The one-step pyrolysis process of rattan-based silicon carbide multiphase ceramics prepared by sol–gel method

Shijie Li1, Heshuai Cui1, Qianli Ma1, Xing'e Liu1, Youhong Wang2, Lili Shang1* and Yu Zheng3*

Abstract
The sol–gel method was used to prepare rattan-based silicon carbide (R–SiC) composite ceramics under different pyrolysis parameters through adjustment of the temperature and retention time of the one-step pyrolysis process. The crystalline phases, microscopic morphology, element distribution and specific surface area of the silicon carbide (SiC) were characterized by X-ray diffraction (XRD), Fourier transform-infrared spectroscopy (FT-IR), X-ray fluorescence spectrometer (XRF), field-emission scanning electron microscope (FESEM), transmission electron microscopy (TEM), energy dispersive X-ray analysis (EDX), and N2 physisorption. The results showed that the R–SiC prepared at different pyrolysis parameters was able to retain the porous structure of pristine rattan stem. The R–SiC prepared at 1500 ℃ for 120 min possessed the lowest density (0.25 g/cm³), the largest specific surface area (43.38 m²/g) and the highest SiC yield (44.24%). The SiC whisker was the major SiC morphology on the cross section of the R–SiC. Furthermore, the pyrolysis parameters were optimized with the SiC preparation process reaction mechanism, and material transformation methods were also discussed. This one-step pyrolysis process simplified the preparation of biogenic SiC ceramics and thus provided a potential route for the value-added utilization of rattan.

Keywords: Rattan, SiC ceramics, Sol–gel method, Pyrolysis process

Introduction
Silicon carbide (SiC) ceramics is a high-performing structural ceramic material. It has extremely high hardness and mechanical strength, great thermal stability, superior electrical properties and chemical inertness [1]. It is widely used in many industries such as automobiles, aerospace, electronic machinery, and nuclear magnetic field, and potentially has wide applications [2]. SiC ceramics is generally synthesized through the chemical vapor deposition (CVD) of silane compounds [3, 4], precursor infiltration pyrolysis (PIP) of organosilicon polymers [5–7], direct carbonization of elemental silicon (Si) and carbothermic of silicon dioxide (SiO₂) reduction. Of them, CVD can produce high-purity SiC ceramics, but expensive raw materials, complex reaction equipment and the harmfulness precursors restrict its large-scale industrial production. The molecular structure of the PIP precursor is designable desirable. Although its molding process is simple, the reaction temperature is low, and it has a certain ceramic yield, its precursor releases a large amount of gas during the thermal cracking process, which easily leads to large volume shrinkage, deformation, cracking and collapse of the ceramic matrix [8, 9]. The elemental silicon direct carbonization and the SiO₂ carbothermic reaction are also considered to be ideal SiC preparation methods thanks to their inexpensive raw materials, simple processes and equipment and high production efficiency. However, they have some shortcomings. Their maximum reaction temperature can reach 2700 ℃ due to the lack of effective contact between the
reactants. It may result in the low probability of intermolecular collisions. As a result, higher temperatures are needed. In order to effectively overcome this problem, more suitable raw materials are used to increase the contact area between the reactants to reduce the reaction temperature [10, 11].

The SiC material made from biomass through organic–inorganic transformation is a new environmentally friendly material with unique biological structure and ceramic properties [12]. So far, researches on SiC composite ceramics are limited mainly to wood, crops and their products and wastes as raw bio-materials. Rattan is also a renewable bio-material after bamboo and wood. This raw material is composed mainly of parenchyma cells, fibers and vessels [13, 14]. Due to the absence of secondary growth, the rattan stems have a complete primary form in nature, and do not produce phloem outwardly and xylem inwardly [15]. Rattan usually has three different macroporous pore diameters of 300 µm, 6–20 µm and 1–3 µm [16]. Therefore, using rattan as a raw material for the making of SiC multilphase ceramics makes a larger amount of external silicon sources easier to enter the pores of the rattan, thus increases the contact area between the reactants. The chemical composition of rattan, similar to that of wood, is composed mainly of α-cellulose (42–53%), hemicellulose (19–27%) and lignin (20–40%) [17]. Thanks to its high carbon content, rattan provides sufficient carbon source enabling the Si source to enter into the material. Moreover, rattan has a short growth cycle, low density, low transverse stem shrinkage, excellent toughness, easy processing, and high carbon content [18, 19]. These characteristics make rattan an ideal raw material for biomass-based SiC composite ceramics. Using rattan as a raw material for the manufacturing of SiC composite ceramics make external silicon sources easier to get into the rattan pores, thus increases the contact area between the reactants and reduce the reaction temperature. This serves as a value-added application for rattan.

In our previous study, rattan-based silicon carbide (R–SiC) ceramics was prepared using SiO\textsubscript{2} sol and liquid melt-Si method. Our study found that the SiO\textsubscript{2} sols from rattan were more appropriate than that from wood for the preparation of SiC with higher yield and purity from rattan [20]. The present study utilized rattan and SiO\textsubscript{2} sol as raw materials for the manufacturing of SiC composite ceramics through the sol–gel method under one-step pyrolysis. The SiO\textsubscript{2} sol was prepared with ethyl orthosilicate, absolute ethanol, and deionized water as raw materials, using hydrochloric acid as a catalyst. A vacuum-pressure impregnation method was used to impregnate the rattan into the sol to obtain a SiO\textsubscript{2} sol–gel/rattan mixture, which is pyrolyzed to generate R–SiC. The R–SiC materials are stable in nature and have good resistance in many aspects. Therefore, these R–SiC materials can be used in the fields of electricity, adsorption, new materials, fabrication and machinery [21, 22]. The morphology, yield and phase structure of the synthesized R–SiC were investigated. This one-step pyrolysis process simplified the preparation of biogenic SiC ceramics and thus may provide a value-added end-use for rattan.

Materials and methods

Materials

Canes of *Daemonorops jenkinsiana* (Griffith) Martius were collected from Nanmeiling Forest Park in Baisha county, Hainan province, China. The rattan diameter without sheath ranges from about 1.0 cm to 2.5 cm. Ethyl orthosilicate (TEOS, analytical reagent) was purchased from Sino pharmaceutical Reagent Co., LTD. Anhydrous ethanol (EtOH, analytical reagent) was purchased from Tianjin Damao Chemical Reagent Factory. Hydrochloric acid was purchased from the National Quality Inspection Center for Chemical Reagents. The standard titration solution (1 mol/L) and deionized water was made in the lab.

Experimental method

Raw material pretreatment

Samples preparation Three mature air-dried canes without obvious defects were selected. A 5-mm-thick piece was sampled at the middle of each canes. These samples were kept in the dry place for use. The content of SiO\textsubscript{2} in the original rattan is 0.11%.

Silicon sol preparation Ethylene-silicate, anhydrous ethanol and deionized water were mixed in accordance with the molar ratio of 1:4:6. The pH value of the solution was adjusted to 2.5 by applying hydrochloric acid, and SiO\textsubscript{2} sol was fully stirred until it became colorless and transparent. When the temperature was cooled down to room temperature, the sol was stored in refrigerator [23].

Impregnation The SiO\textsubscript{2} sol was ultrasonically treated for 10 min. Then, it was impregnated with rattan piece using the vacuum-pressure impregnation method. After drying, a gel/rattan piece mixture with a mass ratio of 7:39 was obtained (see Fig. 1).

Preparation of the SiC composite ceramics by one-step pyrolysis

10.00 g of the gel/rattan piece mixture was put into a crucible, covered with a lid and placed in a vacuum high-temperature tube furnace. The mixture was held at the desired temperature (1300, 1400, 1500 °C) for 120 min under the argon atmosphere.
curve of the ceramics is shown in Fig. 2a. The SiC composite ceramics prepared at three pyrolysis temperatures are marked as R–SiC T1300t120, R–SiC T1400t120, and R–SiC T1500t120. The R–SiC stands for the SiC composite ceramics prepared from rattan, and the others represent the composites prepared at pyrolysis temperatures of 1300, 1400, and 1500 °C, respectively. All the experiments were conducted in triplicate.

**SiC composite ceramics at different retention times**
10.00 g of the gel/rattan piece mixture was put into a crucible, covered with a lid and transferred to a vacuum high-temperature tube furnace. Under the argon atmosphere, the mixture was maintained at 1500 °C for 30, 60, and 120 min, respectively. The pyrolysis curve of the ceramics is shown in Fig. 2b. The SiC composite ceramics prepared at three retention times are marked as R–SiC T1500t30, R–SiC T1500t60, and R–SiC T1500t120. The R–SiC stands for SiC composite ceramics prepared from rattan, and in others “T” refers to the temperature with and “t” is refers to the duration of retention time (30, 60, 120 min). All the experiments were conducted in triplicate.
Characterization

The mass changes of the samples during the transformation of the rattan/silica material into the SiC ceramic were measured to 0.0001 g using an analytical balance (PL303-IC, Mettler-Toledo, Switzerland). The phase composition and crystallographic structure of the SiC samples were determined by X-ray diffraction (XRD, Bruker AV300, Philips, Holland) with a nickel filtered Cu Kα radiation over the 2θ range of 5–90 at a scanning speed of 4°/min. In addition, the elemental analyses of the SiC samples were conducted using the X-ray fluorescence (XRF, Shimadzu1800, Shimadzu, Japan). The chemical bonds and functional groups of the SiC samples were investigated using a Fourier transform-infrared spectroscopy (FT-IR, Nexus670, Nicolet, USA) at ambient conditions. The morphology and microstructure of the SiC samples were examined under a field-emission scanning electron microscope (FESEM, XL-30, FEI, USA). An energy dispersive X-ray analysis (EDX, FEG132-10, FEI, USA) equipped with FESEM was also carried out to determine the element composition of the SiC samples corresponding to the FESEM images. High-resolution transmission electron microscope (TEM, JEM-2100F, JEOL, Japan) was used to study the interface binding between the SiC and carbon. The specific surface area of the SiC samples was obtained by N2 physisorption instrument (ASAP2020, Micromeritics, USA).

Results and discussion

Mass residue and density analysis

The mass residue and density of the SiC composite ceramics formed by the gel/rattan piece mixtures at different pyrolysis temperatures and the retention times showed significant differences, as shown in Table 1. The samples exhibited a trend of R–SiCT1300t120 > R–SiCT1400t120 > R–SiCT1500t120 and R–SiCT1500t60 > R–SiCT1500t120. The mass residue of the R–SiC samples decreases with increasing temperature and retention time. For example, the mass residue of the sample R–SiCT1500t120 is only 68.91% of the R–SiCT1400t120 and 82.73% of the R–SiCT1500t30. Besides, the density of the R–SiC samples shows a decreasing trend with increasing pyrolysis reaction temperature and duration of the retention time.

Analysis of specific surface area

The specific surface areas of R–SiC are shown in Table 1. The specific surface areas of the samples goes up with increasing pyrolysis temperature and retention time, as the SiO2 sol is coated on the surface of the pore structure or accumulated in the pores after passing through the gel before pyrolysis. With the progress of the carbothermal reduction reaction, the SiO2 gel is gradually consumed by reduction and deoxidization so that the pore structure is gradually exposed. As the temperature rises and the retention time prolongs, the progress of the carbothermal reduction reaction and the formation of SiC will be promoted. This results in more SiO2 gel consumption, fewer pores covered by residual SiO2 gel, and an increase in specific surface area.

Phase analysis

The XRD was used to identify the phase composition of the obtained R–SiC samples prepared at different conditions. As shown in Fig. 3a, the XRD curve at different temperatures of 1300–1500 °C exhibits two typical peaks at 2θ = 35.8° and 22.0°. The peak at 2θ = 35.8° corresponds to (111) crystal planes of β-SiC according to the joint committee on powder diffraction standards (JCPDS) #29–1129 [22]. This indicates that the gel/rattan piece mixture can be transformed into the SiC complex ceramics at all designed temperatures. The intensity of the peaks is R–SiC T1300t120 (848 a.u.) < R–SiC T1400t120 (880 a.u.) < R–SiC T1500t120 (1292 a.u.). This indicates that the intensity of the peaks rises with increasing temperature. The diffraction peaks with 2θ values of 22° corresponds to the 101 crystal planes of β-SiC according to the joint committee on powder diffraction standards (JCPDS) #39–1425, showing the existence of crystalline SiO2 in all the samples. In addition, two weak peaks representing quartzite were found at 28.5° and 31.5° in R–SiC T1300t120, corresponding to (111) and (102) crystal planes of

Table 1 The mass residue, density and specific surface area of the R–SiC prepared at different pyrolysis conditions

| Sample | Temperature (°C) | Time (min) | Mass residue (%) | Density (g/cm³) | Specific surface area (m²/g) |
|--------|-----------------|------------|-----------------|-----------------|-----------------------------|
| 1      | 1300            | 120        | 41.08±0.62      | 0.38            | 30.75±0.31                  |
| 2      | 1400            | 120        | 31.59±0.79      | 0.27            | 40.39±0.50                  |
| 3      | 1500            | 120        | 28.31±0.28      | 0.25            | 43.48±0.87                  |
| 4      | 1500            | 60         | 30.77±0.37      | 0.34            | 21.23±0.32                  |
| 5      | 1500            | 30         | 34.22±0.60      | 0.36            | 7.47±0.18                   |
quartzite, respectively. As the temperature goes up, the diffraction peak of the crystal planes (111) of SiO$_2$ is not found at $2\theta = 28.5^\circ$ in R–SiC$_{T1400t120}$ and R–SiC$_{T1500t120}$. This indicates that temperature considerably affects the SiO$_2$ thermal reduction reaction. In the R–SiC$_{T1500t120}$, there are two peaks at 60.1° and 71.9°, corresponding to (220) and (311) crystal planes of the β-SiC, respectively. However, there are no two peaks at the same positions of the R–SiC$_{T1300t120}$ and the R–SiC$_{T1400t120}$. In combination with the diffraction peak intensity change of the β-SiC (111) crystal plane at $2\theta = 35.8^\circ$, it appears that there are more SiC in the R–SiC$_{T1500t120}$ sample. Gu et al. [24] reported that the low yield of the SiC was due to the low pyrolysis temperature, and more Si crystals were produced with increasing temperature. Similarly, Kuang et al. [25] also found that there was some unreacted SiO$_2$ at low temperature. When the temperature rises, the peak signal of the SiO$_2$ decreases, whereas the peak signal of the SiC increases. The pyrolysis temperature affects the yield and purity of the SiC in the R–SiC samples.

The XRD of the R–SiC$_{T1500t30}$, the R–SiC$_{T1500t60}$ and the R–SiC$_{T1500t120}$ under the temperature of 1500°C is shown in Fig. 3a. Figure 3b shows that there are three diffraction peaks in the R–SiC$_{T1500t120}$ at $2\theta$ value of 35.8°, 60.1° and 71.9°, corresponding to (111), (220) and (311) crystal planes of the β-SiC. The (111) crystal plane of the β-SiC is also observed in the R–SiC$_{T1500t30}$ and the R–SiC$_{T1500t60}$ and the peak intensity is R–SiC$_{T1500t120}$ (1292 a.u.) > R–SiC$_{T1500t60}$ (1097 a.u.) > R–SiC$_{T1500t30}$ (1044 a.u.). In other words, the intensity increases with increasing retention time. In addition, there are two typical diffraction peaks at $2\theta = 22^\circ$ and 31.5° in three retention time samples, corresponding to (101) and (102) crystal plane of quartzite, respectively. It is clear from the diffractograms that the diffraction peaks are present at $2\theta = 28.5^\circ$, corresponding to (111) crystal plane of cristobalite in the R–SiC$_{T1500t30}$ and the R–SiC$_{T1500t60}$. This indicates the existence of crystallization of the SiO$_2$. Shatumbu et al. [23] reported that the yield of SiC increased with the extension of the retention time. Therefore, prolonging the retention time is beneficial to the formation of β-SiC.

**Chemical composition**

FT-IR spectra of the R–SiC samples prepared at different conditions are displayed in Fig. 3b. The peak at 781 cm$^{-1}$ is due to the anti-symmetric stretching vibration of the Si–C bond, indicating the presence of the SiC in the samples. The stretching vibration peak of the Si–O–Si bond at 1090 cm$^{-1}$ indicates that excessive SiO$_2$ is still retained in the SiC complex ceramics. The transformation ratio of the Si–O–Si bond/Si–C bond as well as the content ratio of the SiO$_2$/SiC changes in the trend of R–SiC$_{T1300t120}$ (1.06) > R–SiC$_{T1400t120}$ (1.00) > R–SiC$_{T1500t120}$ (0.94). It indicates that with increasing pyrolysis temperature, the residual SiO$_2$ decreases and the formation of SiC increases. The stretching vibration peaks at different retention times were also located at 1090 cm$^{-1}$ and 781 cm$^{-1}$, respectively, indicating the presence of the SiC and SiO$_2$ in the samples. The transformation ratio of the Si–O–Si bond/Si–C bond as well as the content ratio of the SiO$_2$/SiC bond in the samples showed a trend of R–SiC$_{T1500t30}$ (1.00) > R–SiC$_{T1500t60}$ (0.98) > R–SiC$_{T1500t120}$ (0.94). This means that the amount of the SiC increases.
with increasing retention time, while unreacted SiO₂ decreases. By combining the transmission ratio of the Si–C bond/Si–O–Si bond, the ratio of the R–SiC samples at different temperatures shows a large range, indicating that the temperature has a more significant influence on the composition of the final SiC composite ceramics than the retention time. At the same time, there are weak peaks at 1630 cm⁻¹ and 1250 cm⁻¹, which corresponds to the stretching vibration characteristics of the C=C bond and C–C bond, respectively. This indicates that carbon is left in the R–SiC without complete reaction. Hung et al. [26] also found that there was some unreacted carbon in the sample.

**Microstructure and element distribution analysis**

The microstructure and the element distribution of the SiC multiphase ceramics at different reaction temperatures were investigated by FESEM and EDX and presented in Fig. 4; Fig. a and b are the cross section and longitudinal section of the original rattan, respectively. All prepared samples retain the pore structure of the original rattan. Under low-resolution FESEM images
(Fig. 4c–e), samples show a vessel-like pore structure with a size of about 170 µm. This structure is good for the subsequent absorption. Previous research has verified that higher porosity and average pore size correspond to greater averaged absorbing cross sections [21]. With increasing pyrolysis temperature, the amount of filler in the vessel-like pore increases. Under high-resolution FESEM images (Fig. 4f–k), the vascular bundle and the ground parenchymatous tissues of rattan are completely preserved, but some cell rupture or collapse could be visible. With increasing pyrolysis temperature the cell rupture or collapse increases significantly.

On the cross section, the microstructure and element distribution of the vascular bundle structure are shown in Fig. 4f–h. It is composed mainly of Si, C and a small amount of O. The presence of O indicates that there is SiO₂ residue in the R–SiC samples. The dense distribution of Si and the sparse presence of O indicate that the carbothermal reduction of SiO₂ occurs in the pyrolysis process, and SiC is generated. The microstructure and element distribution of the R–SiC on the longitudinal section are displayed in Fig. 4i–k. It indicates the structure of the pristine rattan is kept in the R–SiC, namely vessel, parenchymal cells and fiber cells exist clearly. From the element distribution of EDX, the R–SiC on the longitudinal section is also composed of Si, C and O, indicating the existence of SiC and SiO₂ in the R–SiC samples. On the longitudinal section of the R–SiC samples, Si and C are distributed regionally with few intersections, indicating that very little SiC is formed within the R–SiC, and a certain amount of carbon and SiO₂ remains. Although SiO₂ sol can enter the interior of rattan piece, the main entry channels are axial cells with large lumen such as vessels. The sol accumulates in the lumen and lacks effective contact with the vessel cell wall. Incomplete reaction during pyrolysis results in the dense distribution of Si in vessel-like lumen, while little SiC is produced in the vessel cell walls in the R–SiC samples. The amount of the SiO₂ sol entering the rattan piece through parenchyma cells with smaller diameter is limited and the distribution is uneven, resulting in the sparse and uneven distribution of Si and the concentrated distribution of element C in parenchyma cell of the R–SiC samples. This phenomenon indicated that the densification process is related to the open porosity. The rattan porous structure is composed of an interconnected network of solid pillars, and the pore size defines the arrangement of raw materials and the interaction with the surrounding environment [22].

![Fig. 5](image_url)
The microstructure and element distribution of the R–SiC at different retention times are shown in Fig. 4e, h, k and Fig. 5. Under low-resolution FESEM images (Fig. 4e and Fig. 5a, b), the cell wall surface is similar to pristine rattan, but it gradually becomes rough or broken, and the filler in the lumen increases with increasing retention time. From the EDX diagram, the element composition of the R–SiC samples is C, Si and O. It indicates that the SiC and SiO₂ coexist in the R–SiC samples. On the cross section (Fig. 4h and Fig. 5c, d), the distribution of C and Si is relatively dense, while the distribution of O is sparse. This indicates that a large amount of the SiC and a small amount of the SiO₂ are distributed on the surface of the R–SiC samples. On the longitudinal section (Fig. 4k and Fig. 5e, f), the Si is densely distributed in the vessel-like lumen, while the C is concentrated and the Si is sparse and uneven in the parenchyma cell. It indicates that the SiO₂ is largely retained in the large cell lumen such as the vessels, and there is less SiC formation in the parenchyma cells.

A flocculent structure was found on the surface of the R–SiC samples and in the vessel-like lumen (Fig. 6a–b). This flocculent structure is distributed in a disorderly and nondirectional state, while the flocculent structure in the lumen is located in the inner wall of the lumen and the top extends to the outside. The EDX analysis shows that the bamboo-shaped structure is SiC. This means the flocculent structure is actually composed of SiC whisker disordered arrangement. With increasing reaction temperature and retention time, the output of the SiC whisker on the R–SiC sample surface and in the lumen increases, leading to the lumen to be filled, blocked or even covered. This is similar to the study reported by Zhang et al. [27] and Shatumbu et al. [23].

The weight percentage and atomic number percentage of each element on the cross section and longitudinal section of the R–SiC were determined based on the EDX energy spectrum, and the SiC content in R–SiC was calculated. As shown in Table 2, the pattern of variation in SiC content on the R–SiC cross section is consistent with that on the longitudinal section, namely

| Sample | Temperature (°C) | time (min) | SiC Cross section (%) | Longitudinal section (%) |
|--------|------------------|------------|-----------------------|--------------------------|
| 1      | 1300             | 120        | 3.82 ± 0.02           | 1.11 ± 0.01              |
| 2      | 1400             | 120        | 8.58 ± 0.11           | 3.47 ± 0.04              |
| 3      | 1500             | 120        | 44.24 ± 0.44          | 18.29 ± 0.18             |
| 4      | 1500             | 60         | 39.28 ± 0.29          | 12.81 ± 0.01             |
| 5      | 1500             | 30         | 8.35 ± 0.15           | 6.87 ± 0.12              |

![Fig. 6](image) a and b FESEM image and the corresponding EDX spectrum of SiC whisker; c and d TEM image of the R–SiC.
proposed a synergistic growth mechanism from the per-
as the raw materials by carbonizing. Zhang et al. [32]
variation e of the SiC content among the R–SiC T1300t120,
time, the amount of the SiC increases. In addition, the
that with increasing pyrolysis temperature and retention
time, the distribution of the SiC on the transverse section
is mainly in the form of the SiC whisker on the rattan skeleton surface or in the lumen, but not in the longitudi-
dinal section. In the pyrolysis process, complex carbo-
thermal reduction reactions exist, involving solid–solid reaction, gas–solid reaction and gas–gas reaction [28,
29]. Considering the SiC whisker forming mechanism of gas–gas phase reaction, it is reasonable to assume that
the R–SiC mainly conducts solid–solid or solid–gas reaction
on the longitudinal section.

Above all, there are two forms of SiC in the rattan-
based SiC composite ceramics: independent SiC whisker
and SiC attached to the cell wall. As shown in Fig. 6c
and d, the interplanar spacing between two adjacent lattice fringes is 0.25 nm, which is consistent with the (111)
planes of the β-SiC, indicating that the SiC whiskers grow along the [111] direction due to its lowest nucleation energy on the (111) crystal plane [30]. The other phase of disordered distribution (marked with red rectangle) is the residual rattan carbon that has not been reacted completely and still retains amorphous structure without crystalization under the pyrolysis conditions. This is consistent with the XRD results shown above, and the formed SiC is bound closely to the residual carbon cell wall. The irregular accumulation and stacking faults during the growth process lead to its bamboo-like morphology, as shown in Fig. 6d. Similar observations were reported by Wu et al. [31]. They prepared diameter-fluc-
tuating SiC nanowires with bamboo-like morphology using bamboo-made paper and Si–SiO2 mixed powders as the raw materials by carbonizing. Zhang et al. [32]
proposed a synergistic growth mechanism from the per-
spective of homogeneous substrate as well as the varying elastic energy to explain the formation of the bambo-
like SiC.

Rattan materials are similar to wood and bamboo, and they have a porous structure that renders them useful for realizing different penetration and treatment proce-
dures [33]. The ceramic made from rattan retained the original structural features and unique microstructure as wood and bamboo being raw materials [34, 35]. On
the surface of the SiC sample of wood and bamboo and
in the vessel-like lumen, there are also flocculent struct-
tures composed of SiC whisker disordered arrange-
ment and distributed in a disorderly and nondirectional state [36, 37]. The high-resolution electron microscopy shows that the R–SiC flocculent structure consists of a single bamboo-like structure. Its characteristic shapes such as string-beads-like, bamboo-like and rod-like in the SiC crystals prepared from wood and bamboo [38–
41]. But the total porosity of rattan is larger than those of wood or bamboo [42]. It provides a more conveni-
ent channel for SiO2 sol impregnation and increases the
space contact with SiO2 sol. Compared with wood and bamboo, the effects of SiO2 sol impregnation is more uniform, and SiC is more fully formed. The natural and
special structure of rattan can bring good performance, and it may be more widely used in the future.

Conclusions

By the sol–gel method, rattan-based SiC composite ceramics (R–SiC) were prepared at the different temperatures and retention times in one-step pyrolysis. It is
found that both temperature and retention time have a
significant influence on the final R–SiC products. With increasing reaction temperature and retention time, the carbothermal reduction reaction of SiO2 gel and carbon is increasingly thorough in the pyrolysis process, and the SiC amount increases. The SiC mainly exists in the surface and pore of the R–SiC in the form of the SiC whisker. A small amount of the SiC binds closely to residual carbon in the interior of the R–SiC.
The pyrolysis parameters studied are able to retain the pore structure of rattan-based SiC composite ceramics completely, and the specific surface area of the R–SiC goes up with increasing temperature and retention time. The R–SiC prepared at 1500 ℃ and kept for 120 min has more complete pore structure, lower density (2.35 g/cm³), higher specific surface area (43.48
m²/g), SiC yield (44.24%) and lower SiO2 residue.

Abbreviations

R–SiC: Rattan-based silicon carbide; SiC: Silicon carbide; CVD: Chemical vapor deposition; PIP: Precursor infiltration pyrolysis; TEOS: Ethyl orthosilicate; EOH: Anhydrous ethanol; XRD: X-ray diffraction; XRF: X-ray fluorescence; FT-IR: Fourier transform-infrared spectroscopy; FESEM: Field-emission scanning electron microscopy; EDX: Energy dispersive X-ray analysis; TEM: Transmission electron microscopy; JCPDS: Joint committee on powder diffraction standards.

Acknowledgements

The particular support by the Fundamental Research Funds of ICBR (Grant Nos. 1632019019) is acknowledged with thanks.

Authors’ contributions

LSJ analyzed the data and was a major contributor in writing the manuscript. CHS performed the experiment. SLL supervised and supported the study. LXE was another major contributor in writing the manuscript. ZY participated in
designing the experiment. MQL and WYH collected the samples. All authors have read and approved the final manuscript.

Funding
This work was supported by the Fundamental Research Funds of ICBR (Grant Nos. 1632019019).

Availability of data and materials
The datasets used and analyzed during the current study are available from the corresponding author on reasonable request.

 Declarations

Ethics approval and consent to participate
The authors declare that they have no competing interests.

Competing interests
The authors declare that they have no competing interests.

Author details
1 Key Lab of Bamboo and Rattan Science and Technology, International Centre for Bamboo and Rattan, Beijing 100102, China. 2 School of Forestry & Landscape Architecture, Anhui Agricultural University, Hefei 230036, China. 3 Beijing Research Institute of Synthetic Crystals Co. Ltd, Beijing 100018, China.

Received: 10 June 2021 Accepted: 1 October 2021 Published online: 15 October 2021

References

1. Wang H, Bi Y, Zhou N (2016) Preparation and strength of SiC refractories in situ β–SiC whiskers as bonding phase. Ceram Int 42:727–733
2. He R, Ding G, Zhang K (2019) Fabrication of SiC ceramic architectures using stereolithography combined with precursor infiltration and pyrolysis. Ceram Int 45(11):14006–14014
3. Zhou X, Wang N, Lai H (1999) β–SiC nanorods synthesized by hot filament chemical vapor deposition. Appl Phys Lett 74(26):3942–3944
4. Kim BG, Yoon JY, Yoo CH (2015) Condensation of vapor species at the outlet of air at high temperature chemical vapor deposition using tetramethylsilane as a precursor for SiC bulk growth. CrystEngComm 17(16):3148–3152
5. Zollfrank C, Kladny R, Sieber H (2004) Biomorphic SiOx/C-ceramic composites from chemically modified wood templates. J Eur Ceram Soc 24(2):479–487
6. Chen L, Yang X, Su Z (2018) Fabrication and performance of micro-diamond modified C/SiC composites via precursor impregnation and pyrolysis process. Ceram Int 44(8):9601–9608
7. Xiang Y, Li W, Wang S (2012) Oxidation behavior of oxidative protective coatings for PIP-C/SiC composites at 1500 °C. Ceram 38:9–13
8. Liu L, Li H, Feng W (2013) Effect of surface ablation products on the ablation resistance of C/C–SiC composites under oxyacetylene torch. Corros Sci 67:60–66
9. Chollon G (2021) The high temperature reaction of ammonia with carbon and SiC–C ceramics. J Eur Ceram Soc 41(1):136–147
10. Zampieri A, Sieber H, Selvam T (2005) Biomorphic cellular SiC/zeolite ceramic composites: from rattan palm to bioinspired structured monoliths for catalysis and sorption. Adv Mater 17(3):344–349
11. Zeni TL, Rambo CR, Magalhães WL (2013) Control of porosity and permeability of wood by fungi action for biotemplating of SiC. J Wood Chem Technol 33(1):33–43
12. Rikonen J, Rantanen J, Thapa R (2021) Rapid synthesis of nanostructured porous silicon carbide from biogenic silica. J Am Ceram Soc 104(2):766–775
13. Yang SM, Xiang EL, Shang LL (2020) Comparison of physical and mechanical properties of four rattan species grown in China. J Wood Sci. https://doi.org/10.1186/s10086-020-1850-0
14. Shang LL, Jiang ZH, Liu XE (2016) Effect of modification with methyl methacrylate on the mechanical properties of plectocoria kerrana rattan. Bioresources 11(1):2071–2082
15. Yudodibroto H (1985) Anatomy, strength properties and the utilization of some Indonesian rattans. Proceedings of the rattan seminar, 2–4 Oct 1984, Kuala Lumpur, Malaysia the Rattan Information Centre, pp. 117–122
16. Liu YE, Tian GL, Shang LL (2014) Compression properties of vascular bundles and parenchyma of rattan (Plectocoria assimandra Griff.). Holzforschung 68(8):927–932
17. Abasolo WP, Yoshida M (2003) Thermal softening of rattan canes: influence of the hemi-cellulose-lignin matrix. World Bamboo Rattan 1(4):317–331
18. Marly A, Das H, Kalta D (2014) Studies on formation and siliconization of carbon template of coir fibroboard precursor to SiC ceramics. J Eur Ceram Soc 34(15):3499–3511
19. Gryshko O, Klyui NI, Temchenko VP (2016) Porous biomorphic silicon carbide ceramics coated with hydroxyapatite as prospective materials for bone implants. Mater Sci Eng C 68:143–152
20. Cui HS, Zheng Y, Ma JF (2017) Effect of different silicon sources on rattan-based silicon carbide ceramic prepared by one-step pyrolysis. J Wood Sci 63(1):95–103
21. Albano M, Delfini A, Pastore R (2016) A new technology for production of high thickness carbon/carbon composites for launchers application. Acta Astronaut 128:277–285
22. Pastore R, Delfini A, Michel D (2019) Carbon foam electromagnetic mm-wave absorption in reverberation chamber. Carbon 144:63–71
23. Shatumbu T, Alweendo A, Oluwagbenga T (2019) Synthesis, optimization and characterization of silicon carbide SiC from rice husk. 2nd International conference on sustainable materials processing and manufactur- ing. Procedia Manuf 35:962–967
24. Gu J, Lee SH, Lee HS (2021) Fast pyrolysis of a SiC-based polymer precursor using a spark plasma sintering apparatus-effects of heating methods and pyrolysis atmosphere. J Eur Ceram Soc 41(4):2297–2305
25. Kuang JL, Xiao T, Hou XJ (2019) Microwave synthesis of worm-like SiC nanowires for thin electromagnetic wave absorbing materials. Ceram Int 45:11660–11667
26. Hung KC, Wu TL, Xu TW (2019) Preparation of biomorphic porous SiC ceramics from bamboo by combining sol–gel impregnation and carbonothermal reduction. Polymers 11(9):1442
27. Zhang NC, Yu AX, Liang AH (2013) Preparation of SiC whisker and application in reinforce of polystyrene resin composite materials. J Appl Polym Sci 130(1):579–586
28. Sujjrote K, Leangsuwan P (2003) Silicon carbide formation from pre-treated rice husks. J Mater Sci 38(23):4739–4744
29. Herzog A, Klingner R, Vogt U (2004) Wood-derived porous SiC ceramics by sol infiltration and carbothermal reduction. J Am Ceram Soc 87(5):784–793
30. Zhang XN, Chen YQ, Xie ZP (2010) Shape and doping enhanced field emission properties of quasi-aligned 3C-SiC nanowires. J Phys Chem C 114:8251–8255
31. Wu ZH, Zheng HK, Zhang GL (2020) Synthesis of diameter-fluctuating silicon carbide nanowires for excellent microwave absorption. Mater Chem Phys 244:122648–122671
32. Zhang M, Zhao J, Li ZJ (2016) Bamboo-like 3C-SiC nanowires with periodic fluctuating diameter: homogeneous synthesis, synergistic growth mechanism, and their luminescence properties. J Solid State Chem 243:247–252
33. Liu HR, Jiang ZH, Fei BH (2015) Tensile behavior and fracture mechanism of moso bamboo (Phyllostachys pubescens). Holzforschung 69(1):47–52
34. Huang TX, Li Z, Huang YQ (2020) Microstructure and wear properties of SiC wood ceramics reinforced high-chromium cast iron. Ceram Int 46(3):2592–2601
35. Guo XZ, Zhu L, Li WY (2013) Preparation of SiC powders by carbothermal reduction with bamboo charcoal as renewable carbon source. J Adv Ceram 2(2):128–134
36. Ershov AE, Klassen NV (2015) Control of functional characteristics of biomorphic carbon matrices, SiC-SiC composites, and SiC-C ceramics by prepressing wood. In: Ong Mater 6(1):79–90
37. Zhu JT, Jia JC, Kwong FL (2013) Synthesis of 6H-SiC nanowires on bamboo leaves by carbothermal method. Diam Relat Mater 45:11660–11667
38. Chiew YL, Cheong KY (2011) A review on the synthesis of SiC from plant-based biomasses. Mater Sci Eng B Adv 176(13):951–964
39. Li Z, Shi TJ, Tan DX (2013) Conversion of a wood flour–SiO$_2$–phenolic composite to a porous SiC ceramic containing SiC whiskers. J Serb Chem Soc 78(8):1213–1223
40. Yukhymchuk VO, Kiselov VS, Valakh MY (2016) Biomorphous SiC ceramics prepared from cork oak as precursor. J Phys Chem Solids 91:145–151
41. Liensdorf T, Schoch R, Lang M (2020) Silicon infiltrated silicon carbide from extruded thermoplastic wood polymer composites. Int J Appl Ceram Tec 17(1):3–10
42. Lin YJ, Lo XW (2011) Fabrication of SiC and SiC/aluminum-silicon composites from rattan charcoal. Key Eng Mater 479:119–123

Publisher’s Note
Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.