MICROSTRUCTURAL DIFFERENCE BETWEEN THE CORE AND SKIN OF A T700 CARBON FIBER IN THE C/C COMPOSITE AFTER HEAT-TREATED

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Introduction

The microstructure of PAN-based carbon fibers has drawn large interest from material scientists during the past four decades. The microstructures of PAN-based carbon fibers have been analyzed and reviewed extensively [1-3]. Several descriptive models have been proposed to describe the microstructure of PAN-based carbon fibers. The major models are the dog-bone model [1], the sheath-core model [4], the circumferential model [5], the circumferential sheath-radial core model [6], the thin-skin-random-core model [7] and the progressive-variation model [8]. These descriptive models are not consistent well with each other and questionable. To understand the intrinsic structural model of PAN-fiber thoroughly, the continuous microstructure evolution from fiber surface to core still needs to be revealed in detail.

Experimental

The carbon fiber used in this study is commercial T700 PAN-based carbon fiber, which is fabricated to preform for chemical vapor infiltration (CVI) in order to obtain bulk samples. The carbon fiber preform was densified by CVI in C_3H_6+N_2 atmosphere at 1000 °C. Then the specimens were cut into three parts marked sample 1, 2 and 3. Sample 1 was used to investigate the microstructure of carbon fibers heat treated at the lowest temperature of 1000 °C. Sample 2 and 3 were heat treated at 2300 °C and 2800 °C, respectively, in order to study the structural evolution at high temperatures. In order to obtain thin area with a whole carbon fiber, at least 10 samples were carefully prepared for each C/C composite. All thin foils were examined on a Technai F30 high-resolution TEM operated at 300 kV.

Results and Discussion

Typical transversal and longitudinal morphologies and their corresponding selected area electron diffraction (SAED) patterns of carbon fibers treated at temperature s of 1000 °C, 2300 °C and 2800 °C are shown in Fig. 1 and 2. A core-skin feature of the fiber is evidently shown. The core region is marked by two parallel dashed lines in Fig .1 (a), (b) and (c). It can be seen that the diameter of the core zone decreases with increasing heat-treatment temperature. Besides, there is a dark contrast ring that separates the skin and core zones, especially clear in fig .1 (a). This ring seems to disappeared after heat treatment at high temperature of 2800 °C (see fig. 1(c)). We named this ring transition zone (TZ). Structural characteristics and chemical composition of the TZ are still in investigation.

All SAED patterns on transversal section show a quite uniform 002 ring, indicating a random orientation of basal planes in the transversal section. The broadening of the 002 rings in the radial direction in all SAED patterns of sample 1 indicates that the interlayer spacing (d002) slightly varies. Besides, the strong 002 rings but weak 004 rings illustrate that the crystallite sizes are small. For sample 2 and 3, with increasing heat-treatment temperature, a weak 110 ring emerges besides the 002 ring. This slight change indicates that the crystallite size has increased after heat treatment at 2300°C and 2800 °C. The decrease of the

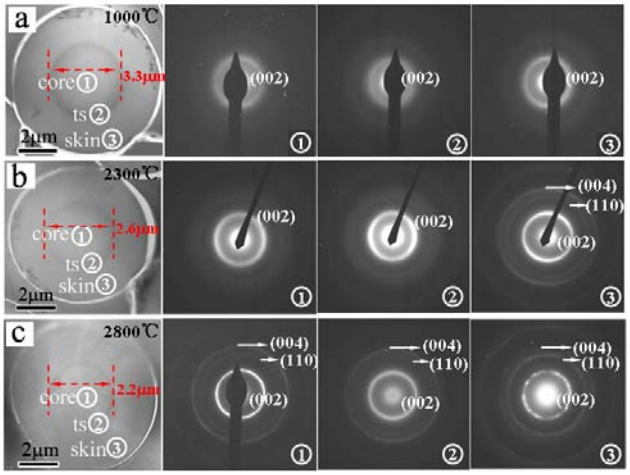

Fig. 1 Low-magnification TEM micrographs of transversal sections of fibers after heat treatments at 1000 °C (a), 2300 °C (b) and 2800 °C (c) and their corresponding SAED patterns of the core (①), transition (tz) (②) and skin regions (③).

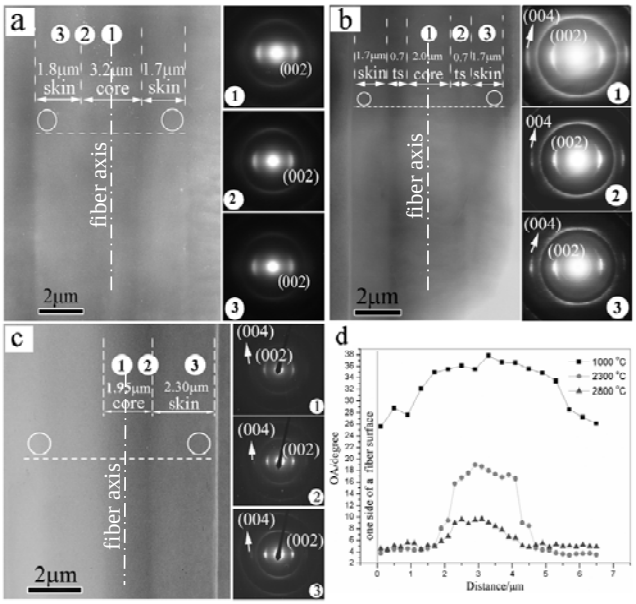

Fig. 2 Low-magnification TEM micrographs of longitudinal sections of a fiber and their corresponding SAED patterns of the core, intermediate and skin regions after heat treatment at 1000 °C (a), 2300 °C (b) and 2800 °C (c). The OA as a function of the distance from the fiber surface is shown in (d)

broadening of the 002 ring in radial direction indicates that the interlayer spacing has become more uniform. Orientation angles (OA) derived for all three samples in the longitudinal section as a function of the distance along the diameter from a fiber surface to the other surface is shown in fig. 4 d. It can be seen that, for sample 1, the alignment of the basal plane with respect to the fiber axis is slightly better in the skin than in the core zone. However, although the alignment increases both in the skin and core zone after heat treatment at 2300 °C, it increases most significantly in the skin zone. Moreover, it increases in the core zone only after further het treatment at 2800 °C.

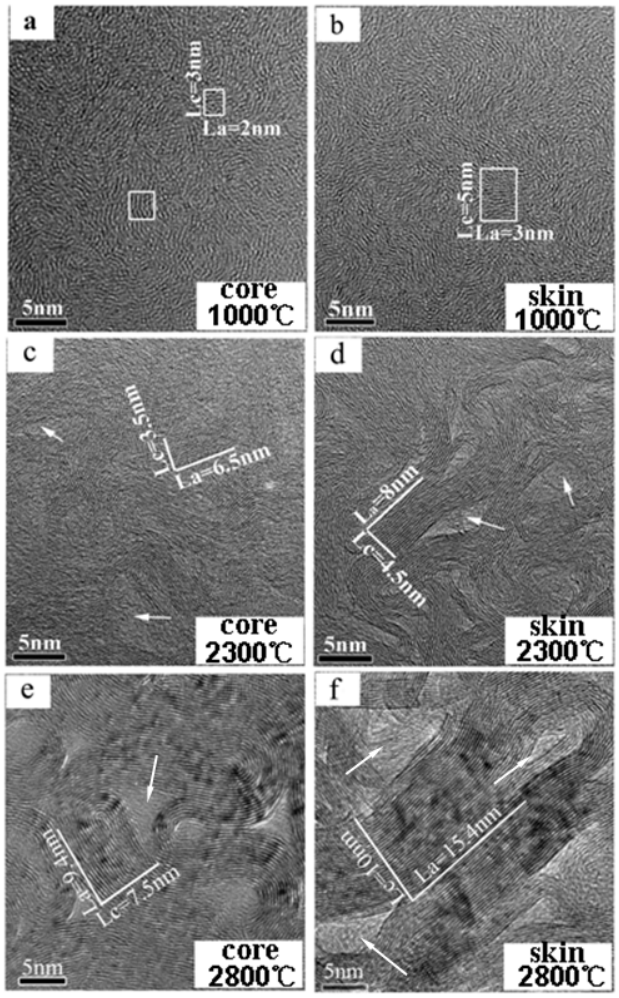

Fig. 3 HRTEM images of a transversal section, showing the crystallite size and shape in the core and skin regions

The HRTEM lattice images in Fig. 3 show that the crystallite sizes in the core and skin regions are not significantly different in sample 1. However, in sample 3 L_a is much different in the core and in the skin, being 9.4 nm in the core and 15.4 nm in the skin. It shows that the crystallite size starts to increase rapidly in the skin zone and then in the core zone with increasing heat-treatment temperature. Besides this, the arrows in fig. 3 indicate that, with increasing crystallite size, nanopores start to form among the crystallites. The pore size increases with increasing crystallite size.

Conclusions

The degree of longitudinal basal-plane alignment in carbon fibers can quantitatively be represented by the OA value across the fiber. This method can be used to identify the microtexture of any type of carbon fiber. The OAs of a T700 PAN-based carbon fiber on longitudinal section show that the orientation of the basal planes gradually decreases from the skin region to the core zone after heat treatment at 1000 °C. With increasing heat-treatment temperature, the basal-plane orientation changes to parallel to the fiber axis, and OA becomes more sharp first in the skin region and then in the core zone. With increasing heat-treatment temperature, the crystallite size increases significantly, faster in the longitudinal direction than in the transverse direction. Besides, nanopores form with increasing crystallite size.

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