

Fabrication of Silicon Carbide from Recycled Silicon Wafer Cutting Sludge and Its Purification

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Featured Application: This work may be used to develop the production of SiC by the carbothermal process with recycled Si ingot cutting sludge waste.

Abstract: Around the world, silicon carbide (SiC) is used as a raw material in several engineering applications because of its various beneficial properties. Currently, though the Acheson method is one of the most emblematic to manufacture SiC, the direct carbonization of metallic silicon is simple and beneficial. In this reaction, silicon wafer cutting sludge can be used as an alternative silicon source material. The silicon wafer sludge contains silicon, ethylene glycol, cooling water, and a small amount of impurities. In this study, SiC was synthesized using silicon wafer sludge by a carbothermal process. In a typical experiment, the silicon sludge was mixed with carbon at different molar ratios. Then, the mixture was turned into pellets, which were placed in alumina crucibles and heat-treated at a temperature from 1400 °C to 1600 °C to fabricate SiC. To deduce the optimum condition for the synthesis of SiC, an investigation was carried out on the effects of different mixing ratios, temperatures, and heating times. To ensure sufficient carbonization, excess carbon was mixed, and the synthesized SiC was characterized by X-ray diffraction (XRD). Subsequently, purification of the synthesized SiC products by oxidation of excess carbon was performed. The removal of extra carbon could be confirmed by XRD and attenuated total reflectance (ATR) spectroscopy. This process can give basic information for the development of a technology to produce SiC using recycling Si wafer cutting sludge waste.

Keywords: silicon carbide; Si wafer cutting sludge waste; recycling; carbothermal; oxidation

1. Introduction

Silicon carbide (SiC) is an inorganic compound that has been considered an important structural material, along with silicon nitride [1]. Depending upon its structure, it is classified as various polytypes phases; for instance, β -SiC (or 3C-SiC) has a cubic crystal structure, whereas α -SiC (or 6H-SiC) has a hexagonal structure [2]. SiC is also known as a rigid material, which has very high thermal, chemical, and mechanical properties. In addition to high mechanical strength and high thermal conductivity, SiC also possesses a low thermal expansion coefficient, large band gap, low density, high hardness, and excellent corrosion resistance [3–5]. SiC can also be used as an antacid and an antalkali, in sandpaper, grinding wheels, disks, wire saws, as well as many abrasive products. Also, it can be used for composite material applications with steel [6].

In many manufacturing processes that produce abrasive pipes, impellers, and pumping chambers, SiC is the preferred raw material because of its high hardness, abrasion, and strength stability over a wide temperature range [7]. Recently, it has been used in the production of ferroalloys using an electric furnace in order to improve the efficiency. Furthermore, SiC can be used to produce

many other products, including fixed and moving turbine components, suction box covers, seals, bearings, ball-valve parts, hot gas flow liners, heat exchangers, and semiconductor process equipment.

Various methods such as the Acheson method, Chemical Vapor Deposition (CVD), direct carbonization method, sol-gel method, gas-phase reactions, and self-propagation high-temperature synthesis have been used in the production of SiC [8]. Historically, Edward G. Acheson, while searching for a strong material by reacting clay and carbon, produced a small amount of SiC [9]. Clay and coke were mixed in an iron bowl, and a strong electric current flow was applied to the mixture through the electrode. After analyzing the product and working out the formula, it was confirmed that SiC had been synthesized. This method is known as the Acheson method, which uses high temperature, is suitable for producing α -SiC on an industrial scale, and remains the most common method in the world. However, for high-purity synthesis, the sol-gel method is more reliable than the Acheson method, as Si and C powders are used to produce a hybrid gel [10–12]. Moreover, Javier Narciso has done a lot of work on the synthesis of SiC and its composites. Most of his work was done by reactive infiltration, as they mention that this process is low-cost and is made in a low processing temperature [13–15]. Nevertheless, among all methods, direct carbonization is the most economical and effective as less energy is required when high-volume production is required. In the carbonization method, metallic silicon is mixed with carbon powder to produce β -phase SiC. This is the most beneficial and approachable method [16–18]. The reaction formula is given as:



On the other hand, the silicon sludge discharged from the silicon ingot cutting process can be used as a silicon source in this reaction. Currently, in the semiconductor and photovoltaic industries, a high-purity silicon ingot is being sliced for manufacturing Si wafers, and more than 40% of the ingot is lost to sludge in the cutting process [19–21]. The discharged silicon sludge is expected to be increasing worldwide, owing to the development of electronic devices and the growth of the photovoltaic industry. Two types of Si sludge are generated according to the cutting method. The first one is obtained by a slurry method in which a certain ratio of SiC powder is serving as an abrasive, and water and ethylene glycol are serving as a lubricant and a coolant. The slurry is continuously supplied to the sawing wire during the cutting process. Thus, the sludge consists of SiC, Si, iron powder, and cooling oil. To concentrate Si and SiC from the discharged Si sludge and to recycle it, proper methods other than landfill can be used, as described in several works [22–26]. Jung et al. have tried to produce a ferrochrome alloy successfully by reducing chromium ore using Si wafer sludge waste [27,28]. The second type of Si sludge is obtained through a recently developed method in which a silicon ingot is cut with a sawing wire that the diamond particles are electrodeposited on the surface. Oil or cooling water is supplied continuously to the sawing wire to remove heat generated during cutting and to discharge the kerf generated by cutting. Hence, the sludge generated in this process does not contain SiC but contains only oil, water, Si, and iron particles. So, if oil or water are removed, highly pure Si powder can be easily recovered by diverse methods such as by centrifugation and specific gravity separation [19–21].

Our aim in the present work was to utilize Si sludge waste by recycling it. Our plan was to recycle Si sludge in the presence of C and to fabricate SiC with moderate quality. In the present study, previously mentioned the second type of silicon sludge was used as the primary material to synthesize SiC powder from the recycling of waste material. In order to determine the optimum condition, an investigation was carried out on the effects of the mixing ratio of Si sludge and carbon and of the synthesis temperature, as well as on the purification conditions to obtain SiC powder. The synthesized SiC as well as raw materials were characterized by X-ray diffraction (XRD) analyses, attenuated total reflectance spectroscopy (ATR), and field-emission scanning electron microscopy (FE-SEM).

2. Materials and Methods

2.1. Raw Materials

In this study, the Si sludge used as the main raw material was obtained from the diamond saw-cutting process of a single crystal Si ingot. The other important material for this work was high-purity carbon powder (99.99%, <325 mesh), which was purchased from TASCOS (Seoul, Korea).

2.2. Methods

Si sludge was mixed with carbon powder in various ratios by 1 h ball milling. After mixing, pellets with a 13 mm diameter and 2–4 mm thickness were made by mold and press. The pellets were prepared at 300 psi pressure. Then, the pellets were placed in an alumina crucible and heated at different temperatures in a Kanthal Super furnace (Korea Furnace Development Co., Yangju, Korea), as shown in Figure 1. The alumina reaction tube (OD 60 mm × ID 50 mm × L 600 mm; Samwha Ceramics Co., Seoul, Korea) was enclosed with a water-cooled jacket. High-purity argon gas was continuously fed into the reaction tube at a flow rate of 300–500 SCCM during the heat treatment. Although the holding time at the reaction temperature was the same in every synthesis (240 min), the reaction temperature was varied between 1400 °C and 1600 °C, as shown in Table 1. After holding at the maximum temperature, the pellets were cooled to room temperature. The pellets were then crushed into powder for characterization.

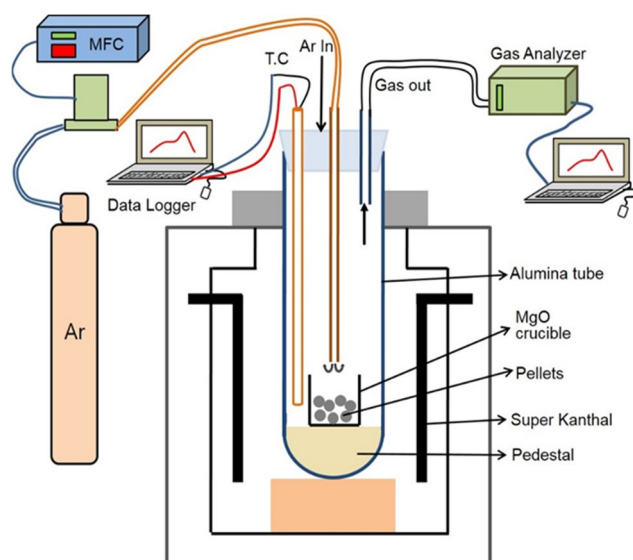


Figure 1. Schematic diagram of the experimental settings.

Table 1. Preliminary experimental conditions for the synthesis of SiC from Si sludge.

Mixing Ratio (Si Sludge/C)	Reaction Temp. (°C)
1:1.2	1400
1:1.4	1550
1:4.0	1600

Raw material as well as the obtained product were characterized by several analyses. The particle-size distribution for the starting material, i.e., Si sludge, was done with Multisizer 4e (Beckman Coulter, Brea, CA, USA). For the XRD measurement, the powder was sieved through 200 mesh and was analyzed using a RIGAKU D/MAX-2500V (Rigaku, Tokyo, Japan) and Cu K α radiation under an accelerating voltage of 40 kV and a current of 200 mA. The diffraction data for the obtained product powder were recorded at 2 θ angles between 10° and 90°. Again, for morphological analysis, FE-SEM and energy dispersive X-ray spectrometry (EDS) analysis were performed using a JEOL

JSM-7401F (JEOL, Tokyo, Japan) at a voltage of 10.0 kV. Similarly, attenuated total reflectance (ATR) was recorded on a combinatory vibrational spectrometer (Nicolet iS 50, Thermo Fisher Scientific Inc., USA). In addition, the carbon removal treatment was performed using the same furnace employed in the heat treatment during synthesis, in the open air.

3. Results and Discussion

3.1. Raw Material Characterization

In the present work, the starting materials were silicon sludge and pure carbon powder. Figure 2 shows the XRD pattern of the silicon sludge, with only silicon peaks visible. Figure 3 shows the scanning electron microscopy (SEM) image and EDS analysis results. Most of the particles were found to be silicon, as shown in Figure 3b. Moreover, it was found, from the SEM image in Figure 3, that the shape of the particles was elongated, irregular, and of diverse sizes, which corresponded to the particle analysis shown in Figure 4. The average particle size in the silicon sludge was 1.6 μm , and most particles were under 3 μm in size. Although only silicon was recognized in the XRD data shown in Figure 2, small amounts of carbon and oxygen were also detected, as shown in Figure 3. This was attributed to the coolant solution used with the diamond saw in the silicon ingot cutting process. It was estimated from the content of oxygen that the particle containing oxygen was not silicon oxide and was only partially oxidized. Since the amount of oxygen and carbon was quite small, a minor effect was expected in the synthesis of SiC in the present work.

The XRD pattern for carbon used in this work showed a strong, sharp peak near $\sim 2\theta = 26^\circ$, which corresponds to the stacking of carbon-like crystalline graphite.

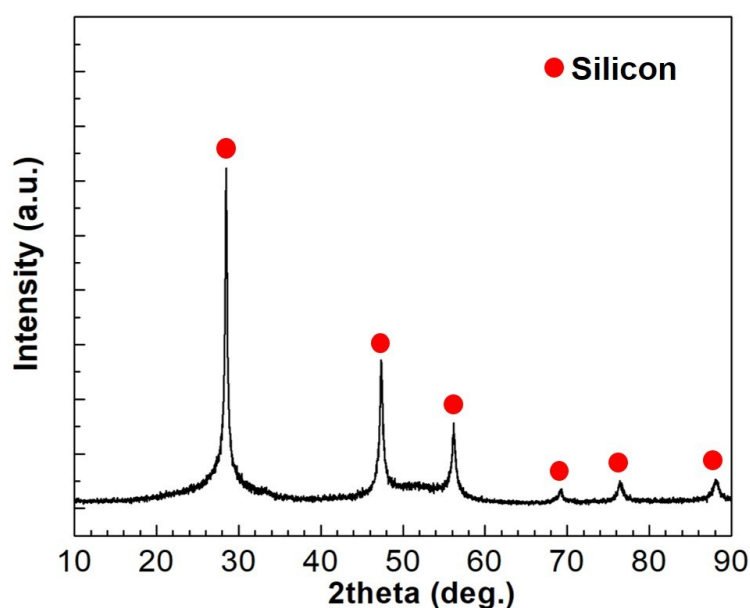


Figure 2. XRD pattern of the Si sludge which was used in the present study.

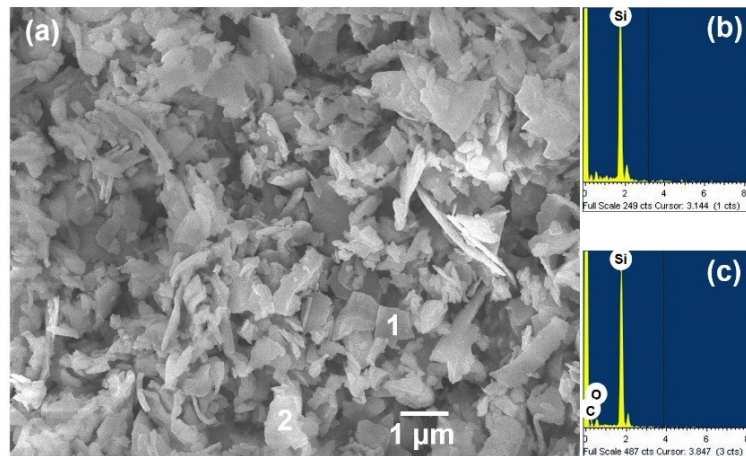


Figure 3. (a) SEM image of the Si sludge, (b) and (c) energy dispersive X-ray spectrometry (EDS) spectra for points 1 and 2, respectively.

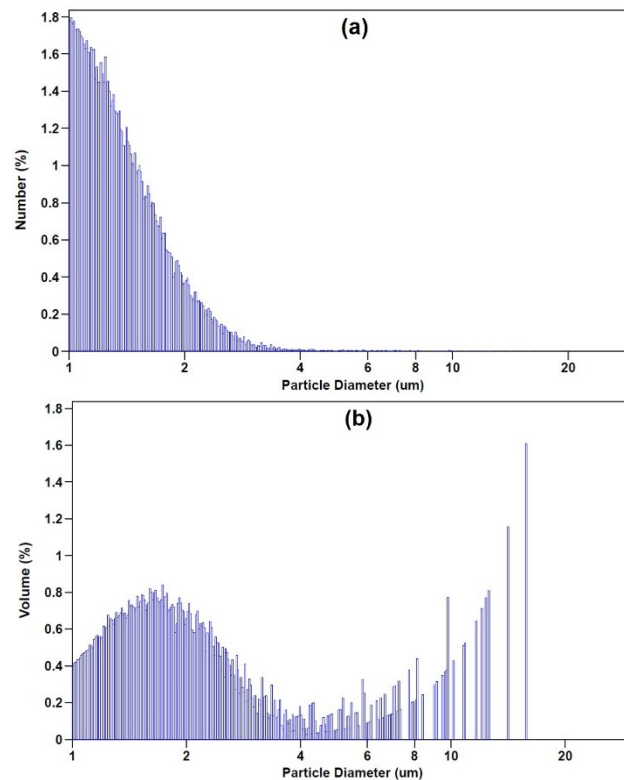


Figure 4. Particle analysis of the Si sludge used in the present study: (a) number of particles, (b) volume of particles.

3.2. Synthesis of SiC

In a preliminary test, the Si sludge and C were mixed in such a ratio that, after the synthesis, no excess silicon would remain in the product, as Si removal is a complicated process. The heat treatment was also performed at various temperatures to get a rough idea on the formation of SiC from Si sludge and C. Table 1 shows the preliminary experimental molar ratios and temperatures. The XRD patterns of the synthesized products with different molar ratios at different temperatures are shown in Figure 5. All the mixtures were suitable to form SiC with an excess amount of C at the provided temperatures. However, it was clearly seen that a higher amount of SiC was formed at 1550 °C from a mixture with a Si sludge-to-C molar ratio of 1:1.4, than from mixtures with 1:1.2 and 1:4 molar ratios at 1400 °C and 1600 °C, respectively.

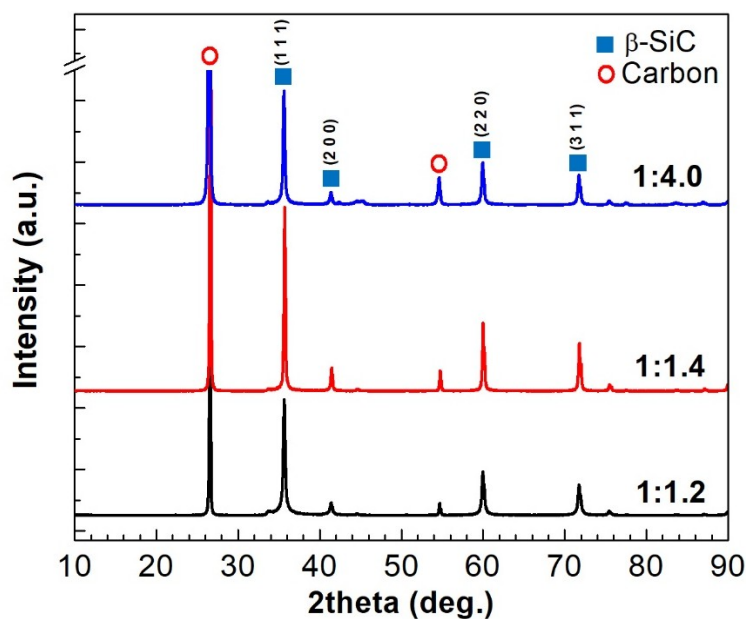


Figure 5. XRD patterns of the products after SiC synthesis treatments.

On the basis of this preliminary experimental result, the mixing ratio was fixed at 1:1.4, and then the temperature of the heat treatment was changed to find the optimum temperature for SiC formation. The comparison among the XRD patterns of SiC synthesized with a Si sludge-to-C molar ratio of 1:1.4 at different temperatures and with the raw mixture is shown in Figure 6. At 1450 °C, the reaction between the Si sludge and C was not complete, a significant amount of both Si and C remain unreacted, and, as a result, very little SiC formed. A small increase in temperature, i.e., to 1500 °C, showed that SiC formation was possible, and the amount of SiC formed at 1550 °C was even higher than that at 1500 °C.

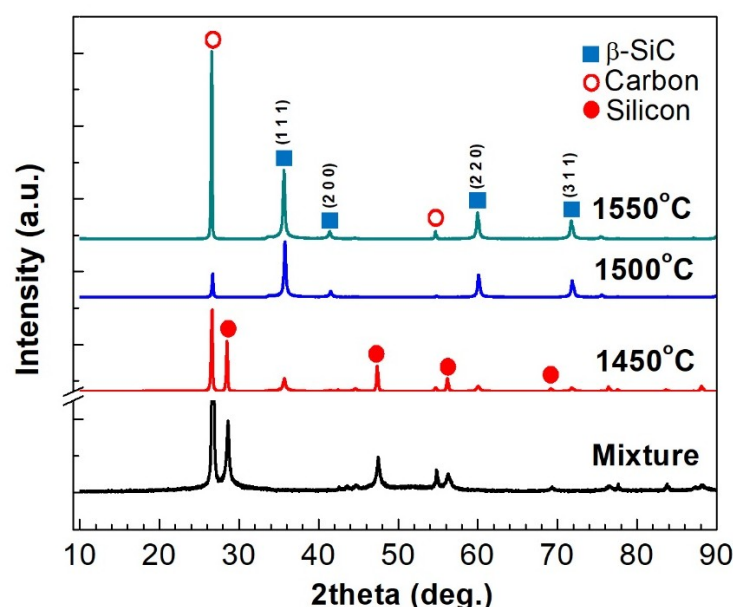


Figure 6. XRD patterns of the products after SiC synthesis at different temperatures (Si sludge-to-C molar ratio = 1:1.4).

Since the temperature of 1550 °C was suitable for the formation of SiC, in order to find the optimum mixing ratio, different ratios of Si sludge to C were tested at 1550 °C. Figure 7 shows their

XRD patterns and reveals that a Si sludge-to-C molar ratio of 1:1.4 was the best, as it contained a large amount of SiC.

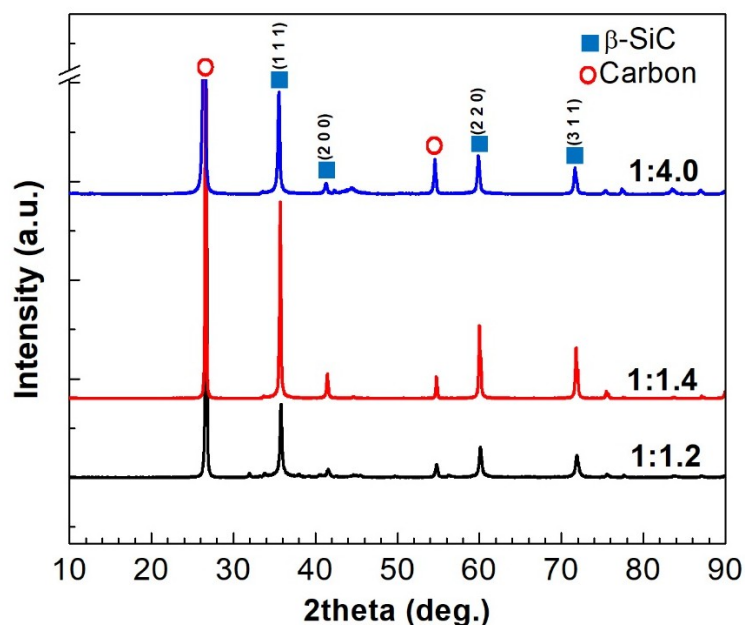


Figure 7. XRD patterns of the obtained SiC with different Si sludge-to-C molar ratio at 1550 °C.

Three types of experiment were performed to find the optimum conditions to synthesize SiC from Si sludge in the presence of C. From these experimental results, i.e., on the basis of the formation of more SiC with minimum impurity, the 1:1.4 molar ratio of Si sludge to C led to SiC formation at 1550 °C in an Ar atmosphere. However, as some C remained in the synthesized SiC as an impurity, the removal of C is necessary to obtain pure SiC.

3.3. Purification of SiC

To remove excess C from the obtained SiC, the product was heat-treated in air at different temperatures. We assumed that C could be removed as CO or CO₂ when it was heated in the presence of air. SiC powder synthesized at 1550 °C from a Si sludge-to-C molar ratio of 1:1.4 was used in the carbon removal experiments. Figure 8 shows the XRD patterns of SiC powder after carbon removal treatment for 1h at different temperatures. It can be seen in Figure 8 that, at 750 °C, the significant peak for C ($2\theta = 26.4^\circ$) is small, compared to those obtained for SiC treatments at 550 °C and 650 °C. In contrast, there is no significant change in SiC.

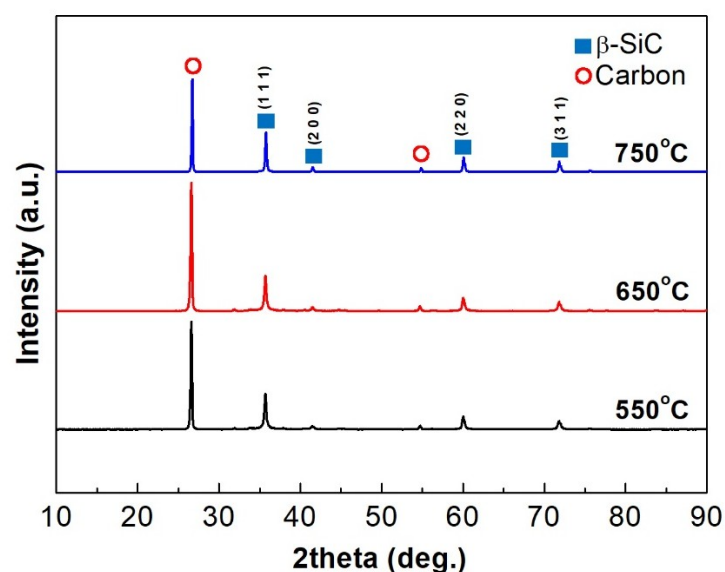


Figure 8. XRD patterns of the SiC products after heat treatment for 1 h at different temperatures.

To confirm the effect of the treatment time on carbon removal from SiC products, heat treatment at 750 °C was performed at different times. A decrease in weight of the synthesized SiC powder was recognized after the carbon removal treatment. Figure 9 shows the XRD patterns for SiC powder at different carbon removal treatment times. It was found that the carbon peak ($2\theta = 26.4^\circ$) decreased with the increase of the treatment time. For the complete removal of carbon, SiC was heated for 300 min at 750 °C, as shown in Figure 9. The trend of C reduction is shown in Figure 10, which represents the change of intensity for the main peak of carbon with the increase of carbon removal treatment time. Apparently, the intensity of carbon decreased with the increase of the treatment time. According to the graph, carbon was removed rapidly up to 180 min but, after that, the process became slow. This may be attributed to the decrease of C.

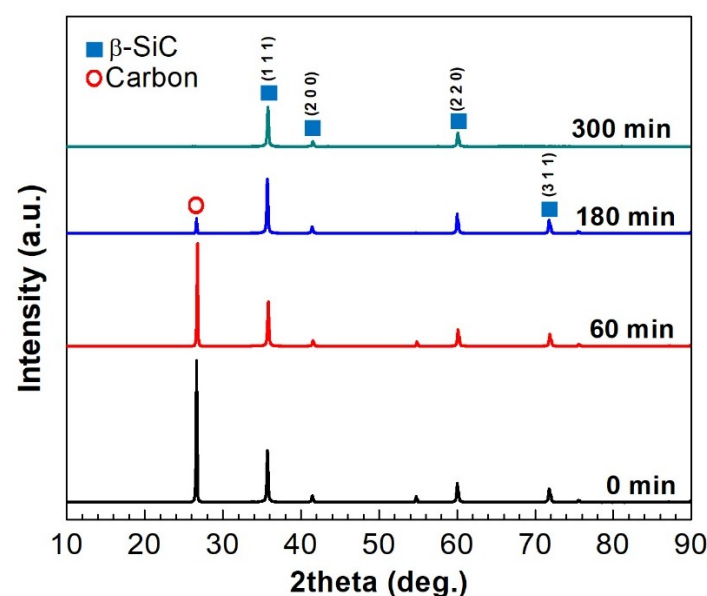


Figure 9. XRD patterns of the SiC powder product at different carbon removal treatment times ($T = 750^\circ\text{C}$).

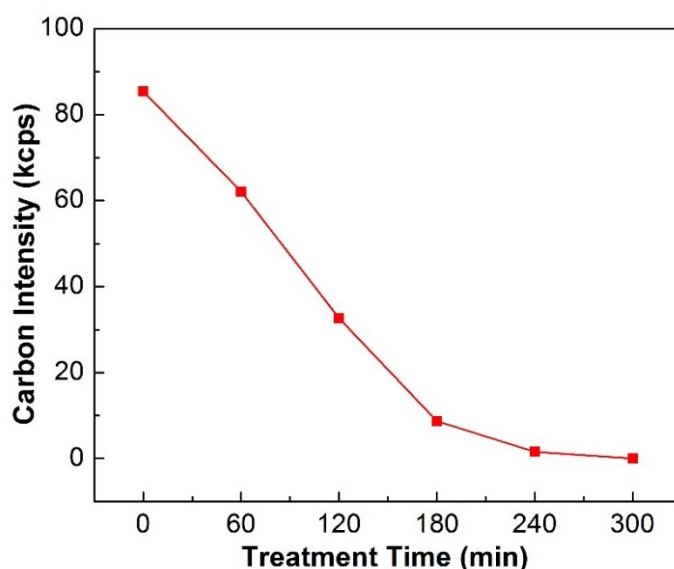
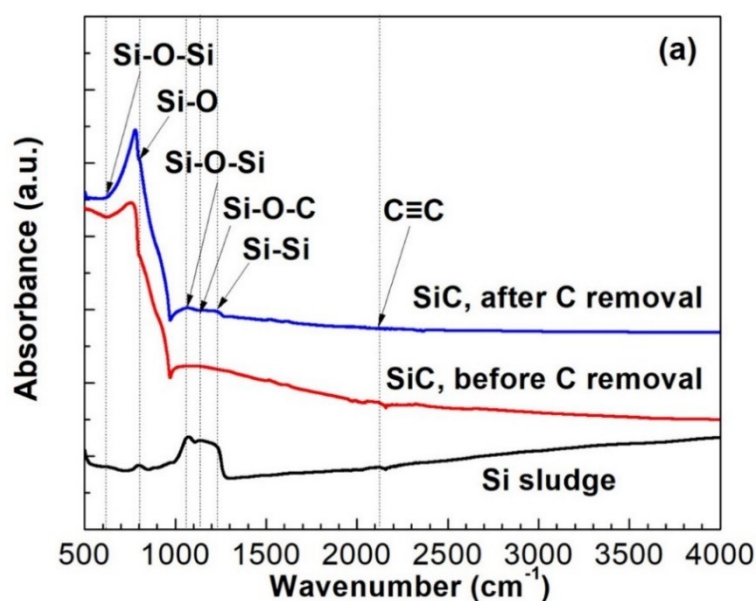


Figure 10. Variation of intensity of the 26.4 degree peak in the XRD patterns for SiC powder products with different carbon removal treatment times.

A confirmation of the removal of C could be obtained from the ATR spectra that also helped to get more information about the morphology of the samples. Figure 11a shows the comparison of SiC before and after the removal of C, along with the Si sludge. The Si sludge shows peaks at 618 cm^{-1} , 1065 cm^{-1} , 800 cm^{-1} , 1134 cm^{-1} , 1225 cm^{-1} , and 2124 cm^{-1} , which belong to Si–O–Si, Si–O, Si–O–C, Si–Si, and C≡C bonds, respectively. This analysis showed similarities with other analyses indicating that the Si sludge contains a little amount of C and O. SiC synthesized at the optimum condition showed peaks at 760 cm^{-1} , 1047 cm^{-1} , and 2110 cm^{-1} , corresponding to Si–O, Si–O–C, and C≡C bonds [29–33]. Both Si sludge and synthesized SiC contain carbon in the samples with trace amounts of oxide. However, after 300 min of heat treatment at $750\text{ }^{\circ}\text{C}$, the peak for C≡C disappeared, as seen in the enlarged spectra in Figure 11b. Both the XRD and the ATR analyses confirmed the formation of pure SiC. Therefore, the removal of extra carbon in SiC products can be verified through ATR (or FT-IR) analysis.



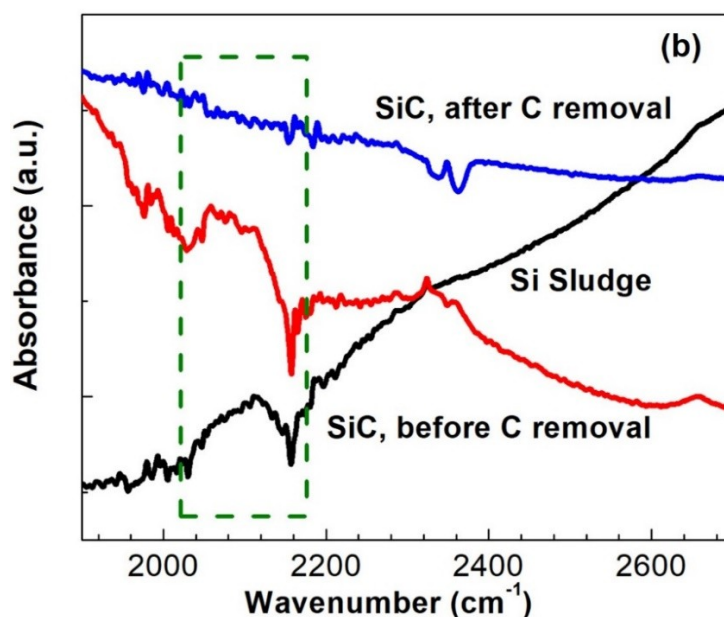


Figure 11. Comparison of attenuated total reflectance (ATR) profiles for SiC powder products before and after the carbon removal treatment, (a) normal scale, (b) enlarged profile around the C≡C peaks in (a).

The morphology of the synthesized SiC, before and after C removal, is shown in Figure 12a,b respectively. Both SEM images show a similar surface morphology with faceted features of few microns in size. However, in Figure 12a, there are some large black particles which are estimated to be covered with C particles. From the EDS pattern (inset of Figure 12a), it can be confirmed that carbon-rich parts are present. The elemental compositions for different points in Figure 12 are shown in Table 2. Before the carbon removal treatment, the amount of excess C in the synthesized SiC was high (point 1 and 2), whereas, after heat treatment, Si and C were found to be 69.05 and 30.95 in wt%, respectively. This is close to the theoretical value of pure SiC (Si:C = 70:30 in wt%).

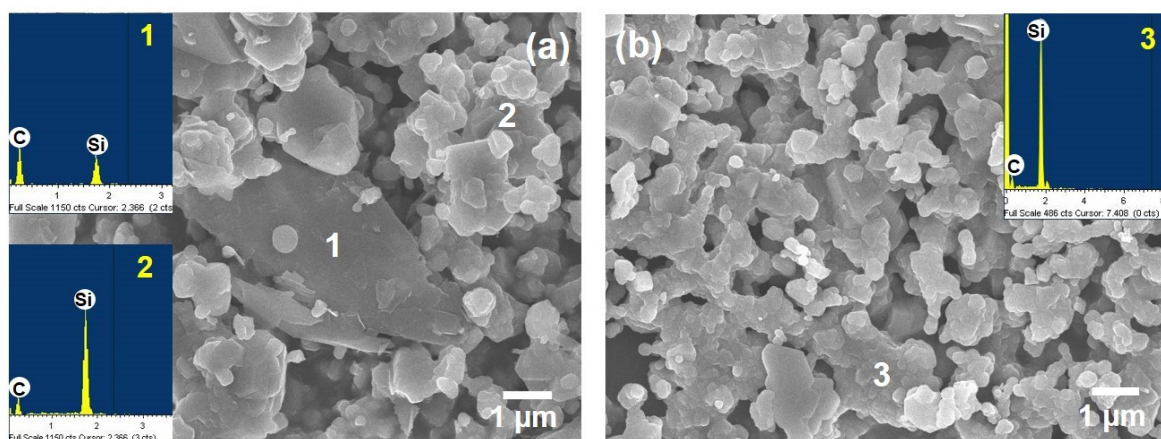


Figure 12. Microstructure of SiC powder (a) before and (b) after the carbon removal treatment; inset of (a): top, EDS spectra at point 1; bottom, EDS spectra at point 2; inset of (b): EDS spectra at point 3.

Table 2. EDS results for the SiC powder products (related to Figure 12).

Point	C (wt%)	Si (wt%)
1	48.35	51.65
2	76.86	23.14
3	30.95	69.05

4. Conclusions

In the present work, SiC was synthesized using recycled silicon sludge waste generated from the silicon wafer cutting process. The synthesis of SiC was performed by the carbonization process in various experimental conditions; it was found that the optimum Si sludge-to-C ratio was 1:1.4 and the optimum experimental temperature was 1550 °C. The formation of the SiC phase was confirmed by XRD, SEM, and EDS analyses. To purify the SiC products containing excess carbon, an open-air heat treatment was performed at 750 °C, resulting in the oxidation of excess carbon. The excess carbon decreased with the increase of the carbon removal heat treatment time. The removal of carbon in the SiC powder could be verified by ATR analysis.

Author Contributions: W.-G.J. conceived and designed the experiments; S.T.H. performed the experiments; S.T.H. and F.T.J. did the characterization and data analysis; W.-G.J., S.T.H. and F.T.J. wrote the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

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