Synthesis of Highly Nanoporous β -Silicon Carbide from Corn Stover and Sandstone

Tongtong Wang,^O Weibo Gong,^O Xin He, Zuhao Kou, Gang Tan, Shaojun Zhou, Hongtao Yu, Maohong Fan,* and Harold H. Kung*



synthesized with a high yield. The synthesized β -SiC was characterized by XRD, Raman, SEM, TEM, and TGA, and the gaseous products were also analyzed with an integrated furnace-MS system. The results show that the produced β -SiC exhibited a nanostructure that followed the graphitic carbon template derived from the pyrolysis of the corn stover. The surface area as high as 397 m^2/g , the pore volume of 0.4 cm³/g, as well as the majority pore diameters of 3-6 nm were achieved. CO and CO2 were released



during the reaction between vaporized SiO and graphite. The effect of temperature in the range of 1000 to 1700 °C was also studied, and the results point to a strong dependence between the process temperature and the yield and density of β -SiC. Also, the possible mechanism of synthesized β -SiC was proposed and confirmed with experimental results. This study provides a simple and an ecofriendly carbothermal reduction approach to produce nanoporous β -SiC with agriculture waste and sandstone, which could help establish the green economy in the US.

KEYWORDS: corn stover, sandstone, nanoporous microstructure, beta-silicon carbide

INTRODUCTION

 β -silicon carbide (β -SiC) is an important material due to its high thermal conductivity, low thermal expansion coefficient, and excellent mechanical and chemical stability, which makes it robust against oxidation, corrosion, and thermal shock and useful in harsh environments.^{1,2} Porous β -SiC with a high surface area has a lot of important applications. For example, SiC is an excellent catalyst support material in hydrocarbon fuels reforming,³⁻⁷ automotive exhaust treatment,⁸ and selective isomerization of paraffinic hydrocarbons⁹ and others. Also, in purification technology, β -SiC is used as membrane components for filtration, ion-exchange, reverse osmosis, and absorption/adsorption.¹⁰⁻¹² Other applications of SiC such as sensor manufacturing and enzyme immobilization are also promising.^{13–15}

Traditional high-temperature processing to produce β -SiC results in low surface area materials (~ 20 m² g⁻¹).¹⁶ In the abovementioned studies, many efforts have been made to prepare high surface area SiC materials ($\sim 100 \text{ m}^2/\text{g}$) with excellent oxidation resistance under high temperatures. Wang and Fu produced SiC material with a surface area of 135 m^2/g

via the sol-gel method that employed furfuryl alcohol as the carbon source and tetraethoxysilane as the Si source. Modification of this method by adding polymethyl-hydro siloxane as a soft template resulted in more porous products.¹⁸ Pol et al. reported a pyrolytic method that used a triethyl silane precursor for producing β -SiC at 800 °C within 3 h. The product had a high surface area of 656 m^2/g with a pore size diameter of 2-4 nm.¹⁹ Parmentier et al. used pyrolitic carbon and organized mesoporous silica produced from hexadecyltrimethylammonium bromide as the organic template, sodium hydroxide solution, and colloidal silica to synthesize the high surface area β -SiC (120 m²/g).²⁰ β -SiC materials with the higher surface area were produced by taking advantage of the porous structures from carbon template. For example,

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Figure 1. Schematic diagram of the experimental setup for the synthesis of β -SiC. (1) mass spectrometer, (2) computer, (3) high-temperature horizontal tube furnace, (4) temperature controller, (5) mass flow controller, (6) mass flow controller power supply/control module, and (7) argon cylinder.

mesoporous carbon (1385 m²/g) was applied to produce porous β -SiC with silicon powder (Si + C = SiC) at a temperature of 1200 °C, and the produced β -SiC achieved a surface area of 147 m²/g.²¹ However, the expensive silane compounds and carbon templates make these methods less attractive.

Nowadays, using biomass as a carbon source and template instead of petroleum coke in the traditional Acheson process for β -SiC production can not only produce the high porous structures derived from the porous carbon of carbonized nature biomass but also significantly reduce the environmental impact. Indeed, it has been demonstrated that β -SiC whiskers and nanotubes can be formed by carbothermal reduction of mesoporous silica (SBA-15) with sucrose-derived carbon.²² β -SiC can also be produced by the reaction of SiO₂ with a solid form of biomass, such as wood-derived charcoal²³ or residues of biomass gasification.²⁴ Another approach is to pyrolyze biomass that has a significant Si content, such as rice husk.^{25–31} Among them, high surface area β -SiC nanowires $(\sim 100 \text{ m}^2/\text{g})$ were synthesized using the KOH activated Crambe stalk (~900 m^2/g) with silicon powder at 1500 °C for 3 h. The reduced surface area may result from the micropore occlusion and coarse particles with a low surface area.³² The raw rice husks containing a high concentration of silicon were used to prepare the β -SiC with a surface area of 150 m²/g.³³ Liu et al. prepared the β -SiC/C porous ceramic using pine wood-derived carbon, silicon powder, and $Fe(NO_3)_3 \cdot 9H_2O$ as a catalyst at 1300 °C with pore sizes in the range of $0.3-3 \,\mu\text{m}$. However, the prepared β -SiC/C porous ceramic had ~37 wt % weight loss in the air at ~737 °C.³⁴ Carbon (965 m²/g) from CO₂-activated plane tree seeds (850 °C for 1 h) and tetraethyl orthosilicate as a silicon source were used to prepare ultralong β -SiC nanowires at 1400 °C for 2 h,³⁵ although the specific surface area of the prepared β -SiC was not reported. Hence, the challenge in the fabrication of high porous β -SiC as catalyst supports structures is the complex carbon template process and the expensive silicon source. Consequently, economical and efficient synthesis routes for high porous β -SiC still needs to be further studied.

Corn stover (CS) is the non-grain part of a corn plant with high cellulose content.³⁹ CS in the US accounts for about half

of the crop yield with an estimated 254 million tons $yr^{-1}\ in$ 2001.³⁶⁻³⁸ Although 5% CS is used for cattle grazing and bedding,³⁷ most of CS as agricultural residues is usually left on the soil surface after harvest.³⁹ Furthermore, the high nitrate concentration in CS may cause nitrate poisoning of animals.^{40,41} Thus, over 90% of CS produced in the US has no practical application.⁴² On the other hand, CS is rich in carbon and its bio-microstructure makes it a potential raw material for producing high surface area porous β -SiC because the structure of SiC highly depends on that of the carbon source.⁴³ Therefore, the new production technology using CS can open a door toward converting low-value CS to high value added functional SiC materials. Furthermore, the reaction of glucose (as a representative biomass chemical) with SiO₂ is thermodynamically favorable $(2SiO_2 + 2O_2 + C_6H_{12}O_6 = 2SiC)$ + 4CO₂ + 6H₂O, Δ H[°] = -342 kJ). Here in this study, we have demonstrated that CS can be converted successfully to high nanoporous β -SiC with yields up to 96% using sandstone (SS) as the natural Si source with high SiO_2 content (96.65%) from the state of Wyoming (average price, ~\$50 per ton). The conversion of these two inexpensive raw materials was achieved by a simple and eco-friendly carbothermal reduction process, resulting in establishing the green economy in the US.

EXPERIMENTAL SECTION

Materials. CS was collected from an agricultural test filed at the University of Wyoming. Before the experiment, CS was chopped and milled into particles with a size of 0.1–0.5 mm and dried at 120 °C in a vacuum oven for 12 h. SS was collected from Plumbago Creek in the state of Wyoming. The collected SS was pulverized to 75 μ m particles, then treated with 1 M hydrochloric acid (10 mL per gram of pulverized SS) overnight to remove metallic impurities, and washed using deionized water until the pH was 7. After which, the obtained sample was filtered and dried in a vacuum oven at 90 °C for 12 h.

β-SiC Synthesis. A mixture of CS and SS, weight ratio of 4:1, was placed in an alumina crucible in a horizontal tube furnace (Carbolite Gero, HTRH 40/500/18, Figure 1). The mixture was heated to 1000–1700 °C with holding times of 0–4 h at a heating rate of 20 °C/min under 50 mL/min argon gas flow. After cooling the furnace to room temperature, the obtained product was collected and weighed (m₀). A mass spectrometer (MS, HIDEN, HPR-20) was used to

analyze the gaseous products generated during the β -SiC synthesis process.

Characterization. The proximate and ultimate analysis of CS was determined by following ASTM D5142-02 (Standard Test Methods for Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures). The organic fraction in CS was determined by the weight loss after air oxidation at 800 °C for 3 h. The C, H, N, and S composition of CS was measured using a Vario MACRO cube elemental analyzer (Elementar Analysensysteme GmbH, Germany), and the O content was determined by calculating the weight difference between the organic fraction and the C, H, N, and S. The element components of the SS and CS ash were analyzed by inductively coupled plasma mass spectrometry (ICP-MS; NexIon 350, PerkinElmer). For the ICP-MS test, 0.03 g sample was mixed with 0.24 g lithium metaborate (LiBO₂, \geq 98%, Sigma-Aldrich), and heated at 1100 °C for 5 min in a cube oven (Carbolite Gero, RHF 16/3), then fully dissolved in 5 wt % nitric acid (HNO₂, ACS reagent, Sigma-Aldrich). The obtained solution was diluted with deionized water to appropriate concentrations within the working range of ICP-MS.

The density of synthesized β -SiC was determined by a Mettler Toledo balance (ML204T/00) equipped with a density kit (ML-DNY-43). To measure the yield of synthesized β -SiC, the obtained product was first treated at 700 °C in 100 mL/min flowing air for 5 h in a tube furnace (Lindberg/Blue M, Thermo Scientific) to remove the unreacted carbon, and then washed in 1 M HNO₃ for 12 h under vigorously stirring, and then rinsed with deionized water. The resulted solid was further washed using 1 M sodium hydroxide (NaOH, \geq 98%, Sigma-Aldrich) with vigorous stirring for 7 days to remove the unreacted SiO₂ followed by washing with deionized water until the pH of the filtrate reached 7. After drying, the weight of the solid, m₁, was measured. The yield of products β -SiC was calculated by eq 1

yield (wt %) =
$$\frac{\mathrm{m_l}}{\mathrm{m_0}} \times 100\%$$
 (1)

The antioxidation ability of synthesized β -SiC was quantitatively determined by a thermogravimetric analyzer (TGA; SDT-Q600, TA instrument). The structure was characterized by Raman scattering (DXR2, Thermo Scientific) using a 532 nm laser and X-ray diffraction (XRD; Smartlab diffraction system; Rigaku; Cu K α_1 radiation at λ = 1.54056 Å, at 40 kV and 40 mA). The morphology of synthesized β -SiC was obtained with scanning electron microscopy (SEM; Quanta 250, FEI) and transmission electron microscopy (TEM; JEOL JEM-2100, equipped with a high-resolution (HR) style objective lens pole piece). The specific surface area and pore size distribution of the prepared samples were measured with a Brunauer-Emmett-Teller analyzer (BET, Quantachrome, Autosorb-iQ system), and the pore size distribution was calculated by the non-localized density functional theory (NLDFT) method. The CO_2 temperature-programmed desorption experiment was performed using a chemisorption analyzer (Quantachrome, Autosorb IQ). SiC sample (100 mg) was loaded into the sample cell, and the temperature was kept at 150 °C with a helium flow for 60 min to remove the adsorbed species on the sample. Then, the system temperature was cooled down to 50 °C, and the CO₂ adsorption process was performed at 50 °C for 90 min, after which the catalyst was purged with helium gas for another 60 min to remove the weakly adsorbed CO2. Next, the CO2 desorption behavior was recorded with the temperature increasing from 50 to 800 °C.

RESULTS AND DISCUSSION

The results of the proximate and ultimate analysis and the inorganic ash compositions of CS are listed in Table 1. CS (81.1%) was volatile, which corresponds to structural compounds like cellulose, lignin, and nonstructural organic matters.⁴⁴ For the nonvolatile CS ash, SiO₂ and K₂O accounted for 31.94 and 40.24%, respectively, and the rest were oxides of iron, magnesium, manganese, calcium, and other metals. The elemental composition of SS is shown in Table 2. The main component of acid-treated SS was SiO₂

Table 1. Proximate Analysis, Ultimate Analysis, and the Ash Content of CS

proximate analysis (wt %)				
fixed carbon	volatile	moisture	ash	total
5.8	81.1	8.3	4.8	100.0
ultimate analysis (wt %, ash-free)				
С	Н	Ν	S	0
48.54	6.04	0.67	0.02	44.73
inorganic ash composition (wt %)				
SiO ₂	Al_2O_3	Fe_2O_3	MgO	CaO
31.94	8.65	3.87	4.23	6.18
inorganic ash composition (wt %)				
K ₂ O	Na ₂ O	MnO_2	BaO	TiO ₂
40.24	3.41	0.09	0.04	0.35

(96.65%) with the balance being oxides of iron, aluminum, calcium, potassium, and sodium. As expected, its the XRD pattern (Figure 2f) showed only peaks of quartz SiO₂.

The dried CS as obtained (Figure 2a) was chopped and milled to 0.1-0.5 mm particles (Figure 2b). As shown in the SEM image (Figure 2c), the vascular system of the CS formed a network of interconnected pores with different sizes, which will result in the highly porous SiC with a high surface area. Figure 2d shows the weight change of CS during pyrolysis. Loss of bound water in cells occurred before 150 °C.4 Pyrolysis began at around 170 °C and most of the reaction proceeded until 407 °C, then continued slowly to 1200 °C with the release of CO₂, CO, and H₂O. The solid product was graphite, as indicated by the 2D peak in the Raman pattern for the CS after pyrolysis in Figure 2e.46,47 The final yield of graphite was 29.46%. The decomposition and volatilization of CS could provide the porous carbon template $(589 \text{ m}^2/\text{g with})$ a pore volume of 0.65 cm³/g shown in Figure 5g,h) for the subsequent synthesis of porous β -SiC.

XRD and Raman were used to follow the phase changes during the β -SiC synthesis process when the pyrolyzed CS and SS mixture was heated from 1000 to 1700 °C. The results are shown in Figure 3a,b, respectively. When the temperature was below 1200 °C, the peaks of cristobalite SiO₂ in the XRD patterns, as well as the D and G bands of graphite in the Raman spectra, become more intensive with the increasing temperature, indicating the crystallization of amorphous SiO₂ from quartz to cristobalite and the formation of more graphitic carbon.¹⁶ The intensity of the cristobalite SiO₂ peaks reached the maximum when the processing temperature was at 1300 °C, then decreased and finally disappeared at 1500 °C. This implies the reaction between the cristobalite and the carbon derived from CS. Peaks of β -SiC appeared at around 1300 °C in the XRD spectra and at 1400 °C in the Raman spectra. The intensities of the β -SiC peaks increased with increasing temperature, due to the more crystallized structures at a higher temperature. It should be noted that when the processing temperature was at 1700 °C, α -SiC was detected by both XRD and Raman; however, the α -SiC should be the major phase at the higher temperature (~2100 °C).^{48,49} The formation of α -SiC at lower temperature could be due to the catalytic effect of the various metal oxides in the mixture, such as oxides of iron, magnesium, manganese, calcium, and others.⁵⁰⁻⁵² Peaks of graphitic carbon appeared in XRD and Raman scattering (D and G bands) when the temperature was

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Table 2. Elemental Composition as Metal Oxides of Acid-Treated SS by ICP-MS





Figure 2. Characterization of starting materials. (a) Photographic images of CS and SS, (b, c) SEM image of CS, (d) TG and differential thermal gravimetry (DTG) of CS in an inert gas atmosphere, (e) Raman spectra of CS after pyrolysis at 1600 °C, and (f) XRD pattern of acid-treated SS powder.

at around 1000 °C. The intensities of these peaks increased with increasing temperature up to 1300 °C and began to decrease at higher temperatures in XRD, whereas they increased up to 1600 °C before disappearing at 1700 °C in Raman. The difference in temperature with the two techniques was likely due to the fact that XRD is a bulk technique and more sensitive to heavier elements and crystalline structures, whereas Raman scattering is more surface-sensitive and less dependent on three-dimensional long-range orders. With either technique, the results indicate that the carbon from CS reacted with SiO₂ in SS at around 1300–1400 °C to form β -SiC. When the temperature was increased to 1700 °C, the reaction was almost complete.

Based on these temperature ramp results, 1600 °C was used as the optimal synthesis temperature to further study the fast reaction process and the β -SiC yield. The effect of the reaction time at 1600 °C was studied from 0 (very short) to 4 h. The results, shown in Figure 3c,d, indicate that more crystalline β -SiC was obtained by increasing the reaction time from 0 to 2 h. However, further increasing the processing time to 4 h resulted in the appearance of the α -SiC phase. It is interesting to note that in the Raman spectra, peaks (D and G band) that were assigned to graphitic carbon decreased in intensity with the increasing reaction time up to 2 h but remained unchanged afterward. This indicates that the reaction of carbon with SiO₂ was complete in around 2 h due to the depletion of SiO_2 . Therefore, further heating after 2 h of the reaction did not increase the yield of β -SiC, and the formed β -SiC slowly transformed into the α -phase. This optimal condition for the synthesis of β -SiC is 1600 °C at 2 h of the reaction, which is shorter than other reported processes.^{27,32}

The gases evolved during the synthesis reaction was monitored with an integrated furnace-MS system. As shown in Figure 3e, the evolution of CO and CO_2 began to be detected at about 400 °C, indicating the decomposition of the CS. At this low temperature, there was no sign of the SiO₂ reduction. When the temperature was above 500 °C, the further decomposition of CS resulted in more CO and the formation of graphitic carbon. CO evolution continued at a slow reaction until the temperature reached 1300 °C, after which the reaction rate increased rapidly with increasing temperature, indicated by the more CO and CO₂ realizing, as shown in MS. At this point, the SiO₂ reduction by graphite occurred, and β -SiC was formed as indicated by the XRD and Raman results. This description of the synthesis was also consistent with the visual examination of the solid products (Figure 3e, insert). The grayish black mixture of carbon and SiO₂ changed to green at around 1600 °C, which is the characteristic color of β -SiC.

The yield of β -SiC and the density of the synthesized mixture, which includes β -SiC, unreacted SiO₂, and carbon, are plotted in Figure 4 as a function of synthesis temperature and time. After heating to 1200 °C, the mixture contained about 1.7 wt % β -SiC as estimated from the XRD peak intensities (Figure 3a). After that, the quantity of β -SiC in the mixture increased rapidly above 1400 °C and reached 96.4% at 1700 $^{\circ}$ C. The density of the mixture was 1.65 g/cm³ after heating to 1000 °C, and then decreased to a minimum value of 1.24 g/ cm^3 at 1300 °C due to the loss of CO/CO₂ and the formation of the highly porous carbon. Above 1300 °C, the reaction of carbon with SiO₂ occurred, consuming the low-density carbon and forming the high-density β -SiC. Thus, the density of the synthesis mixture increased gradually and reached a value of 2.78 g/cm³ after heating to 1700 °C eventually. Similarly, heating for a longer time (4 h) at 1600 °C improved the yield of β -SiC from 88.4 to 95.2 wt %, and the density increased from 2.23 to 2.68 g/cm³ (Figure 4b).

To investigate the chemisorption behavior of the prepared sample, which is important to the synthesized β -SiC when it is used as catalyst support,⁵³ the CO₂-temperature programmed desorption (CO₂-TPD) experiment was performed. As shown in Figure 4c, two desorption peaks can be recognized. The

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Figure 3. Characterization of the β -SiC synthesis process. (a) XRD patterns and (b) Raman spectra of the synthesized mixture from 1000 to 1700 °C; (c) XRD and (d) Raman spectra of the synthesized mixture heated to different times at 1600 °C from 0 to 4 h; and (e) online furnace-MS profile of gaseous species released during the β -SiC synthesis process during the heating ramp from 25 to 1600 °C and maintained at 1600 °C for 4 h. Insert: photographic images of SiO₂ and synthesis mixture during this heating ramp from 1000 to 1700 °C



Figure 4. (a, b) Density of the synthesis mixture and SiC yield as a function of (a) temperature from 1000 to 1700 °C, and (b) time from 0 to 4 h at 1600 °C, and (c) CO_2 -TPD profile of the prepared SiC sample

board peak at around 335 $^{\circ}$ C represents the weak basic sites on the surface, while the sharper peak at 573 $^{\circ}$ C is assigned to the stronger basic sites. The presence of the basic sites is beneficial to lots of reaction when loading various active metals or metal oxides, which pointing to the potential for the prepared SiC to be used as excellent catalyst support.

The morphology and chemical composition of β -SiC synthesized at 1600 °C for 2 h were determined. Figure 5a illustrates that the synthesized β -SiC is in the form of particles

with a dark green color. SEM images (Figure 5b,c) shows that the particle size of β -SiC was less than 50 μ m with a few needle-like whiskers. The powder exhibited a microstructure pseudomorphous to the CS-derived carbon template. The porosity and pore structure of the carbon template were preserved. These particles were agglomerated from smaller nanoparticles with a size of around 100 nm, resulting in a rough appearance of the powder surface. These primary particles were β -SiC, as confirmed by the fringes in the bright

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Figure 5. Characterization of the synthesis mixture (1600 $^{\circ}$ C for 2 h): (a) photographic, (b, c) SEM, (d, e) HRTEM images, (f) electron diffraction pattern, (g) nitrogen adsorption-desorption isotherm, (h) pore size distribution, and (i) TG and DTG profiles

field images (Figure 5e) and the electron diffraction pattern in the selected area (Figure 5f). The interplanar spacing of β -SiC was 0.254 nm, corresponding to the (111) plane. The diffraction spots could be indexed to (111), (220), and (311) crystal planes of β -SiC.

The nitrogen adsorption-desorption isotherm and pore size distribution of synthesized β -SiC and carbon derived from CS at the same condition show a similar trend (Figure 5g,h). The BET surface area of the synthesized β -SiC and carbon from CS were 397 and 589 m^2/g with a pore volume of 0.40 and 0.65 cm^3/g , respectively. This indicates that the pore structure of the synthesized β -SiC is from the carbon framework derived from pyrolyzed CS. The stability of the synthesized β -SiC against oxidation was examined by TGA in the air (Figure 5i). There was a slight weight loss below 800 °C due to the combustion of residual carbon. Thus, β -SiC was stable up to 800 $\,^{\circ}\mathrm{C}$ in the air. Above 1000 $\,^{\circ}\mathrm{C}$, there was a noticeable weight gain, suggesting the oxidation of SiC to form a film of SiO₂ on the surface. Thus, the synthesized β -SiC with a high specific surface area and good antioxidation ability at a high temperature can be used as raw materials of reinforcing fillers, catalyst supports, and membrane components in a harsh environment.

Based on the above results, a reaction scheme for the β -SiC synthesis process is proposed in Figure 6. The two parts of this reaction scheme are the formation of the carbon framework from pyrolyzed CS and its reaction with SiO₂. Upon heating the synthesis mixture of CS and SS, the pyrolysis of CS first occurred and resulted in a carbon frame that was preserved from the cellulose and lignin structure of CS. At the same time, the SiO₂ in SS was crystallized and converted to cristobalite. When the temperature reached 1300 °C, the solid-state reaction occurred by surface migration of Si atoms or small clusters of SiO to the carbon and/or vaporization of SiO. The



Figure 6. Proposed reaction scheme for the β -SiC synthesis process

reaction rate increased with temperature and remained at a high level at 1600 $^\circ\mathrm{C}.$

The overall reaction of SS with carbon to form β -SiC is given as R1. We propose that it consists of a number of steps. In the first step, reactions R2 and R3 dominate, in which the reaction of SS particles and carbon leads to the formation of gaseous SiO, CO, and CO₂.^{24,54} Secondl, the SiO migrates to the surface and reacts with the carbon (R4 and R5). The MS result shows that SiC formation is associated with the evolution of CO. Thus, R4 is the dominant pathway. Although a combination of R5 and R6 also results in SiC formation and CO evolution, they would require a multistep process. Its complexity compared to R4 makes it less likely. Third, the formation of SiC by R4 explains the preservation of CS graphitic morphology, which would not be the case if SiC is formed by R5 and R6.

$$SiO_2(s) + 3C(s) \rightarrow SiC(s) + 2CO(g)$$
 (R1)

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$$\operatorname{SiO}_2(s) + \operatorname{C}(s) \to \operatorname{SiO}(g) + \operatorname{CO}(g)$$
 (R2)

$$SiO_2(s) + CO(g) \rightarrow SiO(g) + CO_2(g)$$
 (R3)

$$SiO(s) + 2C(s) \rightarrow SiC(s) + CO(g)$$
 (R4)

$$SiO(g) + 3CO(g) \rightarrow SiC(s) + 2CO_2(g)$$
 (R5)

$$CO_2(g) + C(s) \rightarrow 2CO(g)$$
 (R6)

CONCLUSIONS

We have demonstrated a simple and eco-friendly process to synthesize a high surface area, porous β -SiC using inexpensive materials of corn stover and Wyoming sandstone. The XRD, Raman, and an integrated furnace-MS system were used to monitor the phase changes during the β -SiC synthesis process to analyze reaction progress and study the mechanism in the range of 1000 to 1700 °C and different reaction times up to 4 h. The results show that the reaction condition of 1600 °C and 2 h is sufficient to generate a high yield of β -SiC without the formation of α -SiC impurity. The synthesized β -SiC has pores of 3-6 nm in diameter, a pore volume of $0.4 \text{ cm}^3/\text{g}$, and a high specific surface area of 397 m²/g. The pore structure follows the graphitic carbon template derived from the pyrolysis of CS, which could be preserved because β -SiC resulted from the reaction between gaseous SiO and the carbon template. The β -SiC produced by this method possesses properties suitable for use as catalysis supports and membranes needed in the harsh environment, such as a high temperature in the air or reactive atmospheres.

AUTHOR INFORMATION

Corresponding Authors

- Maohong Fan Department of Chemical and Petroleum Engineering and School of Energy Resources, University of Wyoming, Laramie, Wyoming 82071, United States; School of Civil and Environmental Engineering, Georgia Institute of Technology, Atlanta, Georgia 30332, United States;
 orcid.org/0000-0003-1334-7292; Email: mfan@ uwyo.edu, mfan3@mail.gatech.edu
- Harold H. Kung Department of Chemical and Biological Engineering, Northwestern University, Evanston, Illinois 60208, United States; orcid.org/0000-0001-9599-0155; Email: hkung@northwestern.edu

Authors

Tongtong Wang – Department of Chemical and Petroleum Engineering, University of Wyoming, Laramie, Wyoming 82071, United States

Weibo Gong – Department of Chemical and Petroleum Engineering, University of Wyoming, Laramie, Wyoming 82071, United States

Zuhao Kou – Department of Chemical and Petroleum Engineering, University of Wyoming, Laramie, Wyoming 82071, United States

Gang Tan − Department of Civil and Architectural Engineering, University of Wyoming, Laramie, Wyoming 82071, United States;
[●] orcid.org/0000-0001-5349-4110

Shaojun Zhou – Susteon Inc., Cary, North Carolina 2751, United States Hongtao Yu – Department of Chemistry, Morgan State University, Baltimore, Maryland 21251, United States; © orcid.org/0000-0002-9438-0432

Complete contact information is available at: https://pubs.acs.org/10.1021/acssuschemeng.0c04702

Author Contributions

^OT.W. and W.G. contributed equally to this work.

Author Contributions

T.W.wrote the original draft and performed the investigation, formal analysis, and visualization. W.G. performed the investigation, validation, and formal analysis. X.H. and Z.K. performed the investigation and formal analysis. G.T., S.Z., H.Y., and H.H.K. participated in conceptualization, and reviewed and edited the manuscript. M.F. participated in conceptualization, supervision, project administration, and reviewed and edited the manuscript.

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Notes

The authors declare no competing financial interest.

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Xin He – Department of Chemical and Petroleum Engineering, University of Wyoming, Laramie, Wyoming 82071, United States

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