Preparation of C-shaped silicon carbide fibers

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Silicon carbide (SiC) ceramic fibers are well known for their high tensile strength, high stiffness, excellent heat, oxidation and corrosion resistance, and intermiscibilities with resins, metals, and ceramics [1–3]. In a previous study [4], the trilobal SiC fibers have been prepared by the process of spinning, curing, and heat treatment in N_2 atmosphere. The results showed that the tensile strength of trilobal SiC fibers is on an average 30% higher than those of circular ones with the same effective filament diameter. Study [5] also showed that the structural radar absorbing materials, composed of the trilobal SiC fibers with resin, exhibited a reflection attenuation amount of 10–20 dB in the range of 8–18 GHz. Rhee and his co-workers [6] reported that C-shaped carbon fiber, which is also a kind of profiled fiber, can be prepared. In the present paper, the preparation process of C-shaped SiC fibers and characterization of C-shaped polycarbosilane (PCS) fibers have been reported.

In the study, two kinds of PCS (PCS-1, PCS-2) with different number average molecular weight and different softening point (T_m) were synthesized by thermal decomposition and condensation of polydimethylsilane (PDMS). The PCS was melt-spun through a C-shaped spinneret in N_2 atmosphere, and then the C-shaped PCS fibers were obtained. The degree of profile (D_{pc}) of C-shaped PCS fibers could be calculated by means of the cross-section parameters such as outer diameter D, inner diameter d and stretch angle θ which could be measured by optical microscope (OM, XSP-8C, China). The precursor C-shaped fibers were cured in air, heated to a specific temperature in N_2 atmosphere, and then the C-shaped SiC fibers were prepared.

The curing degree could be characterized with the reaction degree of Si—H bond P_{Si-H} which could be calculated by Infrared Spectroscopy (IR, Nicolet-360, America).

The relationships between D_{pc} of C-shaped PCS fibers and spinning temperature (Fig. 1), N_2 pressure (Fig. 2) and take-up rate (Fig. 3) were investigated respectively. From Fig. 1, we can see that the value of D_{pc} of C-shaped PCS decreased with the rising of spinning temperature at first, then increased. There were two reasons for this phenomenon, on the one hand, when the spinning temperature was low, the outer diameter and inner diameter increased in proportion with the rising of spinning temperature, while θ was on the increase. On the other hand, θ went down when the spinning temperature became higher. Study showed that only the hollow PCS fibers could be attained when the spinning temperature (PCS-1) was above 274 °C.

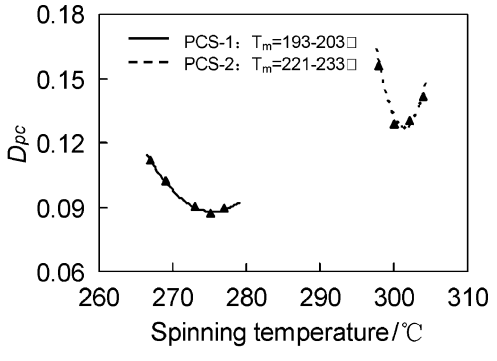

Figure 1 The relationship between spinning temperature and D_{pc} of C-shaped PCS fiber.

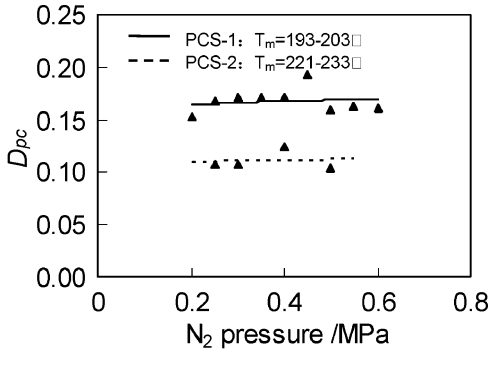

Figure 2 The relationship between N_2 pressure and D_{pc} of C-shaped PCS fiber.

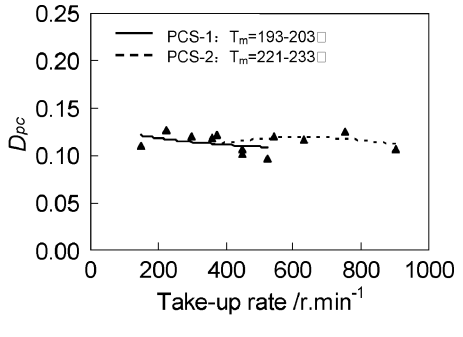

Figure 3 The relationship between take-up rate and D_{pc} of C-shaped PCS fiber.

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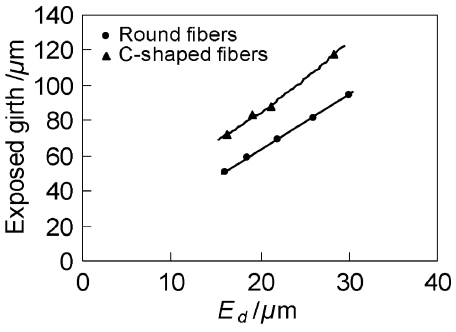

Figure 4 The relationship between E_d and exposed girth of C-shaped PCS fiber.

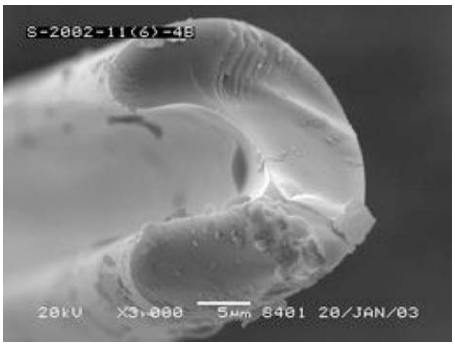

Figure 6 SEM photo of C-shaped SiC fiber.

As can be seen from Figs 2 and 3, the value of D_{pc} remained nearly the same when changing N_2 pressure and take-up rate. The reason for this was that the d/D and θ almost remained steady in the spinning process. The C-shaped PCS fibers with small E_{dc} (<30 μm) and high D_{pc} (0.10–0.15) could be gained under such conditions as low spinning temperature, N_2 pressure, and appropriate higher take-up rate.

Study showed (Fig. 4) that the exposed girth of C-shaped PCS fibers was on an average 30% higher than that of the round PCS fibers with similar effective filament diameter (E_d). The curing process is a gas-solid reaction which depends on the O_2 diffusion rate. The bigger the surface area and the smaller E_d of PCS fibers are, the easier for oxygen to diffuse in the curing process. When the length of PCS fibers was the same, the surface area was dependent on the exposed girth of the fibers. Consequently, it was more effective to deal with C-shaped PCS fibers than round PCS fibers in the air curing process. As shown in Fig. 5 the reaction degree of Si—H bond (P_{Si-H}) of C-shaped PCS fibers was about 5% higher than those of round PCS fibers, which certainly made for the O_2 adequate diffusion in C-shaped PCS fibers. So the curing time of C-shaped PCS fibers could be reduced sharply for the above reason.

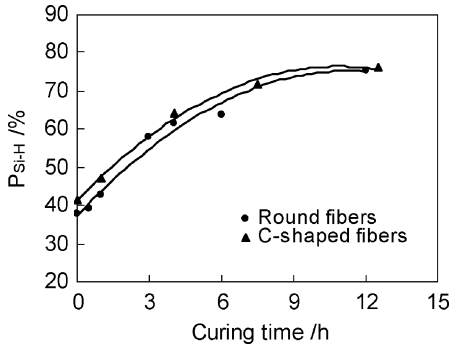

Figure 5 The relationship between curing time and reaction degree of Si—H bond P_{Si-H}.

The scanning electron microscope (SEM, JEOL-5600LV, Japan) photo of C-shaped SiC fiber is shown in Fig. 6. From Fig. 6, we see that the shape of the fibers was unchanged after the air curing and heat treatment. The reason for this was that the reaction and shrinkage in every direction was the same. In addition, the performance of C-shaped fibers and the formation of hollow PCS fibers, melt-spun through C-shaped spinneret, were being studied.

Acknowledgment

The project was supported by the National Natural Science Foundation, "863" National Foundation and Natural Science Foundation of Hunan Province of China, under contacts no. 00282007, 2002AA305101 and 02JJY2083 respectively.

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Received 19 January
and accepted 26 March 2004