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Effect of carbonization temperature on properties of aligned electrospun polyacrylonitrile carbon nanofibers

Cheng-Kun Liu [a,b,*], Yan Feng [b], Hai-Jun He [a], Jing Zhang [a], Run-Jun Sun [a], Mei-Yu Chen [a]

[a] School of Textile and Materials, Xi'an Polytechnic University, Xi'an 710048, China
[b] State Key Laboratory for Mechanical Behavior of Materials, Xi'an Jiaotong University, Xi'an 710049, China

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ABSTRACT

Aligned electrospun nanofibrous bundle was used as the raw material for pretreatment, preoxidation and carbonization processes to prepare carbon nanofibers in a procedure temperature-controlled sintering furnace. Effect of carbonization temperature on the morphology and structural performance of nanofibers was investigated in present study. Results showed that R_I (the relative intensity radio between Disordered peak and Graphite peak) of nanofibers carbonized at 1000 °C is 0.90, carbon content is up to 85.67%, conductivity is 105.44 $S \cdot cm^{-1}$, Young's modulus is 68.8 ± 0.42 GPa, and fiber strength is 306.0 ± 9.0 MPa, all of which endow the fibers with a superior comprehensive property.

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1. Introduction

Carbon nanofiber can be applied in many fields including reinforcing materials, template for the preparation of nanotubes, high-temperature filter, and high-temperature catalytic matrix materials due to its superior properties, such as high specific surface area, high temperature resistance, and good electrical/thermal conductivity [1]. Traditional preparation methods of carbon nanofiber include support catalyst method, spraying method, vapor grown method, and so on. However, these methods are relatively complicated and high-cost. Therefore, a simple and relatively cheap electrospinning process, without the requirement of purification process, is gradually regarded as the optimum process for the preparation of continuous and uniform carbon nanofibers. Carbon nanofibers prepared by precursors, such as polyacrylonitrile (PAN) [2–12], pitch [13], polybenzimidazole [14], and polyimide [15,16], have been reported in the existing literature. Among them PAN is mainly used to prepare carbon nanofibers through electrospinning process because of its high carbonization rate and simple carbonization process. Aligned electrospun PAN nanofiber bundle has been used as precursor fibers, which were pretreated by washing, drying densification, and damp-heat drafting, to prepare preoxidized nanofibers with relatively good properties in our former research [17–19]. Based on this, effect of heat treatment temperature on the morphology and structural performance of carbon nanofibers was further investigated in this article.

2. Experimental

2.1. Materials and apparatuses

PAN (M_W = 100,000 $g \cdot mol^{-1}$) was purchased from Shanxi Hengtian New Textile Fiber Tech, China. *N,N*-dimethylformamide (DMF) was obtained from Tianjin Fuyu Fine Chemical Industry, China, which was used directly without further purification.

The vertical electrospinning set-up, which mainly consisted of a DC high-voltage generator (Chengdu Chuangao Electric Technology Co., Ltd.) and a micro-injection pump (Zhejiang University Medical Instrument Co., Ltd.), was used to prepare nanofibers. The magnetic stirrer (Hangzhou Instrument Motor Co., Ltd.) was selected in the preparation of spinning solution. The procedure temperature-controlled sintering furnace, which has been described in our published document, was homemade [19].

2.2. Methods

The selected electrospinning parameters were as follows: PAN was dissolved in DMF to prepare the solution with the concentration of 15 wt.%; flow rate was 0.3 $mL \cdot h^{-1}$; applied voltage was 12 kV; distance between the needle tip and the collector was 12 cm; the collector was plane aluminum plate; collecting time was 40 min; and environmental temperature and relative humidity were 19 ± 2 °C and 45 ± 5 RH%,

* Corresponding author at: School of Textile and Materials, Xi'an Polytechnic University, Xi'an 710048, China.
E-mail address: fzlck@126.com (C.-K. Liu).

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respectively. The aligned nanofibers were treated through the following steps: washing, drying densification, and fourfold damp-heat drafting to prepare precursor fibers. The prepared fibers were used to prepare preoxidized nanofibers with heat treatment temperature at 283 °C for 1 h and subsequent carbon nanofibers through low-temperature carbonization. The carbonization temperatures were selected as 400 °C, 700 °C and 1000 °C respectively with the heating rate of 5 °C·min^{-1}. The effect of heat treatment temperature on the morphology and structural performance of carbon nanofibers was studied. The fiber might shrink in the carbonization process, leading to the formation of the micro-pits on the surface of fiber, which will deteriorate the properties of fiber. So the tension was selected as 0.02 cN·dtex^{-1} in this article, which must be exerted on the fiber in the carbonization process to keep and optimize the orientation structure formed in the preoxidized process.

2.3. Measurements

The changes of molecular structure were measured by Fourier transform infrared spectroscopy (Vertex 70, Bruker) with the KBr pressed-disk technique. The scanning range was 400–4000 cm^{-1}, and 256 scans with a spectral resolution 4 cm^{-1} were selected during each measurement. Raman spectroscopy (InVia, Renishaw) was used to obtain the Raman spectrum of carbon fiber with a scanning range of 200–2000 cm^{-1}. The scanning speed was 10 s with 3 superpositions. The wave length was 514.5 nm and power was 20 mW attenuated to 10%. The changes of element content in the carbonization process were obtained by an element analyzer (Vario EL III, Elementor). Appearance of fibers was characterized by a scanning electron microscope (Vega II, Tescan). Electrical conductivity of fiber was measured by an electrical conductivity meter (Fluke). Mechanical properties of fiber was measured using a strength tester (5565, Instron).

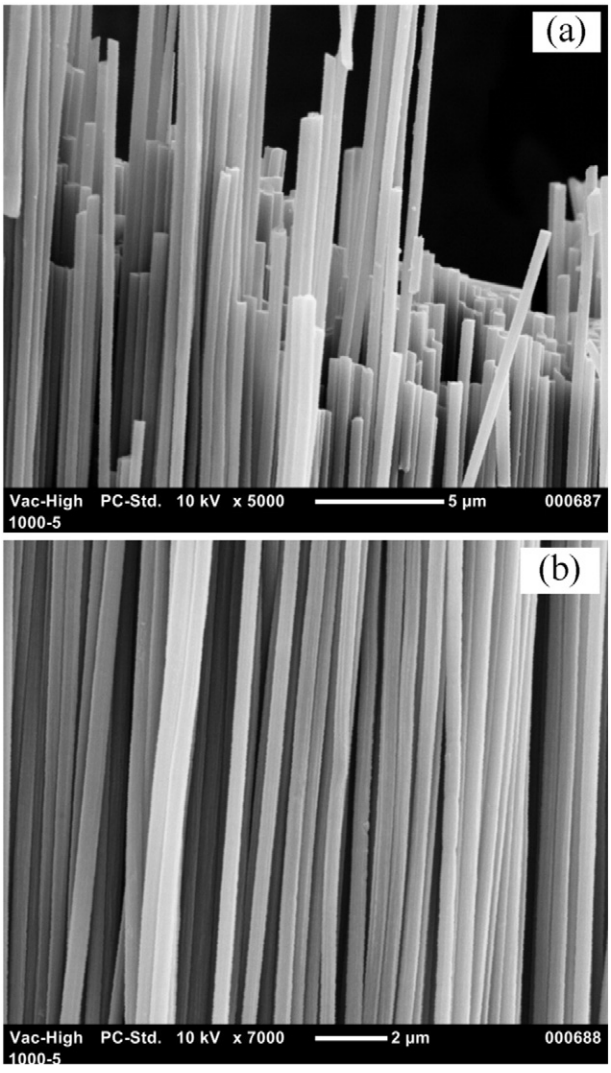

Fig. 1. SEM photos of the fibrous bundle carbonized at 1000 °C: (a) fracture morphology; and (b) surface state.

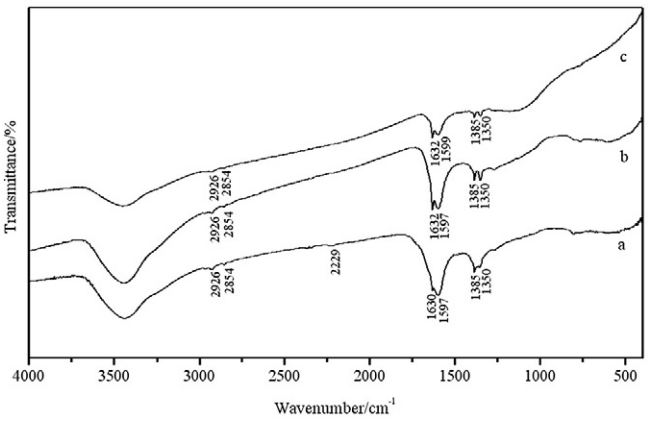

Fig. 2. FTIR curves of fibers carbonized at three different temperatures: (a) 400 °C; (b) 700 °C; and (c) 1000 °C.

3. Results and discussion

3.1. Effect of carbonization temperature on morphology of nanofibers

Fig. 1 shows the SEM photos of the fibrous bundle carbonized at 1000 °C. It is obvious that the fibers do not adhere to each other and being well even-distributed. The diameter of fiber (380 ± 30 nm) after preoxidation and carbonization processes is thinner than the diameter of precursor fiber (410 ± 28 nm) treated by fourfold damp-heat drafting because of the structural change, weight loss of fiber, and the tension exerted on the fiber in the pre-oxidation and carbonization processes. Non-carbon elements are removed through structure rearrangement of PAN preoxidized nanofiber, condensation and polymerization of cyclized molecular chains, resulting in an increased

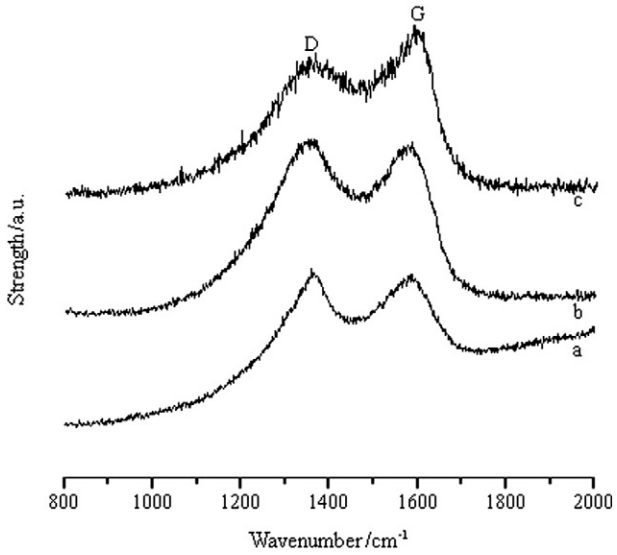

Fig. 3. Raman curves of fibers carbonized at three different temperatures: (a) 400 °C; (b) 700 °C; and (c) 1000 °C.

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