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Mechanical performance of aligned electrospun polyimide nanofiber belt at high temperature

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ABSTRACT

High mechanical performance materials at high temperature are highly desired for the materials used in high temperature industries. This study reports the aligned electrospun polyimide nanofiber belt (A-PI-NFB) fabricated from the electrospinning followed with imidization. The A-PI-NFB showed increased mechanical and thermal mechanical properties as increasing the imidization temperature. At room temperature, the A-PI-NFB imidized at 450 °C (A-PI-NFB-450) had the highest tensile strength (689 MPa) and *E* modulus (13.2 GPa). The thermomechanical analysis (TMA) and dynamic mechanical analysis (DMA) showed that A-PI-NFB-450 possessed 80% tensile strength and 90% modulus when heated at 300 and 350 °C in N_2, respectively. These excellent properties made A-PI-NFB good candidates for use in high temperature filtration, protective clothes etc.

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1. Introduction

Aromatic polyimide (PI) is one class of high performance materials with excellent mechanical properties and thermal stabilities and it has been extensively investigated and applied in aviation, electronics, protection and filtrations, especially in high temperature areas [1–6]. In last decades, many researchers have prepared PI nanofibers (nonwovens, short nanofibers, aligned nanofiber belts and single nanofibers) by electrospinning and applied them in composites and battery separators [3–6]. The results showed that PI nanofibers have good thermal stability and excellent mechanical properties at room temperature. The glass transition and decomposition temperature of PI nanofibers could reach up to 300 and 500 °C respectively [7]. The firstly reported high-strength aligned electrospun PI nanofiber belt (A-PI-NFB) showed a tensile strength of 664 MPa and *E* modulus of 15.3 GPa [5]. Later, Chen et al. found that the single PI nanofiber possessed the super-high tensile strength of 1.7 GPa and modulus of 76 GPa [4]. For high temperature applications, it is very important to study the mechanical properties of materials under high temperature. However, there are countable reports regarding the thermal mechanical properties of nanofibers, especially high performance PI nanofibers. In our previous work, we studied the thermal mechanical properties of electrospun aligned polybenzoxazole nanofiber belts (A-PBO-NFB) and found that A-PBO-NFB showed a retention of tensile strength and modulus over 80% at 350 °C [8]. However, the high cost of the monomer and the complicated preparation process limited the applications of PBO nanofibers [8,9]. Therefore, in this study, we highlight the more conventional material of PI and investigate the mechanical and thermal mechanical performance of electrospun A-PI-NFB under high temperature.

2. Experimental

Preparation of aligned PI nanofiber belt (A-PI-NFB): The precursor polyamic acid solution (PAA, 10 wt%, intrinsic viscosity of 5.73 dl/g measured at 25 °C in N,N-diethylacetamide (DMAc)) was prepared by polycondensation with equimolar 3, 3′, 4, 4′-biphenylteracarboxylic dianhydride (BPDA) and 4, 4′-diaminobiphenyl (BPA) in DMAc at −10 °C for 24 h. The as-prepared PAA solution was diluted with DMAc to 4 wt% for electrospinning. The applied voltage, collecting distance, diameter of spinneret and flow rate were 20 kV, 20 cm, 0.65 mm and 0.4 mL/h respectively. A high rotating disc (diameter: 0.3 m; disc rim: 8 mm; rotating speed: 1500 rpm) was used for collecting aligned PAA nanofiber belt (A-PAA-NFB). After drying (70 °C, 6 h, vacuum oven), the A-PAA-NFB was treated using the following protocol: (1) heating up to 250 °C (10 °C/min, N_2, 30 min annealing); (2) heating up to the final temperature (330 °C, 370 °C, 400 °C, 430 °C and 450 °C, 2 °C/min, N_2, 60 min annealing) to complete the imidization process. The corresponding samples with different imidization temperatures were denoted as A-PI-NFB-330, A-PI-NFB-370, A-PI-NFB-400, A-PI-NFB-430 and A-PI-NFB-450, respectively.

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Characterizations: FT-IR spectra were recorded using a Bruker Tensor 27 spectrophotometer. Quanta 200 scanning electron microscope (SEM) was used to observe the morphologies of nanofibers. Wide-angle X-ray diffraction was carried out using a Siemens D5000 X-Ray Diffractometer (XRD, CuKα radiation, 10°–60°, 5°/min). TGA and DTGA curves were recorded by a thermogravimetric analyzer WRT-3P (Shanghai, 10 °C/min, N_2). Mechanical properties of A-PI-NFB were characterized by using a tensile testing machine (SANSCMT-8102, 5 mm/min). The dynamic mechanical behavior was performed on a Perkin-Elmer Pyris diamond analyzer (DMA, 3 °C/min, N_2, frequency of 1 Hz, amplitude of 5 μm). Thermomechanical properties were carried out on a Perkin-Elmer diamond TMA. The A-PI-NFB was first heated to a final temperature in N_2 with a heating rate of 20 °C/min and then tested with a tension mode with an applied tension speed of 5 μm/min.

3. Results and discussion

Preparation of A-PI-NFB: The smooth and bead-free nanofibers (fiber diameter of 254 ± 27 nm, Fig 1A and 1B) of A-PI-NFB-450 exhibited more than 90% alignment (Fig. 1A, arrow direction) and no melting behavior was observed (Fig. 1A). The imidization process from PAA to PI was characterized by FT-IR spectra (Fig. 1C). When the imidization temperature was above 330 °C, the disappearance of the peaks at 3038 cm^{-1} (CONH, N–H), and 1707 cm^{-1} (C=O), and the appearance of characteristic imide carbonyl peaks at 1770 cm^{-1} (symmetric C=O stretching) and 1710 cm^{-1} (asymmetric C=O stretching) demonstrated the complete imidization process (Fig. 1C) [10]. No difference between the FT-IR spectra of A-PI-NFB-330 and A-PI-NFB-450 indicated that no change happened to the PI molecules when increasing the imidization temperature till 450 °C. TGA and DTGA analyses showed that all A-PI-NFB exhibited excellent thermal stability up to 500 °C and the main decomposition peaks appeared around 612–625 °C (Fig. 1D).

Mechanical and thermal mechanical properties of A-PI-NFB: Imidization temperature had significant effect on the mechanical properties of PI [11]. Fig. 2A shows the mechanical properties of the A-PI-NFB with different imidization temperatures. The A-PI-NFB-330 exhibited the smallest tensile strength of 326 MPa and *E* modulus of 5.0 GPa. As the imidization temperature increases, the tensile strength and *E* modulus of A-PI-NFB also increased. The A-PI-NFB-450 exhibited much higher tensile strength (689 MPa) and *E* modulus (13.2 GPa) than most of the conventional electrospun nanofibers, e.g. normal PMDA-ODA-PI, PBO, nylon-6 and polystyrene [3,8,12–14].

Both the obtained A-PI-NFB-370 and A-PI-NFB-450 showed excellent high temperature resistance as tested by thermomechanical analysis (TMA, Table 1). Almost no change on the mechanical properties of A-PI-NFB was observed when heating the sample up to 250 °C. When the test temperature was above 300 °C, A-PI-NFB-450 exhibited better heat resistance than A-PI-NFB-370. At 300 °C, the mechanical properties of the A-PI-NFB-450 still had 80% retention of tensile strength but only 56% for the A-PI-NFB-370. Even at 400 °C, a completely-decomposing temperature of the common polymer materials, the A-PI-NFB-450 still had a tensile strength of 330 MPa (52% retention) and *E* modulus of 9.38 GPa (72% retention). In a comparison, the A-PI-NFB-370 exhibited smaller tensile strength of 92.5 MPa (21% retention) and *E* modulus

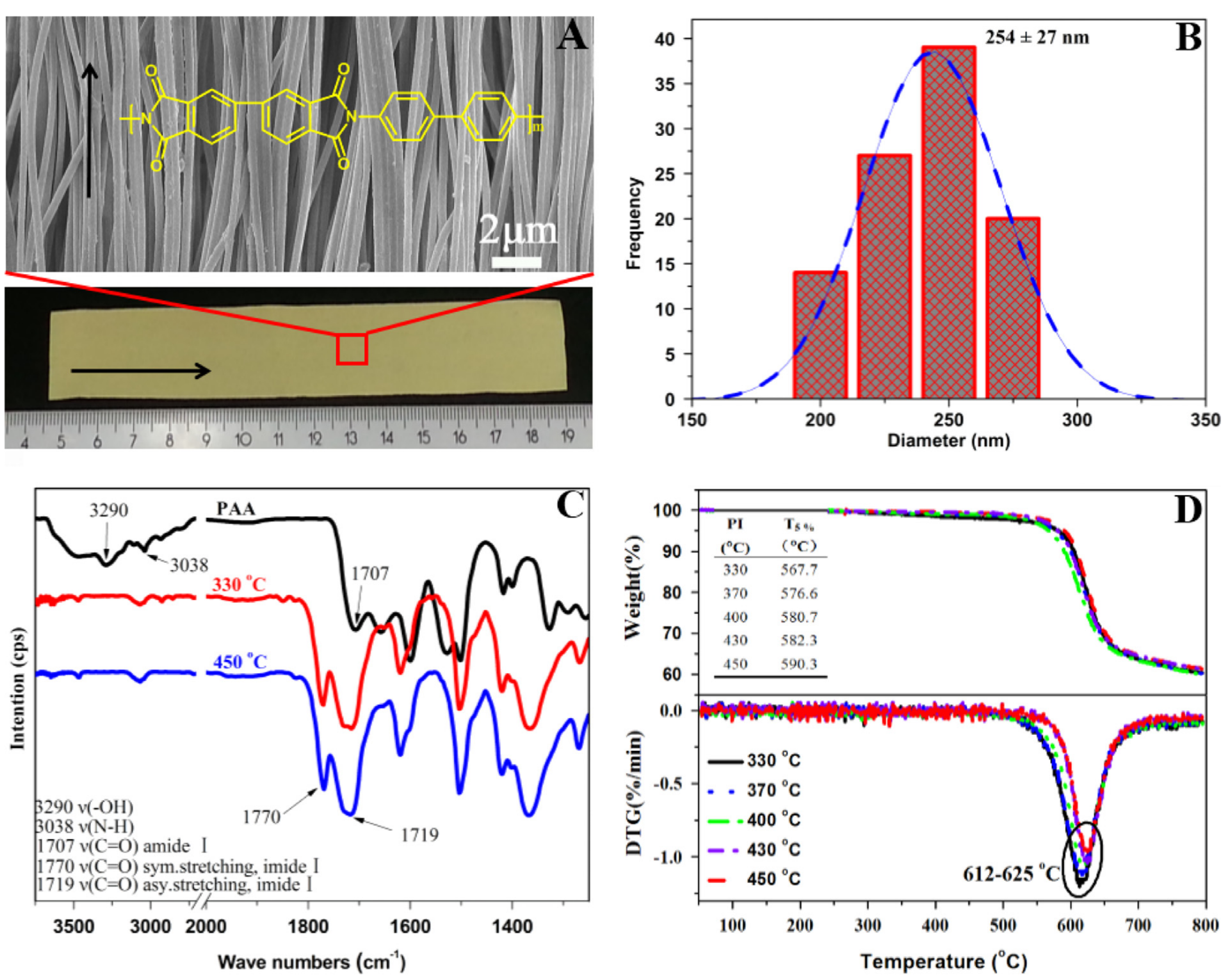

Fig. 1. Morphology of A-PI-NFB-450 (A), diameter distribution (B), FT-IR spectra (C), and TGA and DTGA curves (D) of A-PI-NFB.

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