

# Effect of the addition of TEOS on the SiC fibers fabricated by electrospinning

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**Abstract:** Polycarbosilane (PCS) has been widely used to fabricate silicon carbide (SiC) fibers via pyrolysis. In this paper, for improving the morphology of SiC fibers, tetraethyl orthosilicate (TEOS,  $m = 1$  g, 3 g and 5 g, respectively) was added into the PCS precursor solution (containing 1.5 g PCS). The continuous fibers have been prepared by electrospinning, and then the SiC fibers were synthesized by calcination at 1300 °C, 1400 °C and 1600 °C for 4 h respectively with a heating rate of 10 °C/min in flowing nitrogen (N<sub>2</sub>). The morphologies of the fibers were investigated by the scanning electron microscope (SEM) and it could be seen that the crystallinity of the SiC fibers was lower, the length of the SiC fibers was increased, and the diameter was uniform with the increase of the addition amount of TEOS.

**Keywords:** silicon carbide (SiC); tetraethyl orthosilicate (TEOS); electrospinning; fibers

## 1 Introduction

In the past decade, one-dimensional (1D) nanosized semiconducting materials have attracted attention with their fascinating optical, electronic and chemical properties. The size and morphology can affect their applications as catalysts, solar cells, light-emitting diodes and biological labeling [1–3]. As a semiconducting material, silicon carbide (SiC) possesses excellent physical and electronic properties such as high mechanical strength, high thermal stability and high thermal conductivity [4]. It also has wide applications including field emission displays, nano-sensors and other nanoscale devices [5,6].

Recently, much more effort has been devoted to the synthesis process of SiC fibers with nanostructures,

including carbothermal reduction of silica [7–9], chemical vapor deposition [6,10–12], carbon nanotube-templated growth [13], etc. It has been demonstrated that polycarbosilane (PCS) is used to synthesize SiC fibers by melt-spinning, curing and pyrolysis. In this paper, SiC fibers could be synthesized by a high-temperature carbothermal reduction process using electrospun PVP (polyvinyl pyrrolidone)/PCS composite fibers as precursor. In addition, tetraethyl orthosilicate (TEOS) was added to improve the morphology of SiC fibers. Compared with the reported synthetic methods, the technique used in this work possesses the virtues including simplicity, low cost and absence of template and catalyst.

## 2 Experiment

As a starting material, PCS polymer was used to fabricate SiC fibers due to its rich content of silicon.

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For achieving PVP/PCS/TEOS composite, 1.5 g PCS and TEOS (1 g, 3 g and 5 g, respectively, Tianjin Kermel Chemical Reagent Ltd.) were dissolved into 11 ml solvent including 8 ml tetrahydrofuran (THF, Tianjin Kermel Chemical Reagent Ltd.) and 3 ml absolute alcohol ( $\geq 99.7\%$ , Shenyang Xinxing Chemical Reagent Ltd.). Then 0.8 g PVP (K90, Tianjin Bodi Chemical Co. Ltd.) was added in the solution to increase viscosity. Subsequently, the mixture viscous solution was magnetically stirred for 1 h at room temperature until the floccules were completely dissolved. The solution was transferred into a plastic syringe with conductive stainless steel needle and ejected with an appropriate voltage of 30 kV. The dense net of fibers was formed on the collector which was coated with an aluminium foil. The distance between the needle and the collector was about 16 cm. Whereafter, the fiber mat was immediately dried in air oven at 80 °C for 0.5 h, and then was heated at 190 °C for 6 h. The Si–H bonds in PCS were oxidized and the Si–O–Si crosslinked structure was formed. The melting of PCS was prevented and the morphology of precursor fibers was remained during the process of high-temperature pyrolysis [14,15]. Subsequently, the PCS precursor fibers were calcined respectively at 1300 °C, 1400 °C and 1600 °C for 4 h with a heating rate of 10 °C/min in flowing nitrogen (N<sub>2</sub>) followed by calcination at 600 °C for 4 h in air to remove the remaining carbon.

X-ray diffraction (XRD) patterns were collected on a Shimadzu/XRD-6000 diffractometer with Cu K $\alpha$  radiation. The morphologies of SiC fibers were characterized by JSM-5600LV scanning electron microscope (SEM, JEOL).

### 3 Results and discussion

The XRD patterns of the SiC fibers after calcination at 1600 °C for 4 h which are derived from different precursor fibers are shown in Fig. 1. The main peaks in the patterns match well with the cubic  $\beta$ -SiC phase (JCPDS 74-2307), and their peak positions at  $2\theta = 36^\circ$ ,  $60^\circ$  and  $72^\circ$  are respectively attributed to the (111), (220) and (311) planes of  $\beta$ -SiC. The SiC crystal is successfully synthesized at this temperature. The diffraction peaks of products become weak and broad which indicates that the crystallinity is lower and the grain size in SiC fibers is decreased with the increase

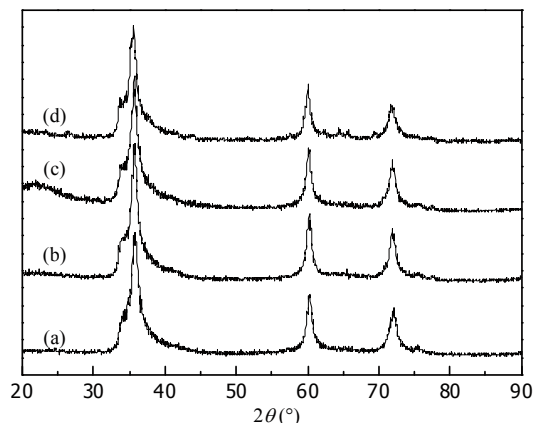


Fig. 1 XRD patterns of SiC fibers after calcination at 1600 °C for 4 h derived from precursor fibers with different TEOS contents: (a) 0 g, (b) 1 g, (c) 3 g, (d) 5 g.

of TEOS content. There are no other obvious diffraction peaks, which imply that the main product is cubic SiC phase and no impurity content is obtained in the fibers.

The SEM images of the PCS/TEOS precursor fibers are shown in Fig. 2. It could be observed that the surface of the precursor fibers is very smooth, which is attributed to the fine particles and the amorphous nature of the fibers. Moreover, with the increase of addition amount of TEOS, the length of the fibers is longer and the diameter is more uniform, which can also be seen in Fig. 3.

The SEM images of the SiC fibers calcined at 1300 °C and the corresponding columnar section of the diameter uniformity with different amounts of TEOS

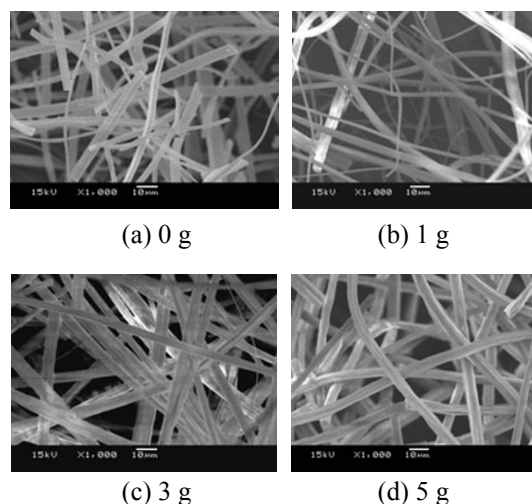


Fig. 2 SEM images of the PCS precursor fibers with different TEOS contents.

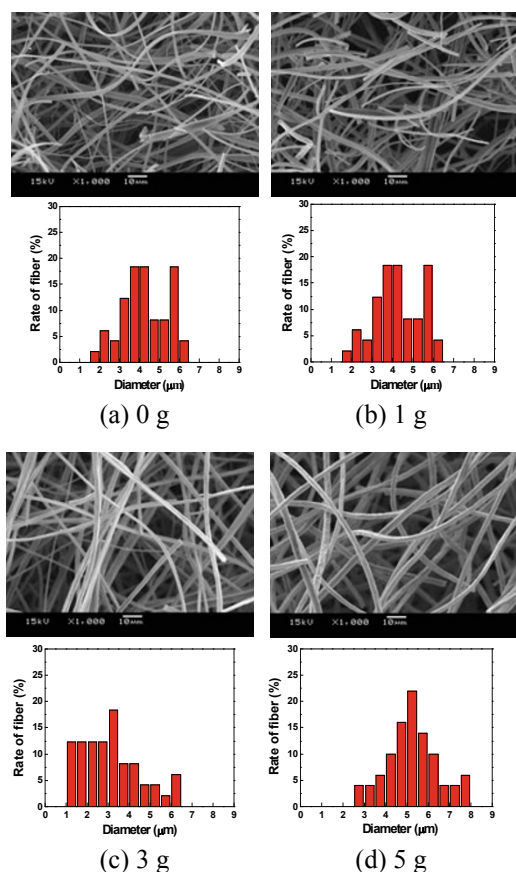


Fig. 3 SEM images of the SiC fibers calcined at 1300 °C and the corresponding columnar section of the diameter uniformity with different amounts of TEOS.

are shown in Fig. 3. We observe that the fibers with diameters in the range of 1–9 μm are brittle and easy to fracture when there is no or 1 g TEOS addition. However, the fibers become longer and their diameters concentrate in the range of 4–6 μm when the addition amounts of TEOS reach 3 g and 5 g.

The SEM images of SiC fibers derived from different precursor fibers are shown in Fig. 4. After calcinations at 1600 °C for 4 h, the SiC grains as shown on the fiber surface are averagely smaller than 500 nm, and the grains are closely connected to each other with clear grain boundaries. With the addition of TEOS, the surface of SiC fibers becomes smoother and the SiC grain sizes in the fibers are decreased, which also have been confirmed by the XRD patterns.

The SEM images of the PCS/TEOS precursor fibers and SiC fibers calcined at different temperatures with 5 g TEOS addition are shown in Fig. 5. The average diameter of the fibers is about 3 μm, and the length could reach several millimeters. The surface of SiC

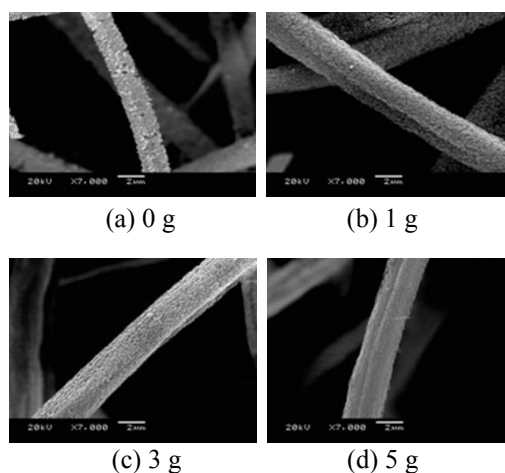


Fig. 4 SEM images of SiC fibers calcined at 1600 °C derived from different precursor fibers with different TEOS contents.

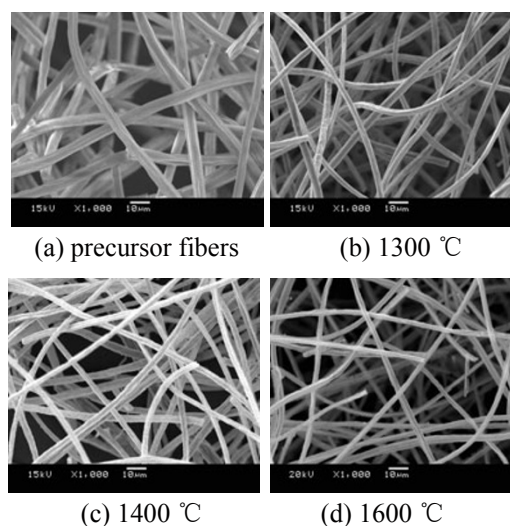


Fig. 5 SEM images of the precursor fibers and SiC fibers calcined at different temperatures.

fibers after calcined for 4 h becomes rough due to the removal of organic components and the crystallization of the SiC phase, while the surface of the precursor fibers (Fig. 5(a)) is very smooth. The fibers show a slightly decrease in diameter due to the burning-off of PVP, whereas the continuous microstructure of the fibers is still maintained.

### 3 Conclusions

In summary, direct electrospinning was used in this work to prepare the PCS/TEOS precursor fibers, and the SiC fibers were obtained successfully after carbonization. The addition of TEOS improved the

morphology of SiC fibers effectively. With the increase of addition amount of TEOS, the crystallinity was lower and the grain size in SiC fibers was decreased. The length of the fibers was longer and the diameter was more uniform.

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