative applications [8, 9]. The use of materials at the nanometer scale allows an increase in the surface to volume ratio in comparison to larger ordered structures, an increase in the number of devices that can be built in a given area, and the ability to control properties by varying the particle size. From this perspective, several research groups have achieved growth of SiC nanostructures. Silicon carbide nanostructures are commonly synthesized to reinforce various composite and nanocomposite materials because of their exceptional mechanical properties [10, 11], and also as a heterogeneous catalyst support [12]. Besides, silicon carbide nanostructures have also received much attention in the last ten years because of their potential applications for nanometer scale light emitters [13]. In fact, bulk SiC shows weak blue light emission at room temperature because of its indirect bandgap [14], but SiC blue light emission can be considerably increased when the crystal dimensions are reduced to the nanometer scale [15].

Very little has been achieved in terms of characterizing the electronic properties of SiC nanostructures. SiC nanowire-based FETs have been achieved by two research groups W. Zhou et al. [16] and Seong et al. [17] to determine the electrical properties of SiC nanowires. It was reported that SiC nanowires can appear as an excellent candidate for harsh-environments nano-electronic devices because of their higher carrier mobility and larger current at higher temperatures [16]. However, these two research publications did not offer an in-depth assessment of the electrical properties of SiC nanostructures. The aim of the present paper is to investigate the different synthesis methods and a methodology for measuring the electrical properties of β-SiC nanostructures, as well as their potential applications in nanoscience and nanoelectronics.
1.2 Silicon Carbide Definition and Classification

Silicon carbide is a binary material that belongs to a group of semiconductors known as wide-bandgap semiconductors. It consists of the group-IV elements silicon (Si) and carbon (C) bonded together in a crystal. Silicon carbide can exist in various crystal structures called polytypes. The term polytype implies a particular group of materials that possesses similar chemical or atomic makeup but whose stacking sequence changes along its stacking direction [18]. In a SiC crystal, each Si-C bilayer, also known as the basal plane, is just a planar sheet of silicon atoms close-packed with another planar sheet of carbon atoms [3, 4]. The stacking sequence of Si-C bilayers varies from layer-to-layer along the crystallographic direction normal to the basal plane known as the c-axis or the stacking direction [3], as illustrated in Figure 1.1.

![Figure 1.1: Cross section schematic along (1120) plane of the 6H-SiC polytype [3].](image-url)
Although silicon carbide exists in a multitude of polytypes, its crystalline structures can be divided into three main categories: cubic, hexagonal and rhombohedral. The cubic crystal structure, also known as β-SiC, refers to only a single polytype called 3C-SiC. However, the hexagonal crystal structure is associated with a wider range of polytypes such as 2H-SiC, 4H-SiC and 6H-SiC. Among all hexagonal polytypes, 2H-SiC is the only one that has pure hexagonal crystal structure. The other hexagonal polytypes are arranged in “quasi-cubic” and “quasi-hexagonal” orders with respect to their neighboring Si-C bilayers [3]. The least common polytype is rhombohedral, which refers to polytypes such as 15R-SiC, 21R-SiC and 33R-SiC. The hexagonal and rhombohedral classes of SiC polytypes are collectively known as α-SiC. With more than 200 SiC polytypes known [19], the most commonly used for electronic devices are 3C-SiC, 4H-SiC, and 6H-SiC [3].

To visualize a stacking sequence of a polytype, one can begin by assigning to each Si-C bilayer a letter. For example, if a Si-C bilayer is assigned to an A-plane in close packing as A, a B-plane as B and to a C-plane as C, then different stacking of these planes along the main crystal axis (c-axis) can generate a series of lattice sites. As is illustrated in Figure 1.1, a 6H-SiC lattice can be generated by (ABCACBABCACB…) stacking along the c-axis or the [0001] direction, which requires six Si-C bilayers to define the repeating distance along the c-axis direction. Similarly, (ABAB…) stacking will generate a 2H-SiC wurtzite lattice, and (ABCABC…) stacking will generate a 3C-SiC zinc-blend lattice. Thus, the number placed in front of the letter C-, H- or R- represents how many Si-C bilayers are in the periodic sequence and the letter itself refers to the resulting structure, cubic, hexagonal or rhombohedral, respectively. By increasing the length along the
stacking direction (c-axis), more complicated polytypes can be identified [20]. Silicon carbide is one of the rare compounds that has such stable and so many polytypes [19]. Despite years of research, there is no very clear explanation behind the origin for the formation of so many SiC polytypes. There are two theories developed to try to explain the occurrence of so many polytypes [19]. The first theory is based on the thermodynamical stability of the common short-period polytypes, which are believed to operate as basic structural parts for the formation of long-period polytypes. The second theory is based on the idea that the growth mechanism for long-period structures occurs around screw dislocations. A screw dislocation is a crystal defect that originates when one part of a perfect crystal is twisted or skewed with respect to another part on only one side of the line [21].

1.3 Silicon Carbide Characteristics

The various arrangements of the Si-C double layers affect the properties of the different SiC polytypes. Optical properties of SiC polytypes have been reported [20, 22] and details about the relationships between the optical properties of various SiC polytypes and their structures have been reported [22]. The cubic SiC has a higher melting temperature and higher maximum operating temperature than its traditional counterparts GaAs or Si, as seen in Table 1.1. The cubic SiC high Young’s modulus coupled with high yield strength and Knoop hardness illustrate its excellent mechanical strength. In addition, silicon carbide has excellent chemical properties because it does not get etched by most acids. Silicon carbide can be etched only by alkaline hydroxide bases, as reported by Mehregany et al. [23]. However, these excellent chemical properties can
appear as a major challenge for SiC semiconductor fabrication process. The next section will mainly focus on the electrical properties of various polytypes.

Table 1.1: Comparison of material properties of 3C-SiC with three important MEMS materials at 300 K [4].

<table>
<thead>
<tr>
<th>Property</th>
<th>3C-SiC</th>
<th>GaAs</th>
<th>Si</th>
<th>Diamond</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melting Point (°C)</td>
<td>2830 at 35 bar</td>
<td>1238</td>
<td>1415</td>
<td>4000</td>
</tr>
<tr>
<td>Max operating temp. (°C)</td>
<td>873</td>
<td>460</td>
<td>300</td>
<td>1100</td>
</tr>
<tr>
<td>Young's Modulus (GPa)</td>
<td>448</td>
<td>75</td>
<td>190 (111)</td>
<td>1035</td>
</tr>
<tr>
<td>Linear thermal expansion coeff.</td>
<td>4.7 x 10^{-6} °C^{-1}</td>
<td>5.9</td>
<td>2.35</td>
<td>0.08</td>
</tr>
<tr>
<td>Physical stability</td>
<td>Excellent</td>
<td>Fair</td>
<td>Good</td>
<td>Excellent</td>
</tr>
<tr>
<td>Chemical resistance</td>
<td>Very good</td>
<td>Poor</td>
<td>Good</td>
<td>Excellent</td>
</tr>
<tr>
<td>Yield strength (GPa)</td>
<td>21</td>
<td>2</td>
<td>7</td>
<td>53</td>
</tr>
<tr>
<td>Knoop hardness (Kg/mm^2)</td>
<td>3980</td>
<td>600</td>
<td>1000</td>
<td>10000</td>
</tr>
</tbody>
</table>

1.3.1 Electrical Properties

Even though each SiC polytype chemically consists of the same amounts of carbon atoms bonded covalently with silicon atoms, each one has a distinct set of electrical characteristics. It has been reported by Neudeck [3] that even within a specific polytype, some electrical properties such as the electron mobility for 6H-SiC, may significantly differ compare to other semiconductor materials depending on the crystallographic direction of current flow and applied electric field. As indicated in Table 1.2, the
comparison of three SiC polytypes to traditional semiconductors such as silicon (Si) and
gallium arsenide (GaAs), illustrates some interesting electrical properties of SiC.

Table 1.2: Comparison of electrical properties of SiC with Si and GaAs at room
temperature [3].

<table>
<thead>
<tr>
<th>Property</th>
<th>Silicon (Si)</th>
<th>GaAs</th>
<th>4H-SiC</th>
<th>6H-SiC</th>
<th>3C-SiC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bandgap (eV)</td>
<td>1.1</td>
<td>1.42</td>
<td>3.2</td>
<td>3</td>
<td>2.3</td>
</tr>
<tr>
<td>Relative dielectric constant</td>
<td>11.9</td>
<td>13.1</td>
<td>9.7</td>
<td>9.7</td>
<td>9.7</td>
</tr>
<tr>
<td>Breakdown field at (N_D=10^{17}) cm(^{-3}) (MV cm(^{-1}))</td>
<td>0.6</td>
<td>0.6</td>
<td>3</td>
<td>3.2</td>
<td>&gt;1.5</td>
</tr>
<tr>
<td>Thermal Conductivity (W cm(^{-1})K(^{-1}))</td>
<td>1.5</td>
<td>0.5</td>
<td>3-5</td>
<td>3-5</td>
<td>3-5</td>
</tr>
<tr>
<td>Electron mobility at (N_D=10^{16}) cm(^{-3}) (cm(^2) V(^{-1}) s(^{-1}))</td>
<td>1200</td>
<td>6500</td>
<td>800</td>
<td>60 or 400</td>
<td>750</td>
</tr>
<tr>
<td>Hole mobility at (N_D=10^{16}) cm(^{-3}) (cm(^2) V(^{-1}) s(^{-1}))</td>
<td>420</td>
<td>320</td>
<td>115</td>
<td>90</td>
<td>40</td>
</tr>
<tr>
<td>Saturated electron drift velocity (10(^7) cm s(^{-1}))</td>
<td>1</td>
<td>1.2</td>
<td>2</td>
<td>2</td>
<td>2.5</td>
</tr>
<tr>
<td>Intrinsic carrier concentration (cm(^{-3}))</td>
<td>10(^{10})</td>
<td>1.8x10(^6)</td>
<td>~10(^{-7})</td>
<td>~10(^{-5})</td>
<td>~10</td>
</tr>
</tbody>
</table>

Among those interesting properties are its remarkably high breakdown electric field,
wide-bandgap, high thermal conductivity, and high carrier saturation velocity. The high
electric field breakdown voltage allows the blocking region of a power device to be
approximately 10 times thinner and 10 times more heavily doped with a blocking region
resistance approximately a 100 times less than for Si-based power device [3]. The wide-
bandgap energy voltage coupled with the high electric field breakdown, could allow
fabrication of much faster power-switching devices compared to silicon power-switching
devices. Additionally, the high breakdown field, high thermal conductivity, and the much
smaller intrinsic carrier concentration, allow much higher power densities and
efficiencies to be achieved [3]. Having a wider bandgap energy and a smaller intrinsic concentration could in theory allow SiC semiconductor devices to operate at much higher temperatures than silicon [3, 24, 25]. For example, the intrinsic carrier concentration of silicon usually limits silicon device operation to junction temperatures below 300°C, whereas the much smaller intrinsic concentration of SiC gives electronic devices the ability to operate at junction temperatures well above 300°C [3]. Therefore, for high temperature and harsh environment applications, silicon carbide appears as a more attractive semiconductor material than its traditional counterparts silicon and gallium arsenide (GaAs). A limitation of using SiC in high temperature environments arise from finding appropriate contact materials that could also withstand high temperatures [26].

In theory, the electrical properties of SiC nanostructures could be much better than those of bulk or thin film because of their one-dimensional nature [16, 17, 27]. Nanostructures are nanometer size structures that consist of nanotubes, nanowires or nanorods. Nanotubes are hollow wire looking-like structures, whereas nanorods and nanowires are rather plain solid wire looking-like structures. Even though nanostructures can appear to be remarkable building blocks for device fabrication and implementation [17], very little research has been conducted on the electrical transport properties of SiC nanostructures. One group, Zhou et al. [16, 27] was able to report on the higher current and larger carrier mobility for a SiC nanowire-based FET (Field Effect Transistor) at elevated temperature. Another group, Seong et al. [17], suggested that the low resistivity, and the very low electron mobility of SiC nanowires could be attributed to their one-dimensionality carrier confinement, enhanced scattering in the nanoscale diameter of the SiC nanowire and/or a poor quality SiC nanowire-gate oxide interface.
1.4 Silicon Carbide Micro/nanostructures Synthesis Methods

After the discovery of carbon nanotubes, there has been greater interest in nanometer scale materials because of their one-dimensional characteristics and generally interesting properties. Several research groups have achieved growth primarily of β-SiC micro/nanostructures using high temperature furnace and chemical vapor deposition (CVD) methods. Even though this section will mainly focus on high temperature furnace and CVD methods, other methods for synthesizing β-SiC nanostructures will be briefly discussed.

1.4.1 High Temperature Furnace

The high temperature furnace method is commonly used as it is perhaps one of the easiest ways to produce silicon carbide micro/nanostructures. A typical high temperature cylindrical furnace made out of alumina can reach temperatures as high as 1500°C or more, with high consistency and low contamination. Most research groups utilized shape memory synthesis (SMS), which is a technique that allows SiC micro/nanostructures to be synthesized by controlling the shape, diameter size and length of a carbon source [12] such as carbon nanotubes, which can exist in two forms, single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs). The synthesis of SiC micro/nanostructures using a high temperature furnace occurs through the reaction between silicon monoxide (SiO) vapor and a carbon source at temperatures generally above 1100°C. It can be achieved in two different ways: a one-step reaction and a two-step reaction. Reactions have been carried out at low pressure in air, or in the presence of an inert carrier gas to remove volatile reactants and prevent unwanted reactions. Argon,
or nitrogen have been used to prevent oxidation, and ammonia have been used to observe the effects of hydrogen in the formation of SiC \([11, 24, 28, 29]\). Research groups have also used different ways to separate reactants inside a crucible in the high temperature furnace. For example, one way consisted of placing a carbon source on top of the SiO powder in a crucible [29], and another one consisted of placing the reactants at different location along the tube furnace [7].

### 1.4.1.1 One-step reaction

A one-step reaction involves carbon nanotubes (CNTs) or activated carbon fibers (ACFs) and silicon monoxide (SiO) powder in a high temperature furnace. The high temperature permits the sublimation of SiO powder at temperatures usually above 1100°C, and the reaction between SiO vapor and CNTs/ACFs to form silicon carbide micro/nanostructures. This reaction occurs according to the following equation:

\[
\text{SiO (vapor) + 2C (solid) } \rightarrow \text{SiC (solid) + CO (vapor)} \tag{1.1}
\]

Sun et al. [7] have reported a reaction between MWCNTs, grown on a silicon substrate, and SiO powder to synthesize predominantly β-phase SiC nanotubes and nanowires. The MWCNTs were placed downstream of a crucible of SiO powder at various points along the length of the furnace. The range of temperatures at these positions was estimated to be 850 to 980°C using a calibration curve. The resulting structures were characterized using scanning electron microscopy (SEM), high resolution transmission electron microscopy (HRTEM), electron energy loss spectroscopy (EELS), and elemental mapping. Using the interlayer spacing, which varied between 3.5 and 4.5 Å, the authors claim to have produced a new polytype of multi-walled silicon carbide nanotubes.
(SiCNTs), in addition to β-SiC nanowires. Morisada et al. [30] have reported the synthesis of β-SiC-coated MWCNTs in vacuum at temperatures between 1150 and 1550°C, and have established that the increase in temperature allowed the increase in crystallization and coating of the MWCNTs with SiC layers.

Silicon carbide nanostructures have also been synthesized by reacting silicon (Si) with a carbon source. It can be described by the following equation:

\[
C \text{ (solid)} + Si \text{ (solid)} \rightarrow SiC \text{ (solid)}
\]  \hspace{1cm} (1.2)

Rummeli et al. [24] have reported the synthesis of β-SiC using equation (1.2). Trying to develop definitive correlations between process parameters and resulting morphology, the authors have proposed a 5-state transition. This 5-state transition begins with a SiC coating on the CNT, to SiC nanorod coated in C, to solid SiC nanorod, to porous SiC nanorod, and finally to hollow SiC nanostructure or nanotube.

Taguchi et al. [31] performed a reaction at 1200°C for 100 hours to transform most of CNTs to SiC nanotubes and C-SiC coaxial nanotubes, which are CNTs covered with a SiC layer. The C-SiC was reduced using heat treatment in air to remove the remaining carbon layer. Both methods, one using Si and the other SiO powder, while varying chemically, result in the formation of β-SiC nanostructures.

1.4.1.2 Two-step reaction

A two-step reaction can be implemented at first by generating SiO vapor from the silicon reduction of silica (SiO$_2$) [28, 29, 32] as illustrated by equation (1.3).

\[
Si \text{ (solid)} + SiO_2 \text{ (solid)} \rightarrow 2SiO \text{ (vapor)}
\]  \hspace{1cm} (1.3)
It is followed by a reaction between a carbon source and the generated SiO vapor described in equation (1.1) to form SiC micro/nanostructures. Using this method, Keller et al. [32] and Ryu et al. [33] have reported the synthesis of mostly $\beta$-SiC microstructures using activated carbon fibers at temperatures varying between 1200 and 1300°C for several hours. The higher temperature and longer reaction time yielded more $\beta$-SiC microstructures at the expense of carbon-base material [32].

Han et al. [11] have used the two-step reaction method to synthesize $\beta$-SiC nanorods at 1400°C with diameters ranging between 3 and 40 nm. TEM micrographs revealed that the thinnest SiC nanorods had a high density of defect planes. Several other researchers have also reported the synthesis of $\beta$-SiC nanostructures using the two-step method [12, 28, 34].

### 1.4.2 Chemical Vapor Deposition

Preparing SiC micro/nanostructures using chemical vapor deposition is a more complicated procedure than the high temperature furnace method. A chemical vapor deposition process allows the constituents of a vapor phase, often diluted with an inert carrier gas, to react or decompose on the surface of a hot surface substrate, usually higher than 300°C, to create the desired product [4]. This method is also known to allow deposition of material with high degree of purity, better control and economy [4]. There are various CVD reactions methods that can be utilized to synthesize SiC nanostructures. As an example X. Zhou et al. [10] utilized hot filament CVD (HFCVD) to synthesize $\beta$-SiC nanorods on a silicon substrate using a solid carbon and silicon source. In fact, the authors used a mixture of silicon and graphite powders pressed at 150°C and $3.2 \times 10^8$ Pa
for 24 hours to form a solid plate. The solid plate contained metallic particles such as nickel (Ni), chromium (Cr) and iron (Fe) present in silicon powder that acted as catalysts in the reaction process. This plate was placed 3 mm above the filament while the silicon substrate was placed 2 mm below the hot filament, which was set at 2200°C. Introduction of hydrogen into the reaction chamber was performed to allow etching of the solid source and give off hydrocarbon and hydrosilicon radicals, which reacted to form β-SiC nanorods onto the surface of the substrate at 1000°C.

Lai et al. [35] have successfully synthesized β-SiC straight nanorods on silicon wafers using exclusively iron particles as catalyst. The authors used a plate made of mixture of carbon, silicon and silicon dioxide powders hydraulically pressed at 100°C and 25 × 10^6 Pa for 1 hour. The reaction took place in the presence of hydrogen, on the surface of the Si substrate to form β-SiC nanorods, with diameters varying between 5 and 20 nm.

Synthesized β-SiC nanorods on patterned nickel-coated Si substrate have been reported by Wei et al. [36]. Nickel acted as a catalyst in the reaction and by adjusting the thickness of the Ni film, the authors could control the relative amounts of β-SiC nanorods produced.

1.4.4 Other Synthesis Methods

Although high temperature furnace and CVD are the most prevalent methods, there exits less common procedures for generating β-SiC nanostructures such as high frequency induction heating and direct chemical reactions. A high frequency induction heating method utilizes a cylindrical furnace surrounded by induction coils, which are heated very fast by a high frequency alternating current. The study published by W. Zhou et al.
[37] reported the synthesis of β-SiC nanowires in a vertically set high frequency induction heating furnace. The synthesis of the nanowires was achieved by creating a reaction between SiO powder and activated carbon fibers at temperature around 1450°C and 50 – 100 Torr for 15 min. The SiC nanowires synthesized had diameters varying between 5 and 20 nm and were coated with an amorphous silicon oxide layer.

Lu et al. [38] used a reaction between silicon tetrachloride (SiCl₄) and carbon tetrachloride CCl₄ in argon gas with the presence of sodium metal (Na), which acted as both catalyst and reductant, at 400°C in a pressurized autoclave. The reaction can be described by the following equation:

\[
\text{SiCl}_4 + \text{CCl}_4 + 8 \text{Na} \rightarrow \text{SiC} + 8 \text{NaCl} \quad (1.4)
\]

Cubic SiC nanorods were synthesized during the reaction with diameters ranging from 10 to 40 nm and lengths up to several micrometers. Similarly, Hu et al. [39] have reported the synthesis of β-SiC, with diameters varying between 15 and 20 nm, through a reduction-carburization method by using silicon powder, and metallic Na as the reductant at 700°C.

Other methods utilized reduction of sol-gel-derived silica xerogels that contain carbon nanoparticles, direct carbothermal reduction of halide [29], and decomposition of organic silicon compounds [11]. These methods are less commonly used because of the complexity of their processes, which require more chemical compounds and more process control.
1.5 Challenges

There are many challenges facing SiC nanostructure synthesis and characterization. First, there is a need for a better understanding of the growth process. A better controlled process to synthesize SiC nanostructures could provide a material with greater level of purity, and reduced defect density which would meet with commercial needs. Despite several ways of synthesizing SiC nanostructures, the ones used until now make it difficult to fully control their diameters, lengths and orientations. In this research, after synthesizing SiC nanostructures using high temperature furnace, the main challenge is to manipulate a single nanostructure in a way to create contact between two metal electrodes. As with CNTs [40], laboratory researchers can use high resolution probing devices such as Atomic Force Microscope (AFM), or Scanning Tunneling Microscope (STM) to manipulate SiC nanostructures at the expense of time and cost. This method is very impractical, requires extensive training of the apparatus and is impractical when it comes to large-scale fabrication. Therefore, randomly dispersing SiC nanostructures on a substrate with predefined arrays of metal electrodes is probably the easiest way to get nanostructures at desired locations. One can then select cases where a randomly deposited nanostructure happens to bridge two or more contacts to electrically characterize it. They have been relatively few reports on their physical and electrical properties, as well as related device designs [13], and therefore there is a need for more investigations.
CHAPTER 2: SYNTHESIS AND PHYSICAL CHARACTERIZATION OF $\beta$-SiC NANOSTRUCTURES

2.1 Introduction

Silicon carbide nanostructures can be synthesized using high temperature furnace, chemical vapor deposition, high frequency induction heating, and other methods, as mentioned in Chapter 1. In this chapter, the successful synthesis and physical characterization of $\beta$-SiC nanostructures is reported. The nanostructures were synthesized in a high temperature furnace and were characterized using powder X-ray diffraction (XRD), scanning electron microscopy, transmission electron microscopy (TEM) and high-resolution transmission electron microscopy. These tools allowed evaluation of the SiC growth mechanism, and the role of reaction time and temperature. Powder X-ray diffraction is a simple method for studying crystal structures by irradiating a dry powdered sample with a collimated X-ray beam of known wavelength [21], for example a CuK$\alpha$ monochromatic radiation ($\lambda = 1.54178$ Å). Powdering the crystal allows a set of crystal planes to receive the X-rays at different incident angles $\theta$ and at many different orientations. The diffraction directions which correspond to well-defined diffraction angle $2\theta$, the interplanar separation $d$ of the diffraction planes and the wavelength $\lambda$ of the X-ray are related through the Bragg diffraction condition [21], which is given by:

$$2d \sin \theta = n\lambda \quad n = 1, 2, 3...$$

(2.1)

$n$ is an integer to allow constructive interference to occur between waves. An illustration of the X-ray diffraction phenomenon is shown in Figure 2.1, where $\theta$ represents the angle between the incident X-ray waves A and B and the atomic plane of incidence. The
path difference between the two reflected waves $A1$ and $B1$ corresponds to $2d\sin\theta$. An X-ray detector positioned at angle $2\theta$ with respect to the through-beam will record a peak in the detected X-ray intensity of the diffracted waves $A1$ and $B1$. The intensity will be plotted to show the intensity of X-rays at detector versus the diffraction angle $2\theta$.

Figure 2.1: X-ray diffraction showing two incident waves A and B and diffracted waves $A1$ and $B1$ on various atomic planes in the crystal.

### 2.2 Experimental Methods

A one-step reaction was performed in a high temperature cylindrical tube furnace 4.1 ft long and 0.3 ft in diameter. This reaction allowed the synthesis of SiC nanostructures through a gas-solid reaction between silicon monoxide vapor and MWCNTs, as described in equation (1.1). Loose MWCNTs provided by the University of Kentucky Center for Advanced Energy Research (CAER) were synthesized by floating catalytic CVD, and had an average length of 50 µm and outer an diameter varying between 5 - 100 nm [41].
Three ways for synthesizing β-SiC nanostructures were studied. The initial process was designed to separate the reactants with a thin sheet of graphite felt. The second method established a direct contact between the reactants, and the third one was designed to separate the reactants by placing them at different locations along a rectangular alumina crucible inside the furnace. Among the three different set-ups, only the first one was conducted under vacuum at approximately 60 mTorr. The other two methodologies were conducted in the presence of argon gas, supplied at a rate of 80 – 100 standard cubic centimeters per minute (sccm) throughout the reaction process to remove volatile reactants and prevent undesirable reactions. All three reactions were performed at temperatures between 1300 - 1500°C in a Barnstead Thermolyne F54548CM high temperature furnace. The heating and the cooling rate of the furnace were 15.5°C/minutes and 8°C/min, respectively. The high temperature range was consistent with those reported in the literature. After completion of the reactions, calcination in air was conducted for 30 minutes on several samples at 750°C in order to burn off the remaining carbon. The samples were cooled to room temperature and after placing the samples in a clean plastic box, the crucible was carefully cleaned with deionized (DI) water. Physical characterization of the samples was conducted using Bruker AXS D8 Discover X-ray powder diffraction. First, the samples were placed onto a powder mount and were positioned into the machine for analysis. The measurements were completed using a step-to-step duration scan of 1.2 s and an angular step scan of 0.02° from 10° to 90°. After determining the composition of the samples, their morphology was observed using a Hitachi S-4300 SEM. The nanostructure and chemical composition of the samples were studied using a TEM (JEOL - 2000FX and 2010F).
2.2.1 Thin Graphite Felt Separation

In this reaction, reactants were arranged inside a quasi-cylindrical alumina crucible that has a top diameter of 4.5 cm, a bottom diameter of 2.8 cm and a height of 4.5 cm. A brownish silicon monoxide powder (Aldrich -325mesh) was positioned at the bottom of the alumina crucible and separated from black MWCNTs by a thin sheet of graphite felt (Alpha Aesar 99%). The assembled crucible was then placed in a Barnstead Thermolyne F54548CM high temperature tube furnace, as illustrated in Figure 2.2.

Figure 2.2: Schematic diagram of the high temperature furnace used in the synthesis of SiC nanostructures. Reactants are separated by a thin sheet of graphite felt inside an alumina crucible.

Several reactions were performed in vacuum of approximately 60 mTorr, over a temperature range of 1300 - 1450°C and a dwell time range of 3 - 5 hours. After cooling the sample, the top part was meticulously collected to be analyzed. The purpose of this
process was to isolate the MWCNTs from the SiO powder so that the resulting SiC nanostructures could be easily collected.

2.2.1 Direct Contact

In this reaction, reactants were also arranged inside a cylindrical alumina crucible with the same dimensions mentioned above. Loose MWCNTs were positioned directly on top of SiO powder, which was placed at the bottom of the alumina crucible. The assembled crucible was then placed in a Barnstead Thermolyne high temperature tube furnace, as shown in Figure 2.3.

Figure 2.3: Schematic diagram of the high temperature furnace used in the synthesis of SiC nanostructures. Reactants are in direct contact inside an alumina crucible and in the presence of argon.
In this experiment, reactions were performed over a temperature range of 1300 - 1400°C and a dwell time range of 0.25 - 4 hours. After cooling the samples, the top part was carefully collected for analysis.

2.2.2 Separated Reactants

In this reaction, reactants were arranged inside a rectangular alumina crucible with dimensions of 7.5 cm × 5.0 cm and a depth of 1 cm. One gram (1g) of loose MWCNTs was positioned at one end of a rectangular alumina crucible and 0.5 g of SiO powder was positioned at the opposite end, and separated from the MWCNTs. The assembled crucible was placed in a Barnstead Thermolyne high temperature tube furnace, as shown in Figure 2.4.

Figure 2.4: Schematic diagram of the high temperature furnace used in the synthesis of SiC nanostructures: reactants are separated along the alumina crucible’s length.
The silicon monoxide powder was placed closer to the inlet of the argon gas flow so that once SiO powder reaches its vapor phase; it gets carried downstream by argon to the other end of the alumina crucible to react with the MWCNTs. In this experiment, reactions were performed over a temperature range of 1300 - 1500°C and a dwell time range of 1 - 3 hours.

2.3 β-SiC Nanowires under X-ray Diffraction and SEM

Following synthesis, the various samples were cooled to room temperature and physically characterized using X-ray powder diffraction and SEM. Using X-ray diffraction allowed identification of the presence of SiC and other material components. SEM analysis was primarily performed on the initial samples, and those that exhibited SiC peaks.

2.3.1 Results

X-ray diffraction was conducted on MWCNTs and SiO powder to be used as a reference for comparison with the reacted samples. The comparison allowed identification of any unreacted carbon or SiO after reaction. Figure 2.5 and Figure 2.8 illustrate X-ray diffraction performed on loose MWCNTs and SiO powder, respectively. The y-axis, which has no unit, represents the intensity of X-rays at the detector and the x-axis represents the diffraction angle $2\theta$. The most significant peak on the MWCNTs occurs at approximately $2\theta = 26^\circ$ with a y-axis intensity slightly above 400. However, the dominant peak on the SiO X-ray spectrum had an intensity approaching 100. After X-ray diffraction was performed on the reacted samples, the nature of the various crystalline
phases present in the samples was checked using the database of the International Center for Diffraction Data (ICDD) to determine the composition of the resulting material.

The initial reactants, MWCNTs and SiO powder, were imaged using SEM, as illustrated in Figure 2.6 and Figure 2.9, respectively. The MWCNTs are not completely straight tubes but rather curved and generally entangled. The typical diameter of the MWCNTs used in this experiment varies between 50 - 100 nm, as seen in Figure 2.7, and the MWCNTs can be over 20 μm long. Under SEM, SiO powder looks like microscopic grains of different diameters. Both MWCNTs and SiO powder structures have distinct morphologies, which allow for visual comparison with the synthesized structures.

![X-ray diffraction of uncreated loose MWCNTs showing characteristic peaks. Dominant peak occurs at $2\theta = 26^\circ$.](image)

Figure 2.5: X-ray diffraction of uncreated loose MWCNTs showing characteristic peaks. Dominant peak occurs at $2\theta = 26^\circ$. 
Figure 2.6: SEM image of loose MWCNTs prior to reaction showing straight and entangled nanostructures.

Figure 2.7: SEM image of loose MWCNTs prior to reaction showing a MWCNT with an approximate diameter of 100 nm.
Figure 2.8: X-ray diffraction of unreacted SiO powder. As expected for an oxide, the peaks are broad and relatively indistinct.

Figure 2.9: SEM image of SiO powder prior to reaction showing grain-like structures.
2.3.1.1 Thin Graphite Felt Separation

Reactions conducted at 1300°C for both 3 hours resulted in a blackish-brownish colored powder, as indicated in Table 2.1. This color palette was characteristic of the initial reactants. At this temperature, there was no peak associated with the presence of SiC according to the ICDD. There was not enough SiO vapor pressure induced inside the crucible to penetrate the carbon felt and react with the MWCNTs. Thus, the amount of MWCNTs was decreased to increase the SiO/MWCNTs ratio (2:1), as described in Table 2.1. The reaction time was also increased from 3 to 5 hours to allow more time for the reaction to take place but the result was identical. There was still no presence of SiC characteristic peaks on the X-ray diffraction plot.

When the temperature was increased from 1300 to 1450°C, after a 3-hour reaction time, a grayish product distinct from that of the initial reactants was formed, as described in Table 2.1. Using both SiO/MWCNTs ratios of (1:1) and (2:1) resulted in the presence of a gray material. X-ray diffraction conducted on these samples revealed the presence of \( \beta \)-SiC with major peaks occurring at \( 2\theta = 35.8^\circ, 41.3^\circ, 60^\circ \) and \( 71.9^\circ \), as indicated by the red markers in Figure 2.10. As a result, the characteristic color of \( \beta \)-SiC nanostructures was identified to be gray. The highest \( \beta \)-SiC peak had a relative intensity between 700 and 800. The X-ray diffraction plot in Figure 2.10 also revealed the presence of silica \( (\text{SiO}_2) \) at \( 2\theta = 22.1^\circ \) and \( \text{Fe}_2\text{Si}_3 \) at \( 2\theta = 45.1^\circ \). Figure 2.11 (a) and (b) illustrate the resulting sample structures following a reaction at 1450°C for a duration of 3 hours. As the reaction time was increased from 3 to 5 hours at 1450°C, for a (2:1) SiO/MWCNTs ratio, very small amount of gray material was formed. The red marker peaks
characterizing β-SiC were much smaller and the dominant peak corresponded to the presence of SiO₂, as illustrated in Figure 2.12.

Table 2.1: Experimental conditions and parameters in the graphite felt experiments.

<table>
<thead>
<tr>
<th>Reactants</th>
<th>Weight (grams)</th>
<th>Temperature (°C)</th>
<th>Time (hours)</th>
<th>Color</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO</td>
<td>.440</td>
<td>1300</td>
<td>3</td>
<td>Brown/Black</td>
</tr>
<tr>
<td>CNTs</td>
<td>.390</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SiO</td>
<td>.400</td>
<td>1300</td>
<td>5</td>
<td>Brown/Black</td>
</tr>
<tr>
<td>CNTs</td>
<td>.290</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SiO</td>
<td>.405</td>
<td>1450</td>
<td>3</td>
<td>Gray</td>
</tr>
<tr>
<td>CNTs</td>
<td>.351</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SiO</td>
<td>.730</td>
<td>1450</td>
<td>3</td>
<td>Gray</td>
</tr>
<tr>
<td>CNTs</td>
<td>.400</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SiO</td>
<td>.700</td>
<td>1450</td>
<td>5</td>
<td>Gray/White</td>
</tr>
<tr>
<td>CNTs</td>
<td>.365</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 2.10: X-ray diffraction on a sample prepared using graphite felt at 1450°C for 3 hours. The red peak markers represent the dominant cubic phase.
Figure 2.11: (a) Silicon carbide-coated carbon microfibers after reaction at 1450°C for 3 hours. (b) Synthesized nanostructure material present after reaction at 1450°C for 3 hours. Remnants of unreacted silicon monoxide powder are clearly visible.

Figure 2.12: X-ray diffraction on a graphite thin film separated sample prepared at 1450°C for 5 hours.
Even though $\beta$-SiC was formed at 1450°C at a duration of 3 hours, the thin carbon felt method was not efficient in isolating SiC nanostructures from SiC microstructures. All the supporting carbon felt ended up breaking up during the reaction process, which added some unwanted materials (SiC-coated carbon felt and unreacted SiO$_2$) to the material collected for analysis.

### 2.3.1.2 Direct Contact

The previous experiments conducted in vacuum suggested that carbon was burning off at a faster rate at elevated temperatures. Thus to avoid a rapid loss of carbon material, argon was introduced in the experiment. The direct contact reactions were carried out in the presence of a neutral gas argon to significantly reduce unwanted reactions particularly between carbon and excess oxygen. The absence of the graphite felt in the direct contact
reactions confirmed the source of the large SiC microfibers shown in Figure 2.11 (a). However, after completing the direct contact reactions, it became very delicate to separate the newly formed product from the unreacted materials.

Both 1 and 2-hour reactions conducted at 1300°C have generated gray and black material, as is described in Table 2.2. A hard brownish residue was also formed at the bottom of the crucible. The black material is believed to be unreacted MWCNTs. Calcination of the samples was implemented to burn off the remaining unreacted carbon. X-ray diffraction on the 1 and 2-hour samples consistently produced \( \beta \)-SiC, Si at \( 2\theta = 26.5^\circ \), \( \text{Fe}_2\text{Si}_3 \), and significant silica peaks, as seen in both Figure 2.14 and Figure 2.16. The relative intensity of the highest \( \beta \)-SiC peaks remained between 400 and 500 for both 1 and 2-hour samples. Additionally, 1 and 2-hour reactions produced \( \beta \)-SiC nanostructures with morphologies comparable to the initial MWCNTs with larger diameters than the source structures, as illustrated in Figure 2.15 and Figure 2.17, respectively.

Table 2.2: Experimental conditions and parameters in the direct contact experiments.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Time (hours)</th>
<th>Color</th>
<th>Calcination</th>
<th>Peaks</th>
</tr>
</thead>
<tbody>
<tr>
<td>1300</td>
<td>1</td>
<td>Gray/Black</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>1300</td>
<td>2</td>
<td>Gray/Black</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>1400</td>
<td>0.25</td>
<td>Gray/Black</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>1400</td>
<td>0.5</td>
<td>Gray/Black</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>1400</td>
<td>1</td>
<td>Gray/Black</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>1400</td>
<td>2</td>
<td>Gray/Black</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>1400</td>
<td>3</td>
<td>Brownish</td>
<td>No</td>
<td>No</td>
</tr>
<tr>
<td>1400</td>
<td>4</td>
<td>Brownish</td>
<td>No</td>
<td>No</td>
</tr>
</tbody>
</table>
Increasing the temperature from 1300 to 1400°C and conducting the reaction for just 15 minutes also produced a gray and black product. Calcination was performed to reduce the presence of unreacted carbon as illustrated in the X-ray diffraction results of Figure 2.18. The relative intensity of the dominant $\beta$-SiC peak increased to a level between 600 and 700. In this reaction, the presence of silica was negligible as opposed to the 1300°C reactions. SEM images showed structures similar to MWCNTs, with diameters between 100 – 200 nm as seen in Figure 2.19.

As reaction time increased from 15 to 30 minutes, and 1 and 2 hours, the presence of silica and Si gradually increased, as illustrated in Figure 2.18, Figure 2.20, Figure 2.22 and Figure 2.24, respectively. The relative intensity of dominant $\beta$-SiC peak increased as well from around 600 for a 15-minute run to 700 for both 30-minute and 1-hour duration. For a 3-hour reaction time, the $\beta$-SiC peaks decreased, as illustrated in Figure 2.26 and Figure 2.27. SEM images of samples synthesized after 30 minutes, and 1 and 2 hours, have all shown relatively straight MWCNTs-like structures, as illustrated in Figure 2.21, Figure 2.23 and Figure 2.25, respectively. However, the surface of the structures appeared rougher in comparison with the source MWCNTs. Figure 2.25 (b) offers a close-up view of hollow $\beta$-SiC nanostructure, with an outer diameter of approximately 100 nm.
Figure 2.14: X-ray diffraction on a direct contact sample prepared at 1300°C for 1 hour followed by calcination. The red peak markers represent the dominant cubic phase.

Figure 2.15: SEM image of a direct contact sample prepared at 1300°C for 1 hour and followed by calcination clearly indicating the presence of β-SiC nanostructures.
Figure 2.16: X-ray diffraction on a direct contact sample prepared at 1300°C for 2 hours followed by calcination. The red peak markers represent the dominant cubic phase.

Figure 2.17: SEM image of a direct contact sample prepared at 1300°C for 2 hours followed by calcination.
Figure 2.18: X-ray diffraction on a direct contact sample prepared at 1400°C for 15 minutes. The black trace corresponds to the sample before calcination, and the green trace associates with the calcinated sample. The red peak markers represent the dominant cubic phase.

Figure 2.19: SEM image of a direct contact sample prepared at 1400°C for 15 minutes followed by calcination.
Figure 2.20: X-ray diffraction results for a direct contact sample prepared at 1400°C for 30 minutes followed by calcination. The red peak markers represent the dominant cubic phase.

Figure 2.21: SEM image of a direct contact sample prepared at 1400°C for 30 minutes followed by calcination.
Figure 2.22: X-ray diffraction on a direct contact sample prepared at 1400°C for 1 hour followed by calcination. The red peak markers represent the dominant cubic phase.

Figure 2.23: SEM image of a direct contact sample prepared at 1400°C for 1 hour and followed by calcination clearly showing β-SiC nanostructures.
Figure 2.24: X-ray diffraction on a direct contact sample prepared at 1400°C for 2 hours followed by calcination. The red peak markers represent the dominant cubic phase.

Figure 2.25: Both (a) and (b) are SEM images of a direct contact sample prepared at 1400°C for 2 hours followed by calcination. β-SiC nanotube structure is shown in image (b).
Figure 2.26: X-ray diffraction on a direct contact sample prepared at 1400°C for 3 hours followed by calcination. The red peak markers represent the dominant cubic phase.

Figure 2.27: SEM image of a direct contact sample prepared at 1400°C for 3 hours followed by calcination.
### 2.3.1.3 Separated Reactants

The final SiC nanostructure synthesis method involved high temperature reactions at 1300°C for 1, 2 and 3 hours. Experiments at this lower temperature have not exhibited discernable SiC nanostructures. X-ray diffraction analysis performed on the 1, 2, and 3-hour samples at 1400°C, have all shown β-SiC peaks, as described in Table 2.3. Calcination was again performed to reduce any unreacted carbon material present in the samples. All three reactions conducted at 1400°C have consistently shown the presence of β-SiC with major peaks occurring at $2\theta = 35.8°, 41.3°, 60°$ and $71.9°$, as illustrated in Figure 2.28, Figure 2.30 and Figure 2.32. In contrast with most of the direct contact reactions, the presence of Silica or Si in the collected samples was essentially undetected. The relative intensity of the dominant β-SiC peak was consistently above 1100. However, traces of Fe were still present as they combined with Si atoms to form Fe$_2$Si$_3$. The nanostructures had rougher surfaces, shorter lengths, and larger diameters than the source MWCNTs, as seen in Figure 2.29, Figure 2.31 and Figure 2.33.

Comparable X-ray diffraction results were observed on the samples from reactions conducted at 1500°C for 1, 2 and 3 hours, as depicted in Figure 2.34, Figure 2.36 and Figure 2.38, respectively. SEM images showed more straight nanostructures with larger diameters and shorter lengths than the source MWCNTs, as seen in Figure 2.35, Figure 2.37 and Figure 2.39. A diameter size of 250 nm typical of the β-SiC nanostructures synthesized at 1500°C is illustrated in Figure 2.35 (d). For the 1500°C samples, the surface of the nanostructures appeared rougher as reaction time increased.
Table 2.3: Experimental conditions and parameters in the separated reactant experiments.

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Time (hours)</th>
<th>Color</th>
<th>Peaks</th>
<th>Calcination</th>
</tr>
</thead>
<tbody>
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<td>1300 °C</td>
<td>1</td>
<td>Black/Red</td>
<td>No</td>
<td>No</td>
</tr>
<tr>
<td>1300 °C</td>
<td>2</td>
<td>Black/Red</td>
<td>No</td>
<td>No</td>
</tr>
<tr>
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<td>3</td>
<td>Black/Red</td>
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</tr>
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<td>Gray/Black</td>
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<td>Yes</td>
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<td>Gray/Black</td>
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Fe$_2$Si$_3$   β-SiC
β-SiC

Figure 2.28: X-ray diffraction on a separated sample prepared at 1400°C for 1 hour followed by calcination. The red peak markers represent the dominant cubic phase.
Figure 2.29: SEM analysis of a separated sample prepared at 1400°C for 1 hour followed by calcination, showing relatively smooth surfaces.

Figure 2.30: X-ray diffraction on a separated sample prepared at 1400°C for 2 hours followed by calcination. The red peak markers represent the dominant cubic phase.
Figure 2.31: SEM images of a separated sample prepared at 1400°C for 2 hours followed by calcination, showing nanotube looking-like structures.

Figure 2.32: X-ray diffraction analysis of a separated sample prepared at 1400°C for 3 hours followed by calcination. The red peak markers represent the dominant cubic phase.
Figure 2.33: SEM images of a separated sample prepared at 1400°C for 3 hours followed by calcination, exhibit irregular surfaces.

Figure 2.34: X-ray diffraction on a separated sample prepared at 1500°C for 1 hour followed by calcination. The red peak markers represent the dominant cubic phase.
Figure 2.35: SEM analysis of a separated sample prepared at 1500°C for 1 hour followed by calcination. Image (d) shows a relatively large diameter nanostructure.
Figure 2.36: X-ray diffraction on a separated sample prepared at 1500°C for 2 hours followed by calcination. The red peak markers represent the dominant cubic phase.

Figure 2.37: SEM images of a separated sample prepared at 1500°C for 2 hours followed by calcination show relatively rough surfaces. Image (a) contains some closely packed nanostructures.
Figure 2.38: X-ray diffraction analysis of a separated sample prepared at 1500°C for 3 hours followed by calcination. The red peak markers represent the dominant cubic phase.

Figure 2.39: SEM images of a separated sample prepared at 1500°C for 3 hours followed by calcination.
Figure 2.40 and Figure 2.41 contain a comparison of weight for reaction components versus time after the 1400°C and 1500°C reactions for durations of 1, 2 and 3 hours, respectively. The gray material generally corresponds to the SiC nanostructures and the black represents unreacted MWCNTs and other carbon by-products.

Figure 2.40: Weight comparison of black and gray reaction products at 1400°C for 1, 2 and 3 hours prior to calcination.

Figure 2.41: Weight comparison of black and gray reaction products at 1500°C for 1, 2 and 3 hours before calcination.
2.3.2 Discussion

2.3.2.1 Thin Graphite Felt Separation

Using the thin film graphite felt separation method demonstrated that temperature is a critical parameter in triggering the formation of β-SiC nanostructures. In fact, reactions conducted at 1300°C for 3 and 5 hours have shown no traces of SiC peaks. This level of temperature was not sufficient to generate a high enough SiO vapor pressure to penetrate the carbon felt and react with the MWCNTs. Most of the SiO powder did not sublimate and it is believed that the very small amount of SiO vapor formed stayed below the carbon felt and did not react.

However, an interesting observation was made as temperature increased from 1300 to 1450°C. After a 3-hour reaction time, β-SiC material was formed and indicated that a relatively high temperature was needed to transform MWCNTs into β-SiC. At high temperatures, SiO vapor pressure increased substantially to penetrate the carbon felt and form SiC. The formation of β-SiC was explained through having a systematic replacement of C atoms by Si atoms with the release of CO during the reaction between the SiO vapor and the MWCNTs. Such a transformation process therefore provided a final material, which had the same general morphology as the initial MWCNTs. The presence of Fe particles in the sample was explained by the use of Fe as a catalyst material in the synthesis of the initial MWCNTs. Iron atoms combined with Si atoms to form Fe$_2$Si$_3$ and therefore the presence of Fe-based impurities became intrinsic to the reaction. Moreover, some amount of silica (SiO$_2$) was also found in the sample. It was suggested that SiO vapor decomposed into SiO$_2$, according the following equation:
2 SiO (vapor) $\rightarrow$ SiO$_2$ (solid) + Si \hfill (2.2)

The larger microstructures in Figure 2.11 (b) appeared to be the results of a comparable reaction between the graphite felt fibers and the silicon monoxide vapor. Keller et al. [32] have also reported the synthesis of SiC microstructures very similar to the ones shown in Figure 2.11 (b). Results depicted in Figure 2.11 (a), typical of the intended reaction with MWCNTs, appeared less curved and smaller in length than the original starting material. The absence of SiC peaks in the 5-hour run at 1450°C suggested that carbon containing material might have burnt off before changing into SiC. Since the reaction occurred in air, despite a relatively low pressure of approximately 60 mTorr, formation of CO gas might have been more significant, which caused most of the carbon material present in the sample to burn off and leave mostly silica.

In addition, the use of carbon felt to separate SiO powder from the MWCNTs did not turn out to be efficient because the thin felt had a tendency to disintegrate at the end of the reactions, which caused the bottom and the top of the reaction products product to come into contact. The carbon felt method did not allow identification of the source of the SiC peaks i.e. if the change resulted from conversion of the felt microfibers or the MWCNTs. Therefore, it was not a very efficient method for synthesizing SiC nanostructures.

### 2.3.2.2 Direct Contact

By flowing argon gas in the tube furnace and creating direct contact between the reactants, SiC nanostructures were formed at 1300°C contrary to the thin graphite felt for the same temperature. As SiO powder transformed into SiO vapor, the reaction started at the interface between the two reactants, which resulted in the formation of $\beta$-SiC
nanostructures. The SiC nanostructures were more visible in the SEM images from this process than in the images obtained for the graphite felt experiments. The presence of Fe$_2$Si$_3$ was still noticeable in the direct contact reactions. Beside Fe$_2$Si$_3$, a significant amount of silica and Si was found in the sample. At high temperature SiO vapor decomposed into SiO$_2$ and Si according to equation (2.2).

The presence of a high amount of silica in the collected samples could be from the method used to gather the reaction products. It was very tedious to try to collect the gray product without mixing it with some of the unwanted product. In fact, after all the reactions, there was always a hard build-up of a brownish product (mixture of SiO$_2$ and Si) at the bottom of the crucible, which very often ended up mixing together with some of the above gray color product.

Similar results were observed from reactions at 1400°C for 1 and 2 hours. Both 1300°C and 1400°C reactions have produced SiC nanostructures in which the smooth surfaces of the starting MWCNTs were transformed into ones that were more irregular, with thicker diameters (between 100 and 200 nm), less curvature, and shorter lengths. It was suggested by Nhut et al. [12] that the disordered structures was probably caused by the loss of carbon atoms to CO gas with a density change when going from C to SiC.

Tang et al. [28] suggested that the straight shape of the SiC nanostructures results from the competition between the oriented growth of SiC crystals and the bending footpath provided by the carbon source [28]. According to Tang et al., even though CNTs have high stiffness, high modulus and axial strength, they vibrate at different temperatures. The vibrations increase with increasing temperature and this property combined with the high flexibility of the CNTs causes them to change their morphology during SiC
nanostructures formation. Shorter length SiC nanostructures are a consequence of the stiffness of the new structures formed. The less flexible ones just break and result in shorter structures. Some of the formed SiC nanostructures indicated that some of nanostructures were nanotubes, as seen in Figure 2.25 (b). No valid theory was developed to explain why reactions at 1400°C for 3 and 4 hours did not provide β-SiC peaks except for potentially higher conversion rates of CNTs into CO instead of SiC.

Direct contact reactions provided a more effective way to synthesis nanostructures than the graphite felt method. However, contamination in the collection of the samples was a direct result of the contact between initial reactants. As a result, silica and silicon were almost always present in the collected samples.

2.3.2.3 Separated Reactants

The absence of β-SiC peaks from reactions conducted at 1300°C for 1, 2 and 3 hours, could be explained by the a very low reaction between SiO vapor and MWCNTs. It was mentioned earlier that the rate of transformation of SiO powder into SiO vapor at 1300°C was not high enough. Therefore, sufficient amounts of SiO vapor did not get transported to the other end of the rectangular crucible to react with the MWCNTs. As a result, no β-SiC peaks were detected using X-ray diffraction on the 1300°C samples.

As temperatures increased from 1300 to 1500°C, transformation of SiO powder to SiO vapor became more significant improving the volume of material transported to the other end of the tray for reaction. The newly formed product was separate from the SiO powder. As a result, the presence of silica and Si peaks were considerably reduced in the collected samples, as illustrated in Figure 2.28, Figure 2.30, Figure 2.32, Figure 2.34,
Figure 2.36 and Figure 2.38. However, Fe$_2$Si$_3$ was still present in all reactions, which suggested that it was an intrinsic part of the reactions because of the presence of Fe in initial MWCNTs. Evidence of the presence of Fe in the initial MWCNTs was also established using Energy Dispersive Spectroscopy (EDS), as shown in Figure 2.42. The separated reaction method helped demonstrate the effect of time on the formation of β-SiC nanostructures. Longer reaction times have shown a steady increase of β-SiC formed up to a certain point and a steady decrease of starting MWCNTs, as shown in Figure 2.40 and Figure 2.41. It was suggested by Keller et al. [32] that because of the Shrinking Core model, a longer time was needed in order to get a high conversion of C into SiC. According to the Shrinking Core model, the reaction at the interface is at its peak at the beginning of the transformation and decreases gradually as the reaction takes place. The entire carbon surface was available to SiO vapor at the beginning of the reaction and as soon as SiC formation started, the conversion rate decreased due to the diffusion limitation of the SiO and CO vapors through the first SiC layers. Such a process would lead to the formation of SiC with a carbon core but that could be significantly reduced by undergoing calcination. As far as the SEM images, the same observations and conclusions as for the direct contact experiments could be drawn, such as rougher surfaces, larger diameter, as seen in Figure 2.35 (d) and shorter lengths than the original MWCNTs. Overall, the transformation of MWCNTs into β-Si nanostructures followed a method called shape memory synthesis (SMS). The final morphology of the β-SiC nanostructures was very similar to the MWCNTs.
2.4 β-SiC Nanowires Morphology under TEM and HRTEM

TEM characterization was carried out with a JEOL-2000FX at 200 kV to explore the internal structures of a separated reactants sample prepared at 1400°C for 1 hour. Figure 2.43 depicts several SiC nanostructure morphologies with an average diameter of approximately 100 nm. The structure in the red box shows a hollow configuration characteristic of a nanotube, whereas the other structures appear solid in nature. A nanotube with a diameter of approximately 200 nm surrounded by an amorphous outer shell is illustrated in Figure 2.44. It has also been observed that many of the nanostructures have varying surface morphology. In some cases SiC nanoparticles assembled together, as shown in Figure 2.45.

HRTEM conducted on a nanostructure indicates 5 nm of an amorphous layer covering the nanostructure, as seen in Figure 2.47. A high magnification image can be seen in Figure 2.48, which also shows a crystalline phase.
Figure 2.43: TEM micrograph of SiC nanostructures on a separated reactants sample prepared at 1400°C for 1 hour, showing a hollow structure marked in red.

Figure 2.44: TEM micrograph of SiC nanostructures on a separated reactants sample prepared at 1400°C for 1 hour, showing an outer shell layer covering a nanotube.
Figure 2.45: TEM micrograph of SiC nanostructures on a separated reactants sample prepared at 1400°C for 1 hour, showing a straight nanostructure made of SiC nanoparticles.

Figure 2.46: TEM micrograph of SiC a nanostructure on a separated reactants sample prepared at 1400°C for 1 hour, indicating irregular structure.
Figure 2.47: HRTEM micrograph of SiC a nanostructure on a separated reactants sample prepared at 1400°C for 1 hour. An amorphous layer is clearly visible.

Figure 2.48: HRTEM micrograph of SiC a nanostructure on a separated reactants sample prepared at 1400°C for 1 hour. Amorphous and crystalline structures are visible.
Figure 2.49: HRTEM micrograph of SiC a nanostructure on a separated reactants sample prepared at 1400°C for 1 hour. A crystalline phase is highlighted in red circle.
CHAPTER 3: DISPERSION OF β-SiC NANOSTRUCTURES

3.1 Introduction

Multi-walled CNTs were used as a template for synthesizing SiC nanostructures, and as a result, the latter inherited the shape of the MWCNTs. The synthesis of SiC nanostructures using a high temperature furnace produced a range of morphologies that were entangled, or distributed in aggregates. For that reason, it is very difficult to directly measure the electrical properties of individual SiC nanostructures. In order to achieve de-aggregation, the as-grown nanostructures need to be dispersed into fluid suspensions so that one could manipulate and study the electrical properties of an individual SiC nanostructure. De-aggregation is a procedure through which separation or detachment of entangled nanostructures can be accomplished.

In this work, ultrasonication and conditioning mixer methods are studied to identify which technique facilitates de-aggregation of the suspended SiC nanostructures. Once de-aggregation is achieved, an effective method for depositing the nanostructures on a substrate is needed. Two methods are studied to disperse the nanostructures on a silicon substrate. One method involved dipping the substrate into the de-aggregated suspension, and the other method is characterized by dispersing the de-aggregate suspension onto the substrate surface with a spin coater. Hilding et al. [42] have reported the dispersion of CNTs in liquids by investigating the effects of ultrasonication and dispersant systems on the morphology of CNTs. The authors suggested that physical damage of the CNTs is almost inevitable in the dispersion process because of the intermolecular and Van Der Waal’s forces that keep CNTs together. SiC nanostructures could be affected by the
dispersion in a fluid phase that may affect the chemical make-up of SiC nanostructures, which could possibly change their electrical properties.

In this study, the most effective de-aggregation method was combined with the most effective dispersion method to achieve optimal nanostructure distribution. This combination randomly positions SiC nanostructures on a silicon wafer patterned with predefined arrays of metal contacts to allow for electrical characterization of the nanostructures.

3.2 Experimental Procedures

3.2.1 Ultrasonication Bath versus Mixing and Dipping versus Spin Coating

The synthesized SiC nanostructures were de-aggregated and suspended in ACS Grade N, N-dimethyl-formamide (DMF) using a 40 kHz Branson Model 5510 ultrasonic cleaner and a Thinky Model AR-250 Conditioning Mixer. Approximately 2 mg of SiC were deposited in solution to achieve 0.02 wt %, as shown in Table 3.1. The first set of experiments described in Table 3.1 studied a suspension of approximately 0.02 weight percent (wt. %) suspension of SiC/DMF at various durations of ultrasonication or mixing. A silicon wafer was cut into small pieces that could be mounted on cylindrical SEM stubs 15 mm in diameter and 5 mm thick. The pieces were cleaned using acetone, isopropyl alcohol (IPA) and Deionized (DI) water. Ultrasonication and mixing of the SiC nanostructures in DMF were conducted. The de-aggregated suspension was deposited on the Si pieces via dipping or spinning. A set of eight experiments were performed. Four pieces of Si wafer corresponding to each experiment were dipped into the suspension for
2 min. Then, the samples were set to dry in a fume hood or placed on a Fisher Scientific hot plate set to 120°C for 1 min. For the alternative method, a clean disposable Pasteur pipette was used to deposit one drop of the suspension on four other pieces of Si wafer and were spun at approximately 3500 rpm for thirty seconds. After spinning, the samples were either left to dry under the fume hood or placed on a Fisher Scientific hot plate set to 120°C for 1 min. Once dried, the samples were examined using a Hitachi S-4300 SEM.

Table 3.1: Methods and quantities used for de-aggregation of SiC nanostructures

<table>
<thead>
<tr>
<th>DMF Volume (mL)</th>
<th>SiC Mass (mg)</th>
<th>Wt.% of SiC</th>
<th>De-aggregation method</th>
<th>Time (hours)</th>
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<td>10</td>
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<td>4</td>
</tr>
</tbody>
</table>

3.2.2 Optimization Experiment using Ultrasonication Bath and Spin Coater

After the set of preliminary experiments conducted in section 3.2.1 and results analysis, which will be discussed in the next section, a method for optimization of the de-aggregation and dispersion experiments was performed. Approximately 2 mg of SiC nanostructures were placed into test tubes that contained 10 mL or 20 mL of DMF, as shown in Table 3.2. The experiments described in Table 3.2 were performed to provide either 0.01 or 0.02 wt. % SiC/DMF ratio suspension for various time durations using the ultrasonication method. The optimization experiment allowed comparing the effects of time and relative density on dispersion characteristics. A silicon wafer was cut into
pieces, which were cleaned using acetone, IPA and DI water. After ultrasonication of the SiC nanostructures in DMF, the de-aggregated suspension was deposited on the Si pieces using a clean disposable Pasteur pipette. The Si pieces were then spun on the spin coater at approximately 3500 rpm for thirty seconds. After spinning, the samples were either left to dry under the fume hood or placed on a Fisher Scientific hot plate set to 120°C for 1 min. The samples were examined using a Hitachi S-4300 SEM.

Table 3.2: Optimum method for de-aggregation of SiC nanostructures

<table>
<thead>
<tr>
<th>DMF Volume (mL)</th>
<th>SiC Mass (mg)</th>
<th>Wt.% of SiC</th>
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</table>

3.3 Results and Discussions

3.3.1 Ultrasonication Bath versus Mixing and Dipping versus Spinning

The primary tool used to evaluate the dispersion of the SiC nanostructures across the wafer was the Hitachi S-4300 SEM. Image analysis of ultrasonication and mixing techniques is qualitative at best because it is difficult to quantify artifacts of mixing methodology from those of dispersion. However, the first striking observation comes from the difference between the dipping and the spin coating methods. The dipping method consistently resulted in more nanostructure aggregations. The 4-hour ultrasonication bath followed by dipping, shown in Figure 3.1 (a), exhibits a larger
amount of aggregation than the spin coating method shown in Figure 3.1 (b). The same observation can be made when comparing the 6-hour ultrasonication bath followed by dipping, as seen in Figure 3.2 (a), and the spin coating method, seen in Figure 3.2 (b). At a lower magnification (80x), one can see a large well-defined area of agglomerations in Figure 3.2 (a). The resulting clumps are created by the surface tension that occurs between the DMF solution and the surface of the wafer, which is caused by the molecular unbalance of cohesive forces in the contact region where the two phases (liquid-solid) meet. Consequently, the presence of a pool traps nanostructures in a smaller area with a higher density, which defeats the purpose of this experiment. In contrast, such large areas of clumped nanostructures are not observed with the spin coating method. The images (Figure 3.1(b) and Figure 3.2 (b)) where the spin coating method was used after ultrasonication show a relatively sparse dispersion of nanostructures. The increased presence of individual nanostructures makes the spin coating method more effective than the dipping method.

For the 2-hour samples using the mixing method, it can also be observed in Figure 3.3 (a) after dipping, that there are more aggregates than in Figure 3.3 (b) after spin coating. The same observation can be made for the 4-hour mixing method. The dipped sample, as seen in Figure 3.4 (a) has more aggregates than for the spin coated sample, as seen in Figure 3.4 (b). As a result, the spin coating technique is more effective than the dipping technique using the mixing method. Additionally, the mixing method does not adequately separate or de-aggregate the nanostructures.

Both Figure 3.1 (b) and Figure 3.2 (b) using the ultrasonication method show more individual nanostructures than in both Figure 3.3 (b) and Figure 3.4 (b) for the mixing
method. Many reasons can explain the effectiveness of the ultrasonication over mixing method. Ultrasonication uses sound waves from a transducer that radiate through the solution to deliver ultrasonic energy with alternating high and low pressures in the solution. Fluid ultrasonication generates three physical mechanisms such as cavitation of the fluid, localized heating, and the formation of free radicals. Cavitation is the mechanism by which bubbles are formed and imploded in the fluid. Depending on the frequency, bubbles can be larger for a lower frequency (about 20 kHz), and smaller for a higher frequency (about 50 kHz). At 40 kHz, bubbles produce high energy forces as they implode or collapse, which can help reduce aggregation and entanglement of the nanostructures. During ultrasonication, cavitation can cause dispersion and fracture of the nanostructures [42]. The conditioning mixer merely helps mixing DMF and the nanostructures without generating enough internal forces that would effectively break apart entanglements and aggregations. Consequently, the best combination results from using ultrasonication and spin coating methods.
Figure 3.1: SEM image of sample after (a) 4-hour ultrasonication, 2-minute dip and (b) 4-hour ultrasonication, spin coating.
Figure 3.2: SEM image of sample after (a) 6-hour ultrasonication, 2-minute dip and (b) 6-hour ultrasonication, spin coating.
Figure 3.3: SEM image of sample after (a) 2-hour mixing, 2-minute dip and (b) 2-hour mixing, spin coating.
Figure 3.4: SEM image of sample after (a) 4-hour mixing, 2-minute dip and (b) 4-hour mixing, spin coating.
3.2.2 Optimization Experiment

After choosing ultrasonication and spin coating as the best methods for the
de-aggregation and dispersion, the goal is to optimize the parameters. The variables in
this optimization experiment are time and suspension concentration. By increasing time
and decreasing the concentration from 0.02 wt. % to 0.01 wt. %, the objective is to
observe how the time and concentration of the suspended nanostructures impact
aggregation, entanglement and dispersion. For both 0.02 wt. % and 0.01 wt. % the
difference appeared to be small. In both cases, more individual nanostructures were
observed despite the variation in time duration. Moreover, the dispersion density of
nanostructures for 0.01 wt. % appears relatively smaller than that of the 0.02 wt. %. As is
illustrated in Figure 3.6 (a) and Figure 3.6 (b), the density is larger at certain locations
than at others for the same time duration and wt. %. For the purpose of this experiment,
the lower density is preferable to effectively isolate individual nanostructures. The shorter
times seemed to result in more of the longer structures, possibly because of the fracture
potential.
Figure 3.5: SEM image of on the wafer after (a) 4-hour ultrasonication, SiC 0.02 wt.%, spin coating and (b) 6-hour ultrasonication, SiC 0.02 wt%, spin coating.
Figure 3.6: SEM image of on the wafer after (a, b) 4-hour ultrasonication, SiC 0.01 wt.%, spin coating, (c) 6-hour ultrasonication, SiC 0.01 wt%, spin coating.
CHAPTER 4: DESIGN FOR ELECTRICAL CHARACTERIZATION OF $\beta$-SiC NANOSTRUCTURES

4.1 Introduction

After synthesis and characterization of the $\beta$-SiC nanostructures using X-ray diffraction, SEM and TEM, it was concluded that of the three methods discussed in Chapter 2, the separated reactants method was the most effective way to synthesize the nanostructures. As a result, $\beta$-SiC nanostructures prepared at 1400°C for 1 hour were selected for dispersion for electrical measurements. The dispersion method used was based on the optimized experimental results. Using a similar dispersion technique on SiC nanowires, Zhou et al. [16, 27] reported the fabrication of a SiC nanowire-based FET (Field Effect Transistor) that conducted a current in the nanometer range. In this Chapter, an attempt to measure the current transport for the synthesized SiC nanostructures is discussed.

4.2 Experimental Procedures

4.2.1 Mask Design and Specifications

AutoCad software was used to design two different patterns for a 4” square mask that would allow patterning of metal electrodes on a wafer. Both patterns are illustrated in Figure 4.1 (a) and (b), where image (a) shows a design that would allow several electrical measurements along a nanostructure’s length from two or more electrode tips and image (b) shows a simple two-electrode pattern designed for fixed electrical measurements. The spacings for the patterns were determined based on the average length of the
nanostructures from SEM images analysis. The length of the SiC nanostructures after dispersion on a wafer varied between 500 nm and 5 μm. Spacing as small as 500 nm between two electrode tips would be preferable but because of some design fabrication constraints of the mask, 2 μm was the smallest spacing allowed. Many researchers have reported spacing as small as 300 nm for CNTs [43] and 1 μm for SiC [16].

Figure 4.1: Mask design showing two different patterns that would be used for electrical measurements. All values indicated are in microns.
4.2.2 Initial Wafer Characterization and Clean

The resistivity $\rho$ of the p-type (100) wafer was determined using a 4-point probe, a Keithley 224 current source, and a 2000 digital multimeter (DMM) and was found to be $1.99 \times 10^{-2} \, \Omega \cdot \text{cm}$. Thus, the doping concentration $N_A$ of the wafer was determined to be $3.80 \times 10^{18} \, \text{cm}^{-3}$. A p-type wafer was used in order to create an easy back gate contact if a FET was planning to be made. The thickness of the wafer was measured at $275 \pm 25 \, \mu\text{m}$.

The wafer was initially cleaned using acetone and isopropyl alcohol (IPA) to remove dust and grease, followed by a rinse with DI water. A dilute solution of DI and hydrofluoric acid (HF) (300 mL: 10 mL) was used to etch the native oxide from the wafer surface. This step was followed by a standard RCA1 clean, which consisted of a solution of DI: hydrogen peroxide ($\text{H}_2\text{O}_2$): ammonium hydroxide (NH4OH) (250 mL: 50 mL: 50 mL). This step removes organic contamination. After RCA1, another native oxide removal was performed. Next, the wafer underwent a standard RCA2 clean, which consisted of a solution of DI: $\text{H}_2\text{O}_2$: hydrochloric acid (HCL) (250 mL: 50 mL: 50 mL) to remove metallic or ionic impurities. The wafer was again rinsed following a native oxide etch.

4.2.3 Dry Thermal Oxidation

After dehydration bake at $120^\circ\text{C}$ to insure that the surface of the wafer was dry, dry thermal oxidation was performed since thermal oxidation requires the wafer to be totally dry and free of any surface contamination. Prior to placing the wafer into the furnace, the furnace was slowly ramped to $800^\circ\text{C}$ at a rate of $15^\circ\text{C}/\text{min}$ and filled with nitrogen at 1500 sccm and 20 psi to purge any external contaminants. Afterward, the wafer was gradually loaded into the furnace to prevent thermal stress, and the temperature was
slowly increased to 1100°C. Nitrogen gas was replaced by oxygen at 500 sccm and 20 psi for approximately 16 hours to grow a dense oxide layer. Post-oxidation annealing was then performed in nitrogen for approximately 20 minutes to minimize the presence of fixed charge. After thermal oxidation, an ellipsometer was used to measure the oxide thickness grown, which was found to be approximately 338.57 nm.

4.2.4 Photolithography: Alignment and Exposure

Following oxide thickness measurements, the wafer was cleaned in acetone, IPA and DI water. Another dehydration bake was performed to ensure the wafer surface was dry. The wafer was placed on the spin coater and a few drops of Shipley S1813 positive photoresist (PR) were applied. The wafer was then spun at 3500 rpm for 30 seconds, followed by a soft bake on a hotplate at 100°C for 1 minute (or whatever you did). The wafer was transferred to the Karl Suss mask aligner and exposed to UV light for 7 seconds. Once the exposure was completed, the photoresist was developed with MF-319 developer using an immersion process with agitation for 40 - 60 seconds. The develop process was followed by a DI rinse. Wafer inspection using an optical microscope allowed verification of the pattern transfer.

4.2.5 Metallization and Lift-off

E-beam evaporation was used to deposit 100 nm of aluminum (Al) at $5 \times 10^{-6}$ torr. Following deposition of aluminum on the wafer, the sample was soaked in a beaker of heated Shipley 1165 resist remover. During the lift-off process, the photoresist under the film is removed with a solvent, taking away the metal film with it, and leaving only the
film that was deposited directly on the substrate. Once the photoresist and metal were removed, the wafer was transferred to a beaker with IPA and rinsed. The wafers were examined using an optical microscope to determine the quality of the lift-off process. Following this step, the contacts designed to enable electrical characterization of the SiC nanostructures were complete.

4.2.6 β-SiC Nanowires Dispersion

In this stage of the work, β-SiC nanostructures synthesized at 1400°C for 1 hour using the separated reactants method were used. Dispersion of the nanostructures was based on the optimization results discussed earlier in Chapter 4. Ultrasonication of 0.01 wt. % SiC/DMF was conducted for 4 hours to de-aggregate the entangled β-SiC nanostructures. A spin-coating process was performed at 3500 rpm for 30 minutes to deposit the suspension containing the β-SiC nanostructures onto the pre-patterned substrate. After drying the dispersed nanostructures on a hot plate, the pre-patterned substrate was taken for electrical measurements. The major steps of the fabrication and dispersion process used with the β-SiC nanostructures are illustrated in Figure 4.2.

4.2.7 Electrical Testing of β-SiC Nanostructures

The current-voltage (I-V) measurements were conducted in a dark chamber of a probe station. After adjusting position of the sample and carefully making contact to the device pads with the probes, calibration was performed with the help of a microscope. An Agilent 4155B Semiconductor Parameter Analyzer was used to perform I-V measurements. Thin film aluminum contact resistance was determined by performing a
voltage sweep from 0 - 0.58 V with a 0.01 V step increment. A voltage sweep from 0 – 5 V was conducted between two adjacent thin film aluminum contacts in increment of 0.01V to perform I-V measurements.

Figure 4.2: Schematic of fabrication process illustrating the major steps before electrical measurements.
4.3 Results and Discussions

4.3.1 Electrical Measurements

At first, the aluminum thin film contact resistance was determined from the I-V plot seen in Figure 4.3. The slope of the linear curve was determined to be 0.0872 \( \Omega^{-1} \). Using Ohm’s law, \( V = I \times R \), the resistance of the curve represents the inverse of the slope, and therefore \( R \) was found to be approximately 11.49 \( \Omega \).

The next step was to measure current between the predefined arrays of metal electrodes by sweeping the voltage from 0 - 5 V. Current in the micrometer or nanometer range was expected. Unfortunately, all measurements consistently resulted in output currents in the picometer range, comparable to noise. This suggested that either there were no SiC nanostructures bridging two aluminum electrodes, or the nanostructures were highly resistive. To verify these assumptions, the surface of the samples was imaged using an SEM.
4.3.2 Device under SEM

The random dispersion of SiC nanostructures on a substrate with predefined arrays of metal electrodes is probably the easiest method to achieve placement of nanostructures on the contacts. However, all SEM images in Figure 4.5 (a) – (n) have demonstrated unsuccessful positioning of the SiC nanostructures on any of the desired metal electrode locations. These results indicate that the dispersion method used to achieve contact between two or more metal electrodes is very unreliable. In fact, there is no control of the location where a SiC nanostructure will fall. Consequently, the absence of contact between two electrodes explains the lack of valid current measurements. The SEM images can be divided in three main groups. The first group shows sufficiently long SiC nanostructures (over 2 µm) that did not land in between the 2 µm-separated electrode tips, as shown in Figure 4.5 (c), (d), (f), (h), (k), (l), (m) and (n).
The second group consists of SiC nanostructures that landed between two electrode tips but were too short to connect both ends, as depicted in Figure 4.5 (b), (e) and (i). The final group shows SiC nanostructures over 2 µm long that contacted one electrode tip but failed to contact the other end because of orientation, as seen in Figure 4.5 (g), (j) and (o). In many cases, the SiC nanostructures were less than 2 µm long, which suggests that patterns with smaller dimension specifications between the electrode tips could have increased the probability for the SiC nanostructures to create contact.
Figure 4.5: (a) – (o) are SEM Images of the pre-patterned wafer surface after dispersion.
5.1 Conclusion

Synthesis of $\beta$-SiC nanostructures by reaction of SiO powder and loose MWCNTs, as well as de-aggregation and dispersion methods to form contacts on a pre-pattern silicon wafer, have been studied. Three synthesis techniques, thin graphite felt separation, direct contact, and separated reactants, have been investigated to determine the most effective way to synthesize SiC nanostructures.

The thin graphite felt experiments conducted under vacuum at 1450°C for 3 hours have shown formation of $\beta$-SiC at both the micro- and nano-scale. However, the elevated temperature caused the thin graphite felt to disintegrate, resulting in a mixture with nanostructures and significant amounts of microstructures. This method was determined to be ineffective in isolating the desired $\beta$-SiC nanostructures from unwanted materials such as the microstructures and silica.

The direct contact experiments in an argon ambient have demonstrated a wider range of temperatures (1300 -1400°C) and synthesis times (1-3 hours) for successful fabrication of 100-200 nm diameter $\beta$-SiC nanostructures. However, it was initially thought that the $\beta$-SiC nanostructures formed on top of the unreacted SiO powder could be easily collected but the final product turned out to be highly contaminated with silica and silicon materials. Even though SiC nanostructures were created for a broader range of temperatures and times, the presence of non-negligible unwanted materials made the method ineffective.
The most effective synthesis method of the three techniques studied was the separated reactants process, which produced 100 -200 nm diameter β-SiC nanostructures over a temperature and time range of 1400 -1500°C and 1-3 hours, respectively. The main advantage of this method over the other two synthesis techniques was the improved isolation of the β-SiC nanostructures from the unwanted materials.

All three methods exhibited components of an Fe-based material, which was caused by the Fe catalyst used in the fabrication of the initial MWCNTs. Purification of the MWCNTs prior to SiC synthesis could reduce these components. Both the direct contact and separated reactants method have clearly shown an increase in the diameters of the β-SiC nanostructures compared to MWCNTs, believed to be caused by a substitution of carbon atoms by the larger silicon atoms beginning at the outer shell of the MWCNTs and progressing inward. Less curved and straighter β-SiC nanostructures have also been observed in comparison with the original MWCNTs.

De-aggregation of SiC nanostructures in DMF using ultrasonication followed by a random dispersion of the solution on a wafer with predefined electrodes using a spin-coater was used to attempt to place SiC nanostructures on desired contact locations. However, the random dispersion method is very unreliable for SiC nanostructures characterization, for engineering applications, and for future large-scale device nanofabrication. Manipulation of nanostructures remains challenging, and more investigation is necessary to achieve valid physical and electrical characterization.
5.2 Future Work

There is a need for a better understanding of the growth process of $\beta$-SiC nanostructures. More investigation is needed to design a better-controlled process to synthesize SiC nanostructures with desirable diameter, lengths and orientations that could provide a material with greater level of purity, and reduced defect density which meet with commercial needs.

Reducing the spacing between pre-defined electrode tips from 2 $\mu$m to a couple of hundreds of nanometers would make the random dispersion technique more effective. However, a major challenge in using silicon carbide nanostructures as semiconductor devices or materials for electronics is getting them to grow at pre-defined locations in order to characterize them. Growth of silicon carbide nanostructures at pre-defined locations would improve the ability to contact the structures for electrical measurements. Fabrication of uniformly ordered vertically aligned SiC nanotube arrays using a porous alumina template would allow contact of individual and groupings of nanostructures. This technique is ideally suited for creating metal or semiconductor nano-dots or nanowires/ nanotubes within an insulating alumina matrix. This method has been reported [44] to synthesize highly aligned carbon nanotubes (CNTs). The alumina matrix would provide a basis for synthesizing a uniform array of electrically insulated CNTs that could be converted to SiC nanotubes using SiO vapor reaction at a high temperature. Contact pads would be used to electrically connect the vertical nanotubes, and a conductive atomic force microscopy tip could be used to measure the properties of individual structures.
REFERENCES


VITA

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