Impact of Fluorination on Microstructures and Surface Properties of SiC Nanocomposites with $\text{Si}_x\text{C}_y\text{F}_z$ Composition

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ABSTRACT: Silicon carbide (SiC) is an effective catalyst for generating fuel from organics through gasification. SiC has shown promising results as a catalyst due to its extraordinary thermal and oxidation resistance abilities. Researchers are yet to identify an efficient silicon carbide composite material that enhances the desired quality of fuel/liquid production. The present study deals with in situ synthesis of fluorine-doped silicon carbide using agriculture waste. Biochar, a waste by-product from the gasification process, proved to be a potential carbon source for fabrication of silicon carbide nanowires (SiCNWs). Pristine SiCNWs exhibited nanospheres and freestanding nanowire (coiled, rods, bamboo-like, or hexagonal prism) structures with transversal optical mode indexed to the $\beta$-phase ($\beta$-SiC). Fabrication of fluorine (F)-doped SiC from a silica–carbon–fluorine (SiO$_x$/C$_y$/F$_z$) precursor resulted in uneven flat-surfaced silicon carbide materials accompanied by progressive pore blockage with increasing F-content. Pore blockage was confirmed by declining the surface area from 60.70 m$^2$ g$^{-1}$ of the lowest dopant to 17.33 m$^2$ g$^{-1}$ of the maximum dopant, compared to neat SiC (63.20 m$^2$ g$^{-1}$). Introduction of fluorine led to decreased silicon contents and collapsed nanowire while the carbon and oxygen contents increased.

INTRODUCTION

Silicon carbide (SiC) is a class of materials that exhibit one-dimensional (1D) polymorphism. It has over 250 polytypes, but the most popular among these are the wide-band-gap $\alpha$-SiC and $\beta$-SiC.$^{1,2}$ In comparison, $\beta$-SiC with zinc blende structure has relatively fewer uses than $\alpha$-SiC. Over the years, silicon carbides have received considerable attention as potential semiconductors in fabricating high technological electronic devices operating at high frequencies, high power, or high temperatures and high radiation; catalytic supports; and reinforcements in ceramic matrix composites and metal matrix composites.$^{2,4}$ Their outstanding material properties have made SiC strategic and biocompatible materials. High thermal conductivity, high electric field breakdown strength, excellent mechanical properties, high chemical and thermal stabilities, and unique optical properties exhibited by SiC are some of the advantages that made SiC and its composites favorable semiconductors in numerous applications.$^{2,6}$

Successful methods used for fabricating SiC nanostructured materials include arc discharge, chemical vapor deposition, carbothermal reduction, and sol–gel methods, amongst others.$^{7,10}$ Synthetic routes are in some instances catalyst assisted$^{11}$ or catalyst free$^{12}$ and have produced various SiC morphological nanostructures. Although efforts have been made to synthesize SiC nanostructures with controlled shape through either pressure variation of precursors$^{13}$ or reaction temperature variation,$^{14}$ morphological control of SiC nanostructures as well as the use of expensive catalysts, raw materials, and sophisticated techniques by most of the synthetic methods mentioned above pose challenges on the large-scale production of SiC.$^{9}$ The need to use affordable raw materials as the carbon source for SiC production is gaining attention over nonrenewable engineered carbon sources such as carbon black, activated carbon, hydrocarbon gases, graphite, carbon nanotubes, and polymers.$^{7,15,16}$ The use of renewable materials$^{7,18}$ as unconventional carbon sources in the fabrication of carbide nanostructures is being considered although their application is still in the minimal scale compared to nonrenewable sources.
One of the important applications is in the field of catalysis, where the surface properties such as surface area and porosity are prime features that display the catalytic efficiency. To achieve the efficiency of silicon carbide, many dopants especially transitional metal ions such as iron and cobalt have been used. In this report, we wish to investigate and understand the effect of fluorine via in situ doping during the formation of SiC. The study of effects of fluorination has been better reported in silicon nanowires than in silicon carbide nanowires (SiCNWs). Fluorination is one of the important reactions in carbon-containing materials like SiC, in which it serves as the chemical modification route and can result in diverse electronic structures. Fluorine-based dry etching of bulk SiC is being explored for widening the SiC applications and device fabrications.

In this work, silicon carbide has been prepared from the biochar of biomass materials as the carbon source through a metal catalyst and template-free carbothermal reduction of sol–gel-derived silica. To assess the impact of halogen incorporated into the lattice of silicon carbide influencing the overall material properties, a fluorine dopant was introduced during the synthesis of SiC nanostructures as a case study. The incorporation of a halide in the SiC matrix was done using NH₄F to make a random chemical composition of SiₓCᵧFz.

The SiC was synthesized using biochar obtained from the gasification process and polysiloxane generated in situ from sol–gel technology. The proximate analyses of biochar, determined in triplicate through the ASTM method (ASTM D1762-84), showed 82% of fixed carbon available to react with polysiloxane.

### RESULTS AND DISCUSSION

Upon carbothermal reduction at 1400 °C in an argon atmosphere, the following reactions 1–6 take place, giving rise to SiC nanowires along with cristobalite and amorphous carbon.

\[
\begin{align*}
\text{SiO}_2(s) + C(s) & \rightarrow \text{SiO}(g) + \text{CO}(g) \\
\text{SiO}(g) + 2C(s) & \rightarrow \text{SiC}(s) + \text{CO}(g) \\
\text{SiO}(g) + 3\text{CO}(g) & \rightarrow \text{SiC}(s) + 2\text{CO}_2(g) \\
3\text{SiO}(g) + \text{CO}(g) & \rightarrow \text{SiC}(s) + 2\text{SiO}_2(s) \\
\text{CO}_2(g) + C(s) & \rightarrow 2\text{CO}(g) \\
\text{SiO}_2(g) + 3C(s) & \rightarrow \text{SiC}(s) + 2\text{CO}_2(g)
\end{align*}
\]

Overall reaction

The electron microscopic analysis at different magnifications: (a−c) scanning electron microscopy (SEM), (a′−c′) transmission electron microscopy (TEM) images with insets (a″−c″) of the selected area electron diffraction pattern and corresponding energy-dispersive system (EDS). Elemental mapping (d, e) of neat and maximum-doped carbide samples.

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**Figure 1.** Electron microscopic analysis at different magnifications: (a−c) scanning electron microscopy (SEM), (a′−c′) transmission electron microscopy (TEM) images with insets (a″−c″) of the selected area electron diffraction pattern and corresponding energy-dispersive system (EDS). Elemental mapping (d, e) of neat and maximum-doped carbide samples.
The $\beta$-SiC formed has shown nanowire dominance of 3C-SiC (dominance) prevalence and 6H SiC, with stacking faults as reported earlier. However, ammonium fluoride when incorporated into the system deteriorated the growth of SiC and gave rise to flat layered structures with gradual reduction of nanowires. The temperature used for the reaction was 1400 °C, and the decomposition of ammonium fluoride at $\sim$100 °C cannot be ignored as per reactions 7 and 8:

$$2\text{NH}_4\text{F}(s) \rightarrow \text{NH}_4\text{HF}_2(s) + \text{NH}_3(g)$$ (7)

$$\text{NH}_4\text{HF}_2(s) \rightarrow \text{NH}_3(g) + 2\text{HF}_2(g)$$ (8)

Gaseous ammonia ($\text{NH}_3$) and hydrogen fluoride (HF) generated at low temperatures of $\sim$100 °C are important components from reactions 7 and 8, respectively. The presence of HF causes typical dissolution of oxides through adsorption of F species on the surface of the oxide layer. This makes Si–O–Si bonds to be unstable under a HF environment due to the bond attack by fluoride species. Reaction of these components with the silica precursor may lead to the formation of intermediate products such as the SiO$_2$–NH$_3$ complex and silica tetrafluoride (SiF$_4$) that would eventually reduce the silicon ion concentration from the precursor and subsequently SiO reductive vapors, required for the formation of SiC. The probable reactions giving rise to products responsible for depleting these precursors are given in reactions 9 and 10:

$$\text{SiO}_2(s) + \text{NH}_3(s) \rightarrow \text{SiO}_2(s) - \text{NH}_3(s)$$ (9)

$$\text{SiO}_2(s) + 4\text{HF}_2(g) \rightarrow \text{SiF}_4 + 2\text{H}_2\text{O}(s)$$ (10)

SiO$_2$–NH$_3$ and SiF$_4$ byproducts are expected to occupy the available pores as evidenced from a significant decrease in porosity and the Brunauer–Emmett–Teller (BET) surface area as discussed later. The Fourier transform infrared (FTIR) absorption peak of fluorine-doped silica (Si–F stretching bond) fabricated at temperatures $<400$ °C is observed around the 850–1000 cm$^{-1}$ region. Such temperatures are imperative for stabilization of fluorine bonds. However, the spreading of strong Si–N and Si–O–S absorptions along 750–1100 and 800–200 cm$^{-1}$ regions, respectively, tends to obscure the Si–F absorption peak of interest. The high volatility of fluorine at a temperature of $\sim$450 °C minimizes the absorption modes signifying the presence of fluorine compounds. The Si–H (str, $\sim$2100 cm$^{-1}$), N–H (bending, 1150 cm$^{-1}$), and Si–N (str, $\sim$860 cm$^{-1}$) peak modes in Figure 2c vanishes at temperatures $>800$ °C or may be observed under newly formed bonds. The high carbothermal reduction temperatures (1400 °C) of as-prepared fluorine-doped silica-biochar (SiO$_{x}/$C/$F_4$) composites could not allow condensation and settling of the SiO$_2$–NH$_3$ complex. The low-temperature-stable complex may have retarded the SiC nuclei initiation and growth but does not prevent the process completely. This is confirmed by a low amount of grown SiCNWs observed on fluorine-doped samples, the presence of Si–F (str, 953 cm$^{-1}$) in the reduced peak intensity when compared to those fabricated at moderate temperature, and reduction in peak intensities of Si–O (str, 1078 cm$^{-1}$) modes of all fluorine-modified materials.

**Influence of the Dopant on the Internal Structure of Silicon Carbide.** Microstructures of carbothermally reduced SiC materials are presented in Figure 1. Figure 1a reveals that the structural morphology of SiC consists of nanospheres and nanowires (SiCNWs). SiCNWs are freestanding and randomly orientated as either coiled fibers, smooth rods, bamboollike, or hexagonal prism structures. Hexagonal prism structures and bamboollike nanowires exhibit stacking fringes or stacking faults (SFs). The rods depict the SiC core–shell structure coated by a SiO$_2$ layer ($\sim$15 nm, thickness). Diameters and lengths showed no uniformity with diameters ranging from $\sim$47 to 95 nm (granules), $\sim$38–138 nm (rods), and $\sim$154 nm (hexagonal prism), while the length of the nanowires range from 127 nm to few 10 μm. SiC structures observed in Figure
1a’ corroborates with Figure 1a, illustrating no interlacing or hollowed nanowires.

Typical flat end features denoting catalyst-free derived silicon carbides are depicted.27 Microscopic images of SiC–F-doped materials in Figure 1b,c,b’,c’ support each other and show collapsing of SiCNW structures with traces of few. Uneven surfaces (rich in Si and O contents) covering underlying agglomerated nanograins are observed (b’, c’), confirmed by elemental compositions and mapping (EDS). The amount of F-contents was negligibly low, and in other materials, none could be detected.

**X-ray Diffraction (XRD) Studies.** The diffraction pattern of SiC (neat) in Figure 2a depicts β-SiC peaks with corresponding lattice planes, at 2θ = 35.8° (111), 43.4° (200), 60.2° (220), 72.0° (311), and 75.6° (222) [JCPD card no. 03-065-0360]. A broad peak at 2θ = 25.9° is indexed to SiO2 (quartz) [JCPD card no. 01-073-3466] and C [JCPD card no. 03-065-0360], while a peak at 2θ = 33.8° is attributed to stacking faults. The interplanar spacing between two adjacent lattice fringes was obtained to be 0.25 nm. Fluorine-doped materials exhibit changes that include decreasing intensities of β-SiC characteristic peaks (2θ = 35.8, 43.4, and 60.2°) and additional SiO2 and amorphous C (graphite)28 trace peaks. The decreasing peak intensities of planes indicate the degrading crystalline quality of SiC–F materials with increasing F-dopants. Noticeable peaks at 2θ = 31.0, 28.6, ∼27.0, and 22.0° are ascribed to SiO2 and an intense sharp peak at 22.0° is ascribed to SiO2 (cristobalite) and C (graphite). Shallow peaks at ∼10.0° of SiC–F (0.50) and SiC–F (1.00) correlate with the (111) plane of carbon, as reported.29

**FTIR Analyses.** FTIR absorption peaks illustrated by an undoped SiC spectrum (Figure 2b) depict the typical Si–C stretching vibration at 798 cm⁻¹ (an indicative of the transverse optical (TO) mode of the SiC crystal), Si–O of Si–O–CH3 symmetric stretching and Si–OH bending modes at 749 and 868 cm⁻¹ indicates the unreacted silica, and Si–O–Si asymmetric stretching of bridging absorbance and Si:C-H rocking/wagging modes are observed at 1080 cm⁻¹. The strength of the 1080 cm⁻¹ peak mode corresponds to the silica sheath on SiC nanowires and the granule surface. Other peaks at 1430 cm⁻¹ denote Si–CH3 or Si–CH2 bending modes, 1900 cm⁻¹ depicts C=–C, 2090 and 2275 cm⁻¹ depict Si=H, and O=Si–H stretching modes respectively, and 1700 and 2515 cm⁻¹ are attributed to the C=O stretching and C–O (of carbonyl), respectively. A broad band in the ∼2800–3400 cm⁻¹ region depicts the C=H modes of alkanes and alkenes, and O–H modes of Si–O–H and water molecules. F-doped spectra illustrate the disappearance of peaks in the 1450–3100 cm⁻¹ region, comprising mostly of Si–H, C–O, and –OH. F-doped spectra exhibit peaks denoting the N–H stretching of 2°-amines (a characteristic of weak aliphatic amines) at 3319 cm⁻¹, N–CH and Si–CH2 bending modes at 1422 cm⁻¹, a Si–O–Si asymmetric stretching out-of-plane shoulder peak at 1192 cm⁻¹, and a strong in-plane peak at 1078 cm⁻¹. The intensity of the Si–C peak at 798 cm⁻¹ decreases with increasing F-dopant. A growing peak at 953 cm⁻¹ is attributed to Si–F,30 while the 714 cm⁻¹ mode denotes –N–C–F with increasing F-dopants.

**Raman Analyses.** Raman spectra of pyrolysed-oxidized SiC (neat) and SiC–F–doped materials analyzed are shown in Figure 2d. The SiC (neat) spectrum peaks at 798 cm⁻¹ (sharp), 940 cm⁻¹ (broad), 1370 cm⁻¹, and 1596 cm⁻¹, attributed to the SiC transverse optical (TO) mode, longitudinal optical (LO) mode, and D band and G band for carbon, respectively. The D band is normally attributed to the presence of amorphous and disorder in carbon-derived materials, while the G band originates from the in-plane tangential stretching of the carbon–carbon bonds. Dominance of the G band over the D band in the SiC (neat) spectrum suggests the prevalence of the ordered in-plane stretching motion between sp² carbon atoms in SiC (neat). SiC–F (0.50) depicts the typical silica peaks at ∼420 and 485 cm⁻¹. The peaks denote the bending mode of oxygen with identical distortions of neighboring Si–O bonds (R line) in n-membered rings (n > 4) and defects in the silica lattice due
to the formation of four-membered rings (D₄ line), respectively. The absence of a vibration peak at 600 cm⁻¹ confirms the existence of solution-derived mesoporous silica. Introduction of F-dopants affects the D and G band positions and intensities.

D and G peak positions of SiC−F (0.25), SiC−F (0.50), and SiC−F (1.00) are (1362 and 1616 cm⁻¹), (1360 and 1608 cm⁻¹), and (1356 and 1610 cm⁻¹), respectively. The Raman D/G band intensity ratio is used to determine the graphic nature of carbon. The intensity ratios of the D peak over the G peak (I_D/I_G) from normalized Raman spectra, gave I_D/I_G = 0.87 for neat SiC, while those of SiC−F (0.25), SiC−F (0.50), and SiC−F (1.00) fluorinated samples were 1.50, 1.44, and 1.30, respectively. When compared to the neat SiC spectrum, the fluorine-modified spectra showed reduced G band intensities with increased D-band intensities, which confirms the existence of structural defects of carbons in SiC-based materials.

**BET Analyses.** Figure 3a−d presents the N₂ adsorption−desorption isotherm measurements of pyrolysed samples. The BET surface areas (BET-SAs) of pyrolysed samples were found to be 63.20 m² g⁻¹ [SiC neat], 60.70 m² g⁻¹ [SiCF (0.25)], 23.39 m² g⁻¹ [SiCF (0.50)], and 17.33 m² g⁻¹ [SiCF (1.00)], illustrating a declining order with an increase in the F-content. These materials exhibit type-IV N₂ adsorption isotherms. Pore-size distributions (PSDs) were calculated via the Barrett−Joyner−Halenda method using the adsorption isotherm. PSDs (3a−d) affirm the existence of micropores (diameter <2 nm) and mesopores in the 2−30 nm range.

The declining order of PSD suggests the closing up of mesopores with subsequent slow movement of N₂ molecules at 77 K through narrow pores. Reduced pore volume results in kinetically limited adsorption of the N₂ adsorbate. Adsorption−desorption isotherms depict 3H type of hysteresis for SiC (neat) and 4H type for SiCF (0.25), which are associated with slit-shaped pores and narrow slit pores, respectively. SiC−F (0.50) and SiC−F (1.00) present open hysteresis at low relative pressures, P/P₀ = 0.15, suggesting possible chemisorption of N₂ on the highest F-containing products.

**CONCLUSIONS**

In conclusion, biochar was used to form organic−inorganic hybrids with polysiloxane derived from sol−gel technology. Carbothermal reduction of as-prepared silica-biochar produced silicon carbide nanowires indexed to the β-phase with various morphological nanostructures. Fluorine-doped silica-biochar (SiOₓ/Cₓ−Fₓ) composites produced carbide materials that illustrated the reduction of nanowires and consequently forming an uneven fused flat surface, with increasing fluorine dopants. The external morphological changes were accompanied by decreasing the surface area from 60.70 to 17.33 m² g⁻¹ of low to maximum fluorine loading with progressive constricted pore volume. Reduction of SiCNW formation could be attributed to the internal structural defects and disorder on carbon materials of SiC samples during possible removal of Si from the sp³ Si−C bond via fluorination. The carbon disorder observed in a F-doped sample may be attributed to finite or nanoscale graphitic planes or other forms of carbon. The I_D/I_G ratio of F-doped samples decreases with increasing F-dopant, indicating an increase in the order of microstructures. Characteristic peaks of β-SiC and fluorine-doped materials depicted in FTIR spectra, Raman spectra, and XRD patterns corroborate with microstructures and chemical compositions of pyrolysed products determined by SEM−EDS and TEM analyses. The successful approach of employing waste byproducts as unconventional carbon sources for synthesizing silicon carbide offers an opportunity to further study and understand the reaction parameters leading to the preferred SiC morphologies.

**METHODS**

Biochar was carbonised in a furnace chamber under N₂ at 1000 °C for 60 min and then used as a carbon source. Sol−gel synthesis of the SiC precursor and its fluorine-doped derivatives, in SiOₓ/Cₓ and SiOₓ/Cₓ/Fₓ forms, proceeded as follows: silane, water, and organic solvent were mixed in a ratio of 3:1:3 for 4 h to obtain silica sol. Carbonised biochar and NH₄F in a molar ratio of 1:1 (mass ratio 4.2:9.0) were added successively to silica sol, homogenized for 2 h, and then left undisturbed to allow incorporation of silica into biochar. The product was dried in a 60 °C oven for 24 h, designated the SiOₓ/Cₓ/Fₓ (or F₁ (1.00)) as-prepared precursor. NH₄F contents were varied to obtain F-doped derivatives and SiOₓ/Cₓ (neat) without NH₄F. Precursors were carbonised in a furnace under an argon atmosphere at 1000 °C and then reduced at 1400 °C for 1 h. Light greyish to dark gray products were obtained with decreasing F-content. Characterization was done using field-emission scanning electron microscopy (SM-7600F), transmission electron microscopy (JEM-2100F), X-ray diffractometer (Rigaku SmartLab), Fourier Transform Infrared spectrometer (PerkinElmer Spectrum 100), Raman spectrometer (WTTech Alpha300 Ar⁺ laser), and Brunauer−Emmett−Teller surface area and pore-size analyzer (Micromeritics TriStar II 3020) instruments.

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The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript. All authors contributed equally.

**Funding**

The University of South Africa (UNISA) WIR support grant and National Research Foundation (NRF) supported this project financially.

**Notes**

The authors declare no competing financial interest.

**ACKNOWLEDGMENTS**

The authors extend their acknowledgment to the Institute for Development of Energy for African Sustainability (IDEAS) research unit (UNISA) for supplying biochar, Dr. Rudolph Erasmus (Physics department, Wits University) for Raman measurements, Dr. J.P. Mofokeng (Chemistry department, University of the Free State) for FTIR measurements, and Kabelo Ledwaba (Chemical Engineering department, UNISA) and Dr. H. Nyoni (NanoWS research unit, UNISA) for their coordinated assistance with BET measurements and other instrumentation.
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