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Preparation and characterization of macroscopically electrospun polyamide 66 nanofiber bundles

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ABSTRACT

In this study, macroscopic polyamide 66 (PA66) nanofiber bundles were successfully electrospun using two electrode pins as collector. One of the pins can rotate with a speed of 300 rpm. Scanning electron microscope, tensile test, wide-angle X-ray diffraction and differential scanning calorimetry were employed to characterize the arrangement of electrospun nanofibers in bundle, tensile property and microstructure of electrospun nanofiber bundles. Results show that the collector pin's rotating speed not only significantly influences the arrangement of nanofibers in bundle but also the crystallinity and porosity of nanofiber bundles, which further influence the tensile strength and failure strain of nanofiber bundles.

1. Introduction

The technique of electrospinning, a simple but versatile method, has attracted intensive attentions since it can fabricate fibers with diameters from micrometers down to a few nanometers. Nowadays, electrospun fibers are generally collected in the form of nonwoven mats, exhibiting many promising applications in fields of filtration, protective clothing, self-cleaning, drug delivery, electronic and photonic devices, etc. [1–4].

As well known, fibers in nonwoven mat are randomly arranged and non-uniform. Such chaotic structure and relatively low mechanical strength have restricted their further applications. In order to solve the aforementioned problem, nanofiber bundles with macroscopic length were fabricated. Recently, macroscopical nanofiber bundles are desired for a wide range of applications, including tissue scaffolds, reinforced composites and ultrasensitive sensors [3,5,6]. Researchers have employed different methods to fabricate macroscopically nanofiber bundles [3–7]. However, in the current open literatures, there have been few investigations on the mechanical property of electrospun nanofiber bundles, let alone the relationship between the mechanical property and microstructure.

In this paper, we report a facile way to electrospun macroscopic PA66 nanofiber bundles. Furthermore, the arrangement of nanofibers in bundle, microstructure and their influence on tensile property of electrospun nanofiber bundles were explored.

2. Experimental part

2.1. Materials

PA 66 (EPR-27) was purchased from Shenma Group Co., Ltd., China. The PA66 samples had an average molecular weight (M_w) of 2.0×10^4 g/mol and a density of 1.15 g/cm^3. Formic acid (analytical reagent) was bought from Tianjin Chemical Reagents Plant.

2.2. Electrospinning

PA66 granules were completely dissolved in formic acid at 70 °C with stirring for 1 h to get a homogeneous solution with a concentration of 15 wt.% [5]. The modified electrospinning apparatus (Fig. 1a) consists of three components: a high voltage supplier (DW-N503-4ACCD, Tianjin Dongwen High Voltage Power Supply Plant); a spinneret with a diameter of 0.5 mm; two electrode pins with a diameter of 0.5 mm serving as collector. It is worth noting that one of the pins can rotate at a speed of 300 rpm. The distance between the two pins is 3 cm. Electrospinning was performed at an applied voltage of 25 kV over a distance of 25 cm from spinneret to collector. Environmental temperature and relative humidity were 25 ± 2 °C and 65 ± 5%, respectively.

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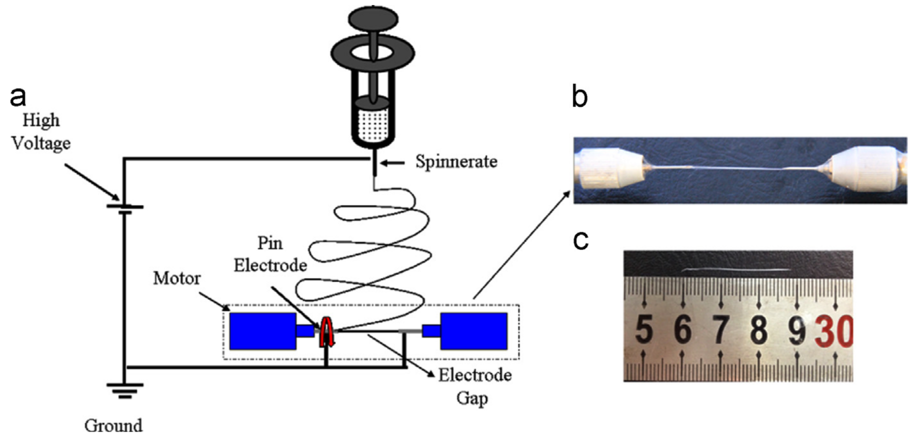

Fig. 1. Schematic of the electrospinning set-up used to fabricate PA66 nanofiber bundle (a); digital photos of the macroscopically electrospun nanofiber bundles (b and c).

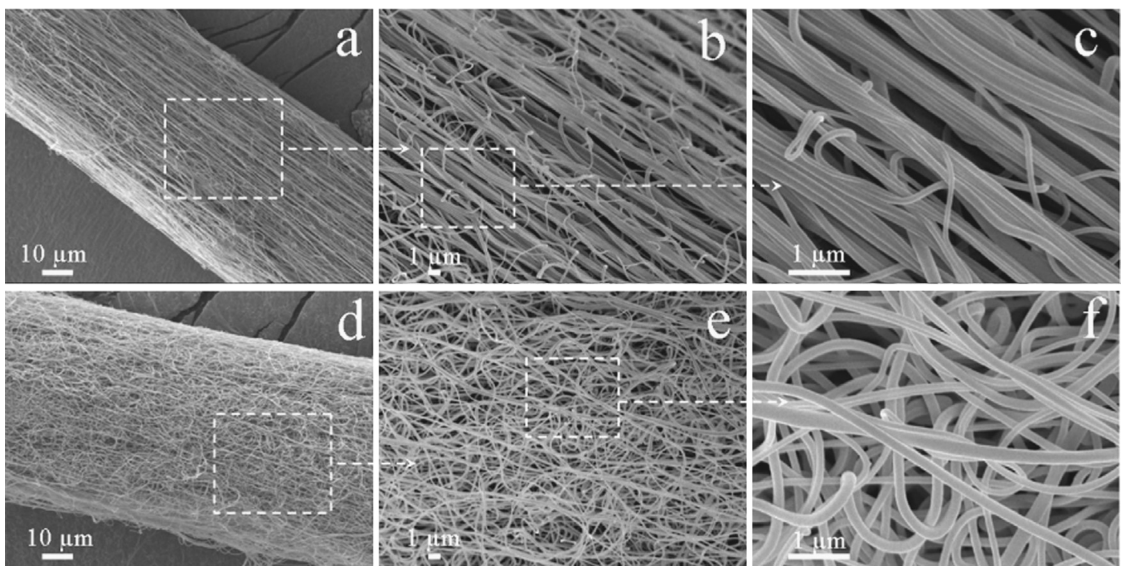

Fig. 2. SEM images of the electrospun nanofiber bundles: PA66-0 (a–c) and PA66-300 (d–f).

The electrospun PA66 nanofiber bundles collected at the speed of 0 rpm, 300 rpm were, respectively, labeled as PA66-0, PA66-300 and dried at room temperature for 24 h before further characterization.

2.3. Characterization

The size and surface feature of electrospun PA66 nanofiber bundles were characterized by SEM (JEOL JSM 7500F) operated with an accelerated voltage of 5 kV. Before observation, the specimen was sputtered with gold.

Tensile property of electrospun PA66 nanofiber bundles were conducted using a universal tensile testing machine (UTM2203, Sun Technology Stock Co., Ltd) at a crosshead rate of 0.1 mm/min with a gauge length of 10 mm. The test was performed at around 25 °C. To avoid inaccurate experiment data caused by personal stretching, we putted the bundle on a self-made paper frame (see Fig. S1 in Supplementary Material). For each condition, the average value reported was derived from at least ten tested specimens.

Crystalline structure of electrospun PA66 nanofiber bundles was characterized by wide-angle X-ray diffraction (WAXD). The measurement was performed using a NanoSTAR-U (Bruker AXS Inc.) with a Cu Kα radiation source (λ=0.154 nm). The generator was operated at 40 kV and 650 μA. The distance from the sample to detector was 1058 mm.

Melting behavior of electrospun PA66 nanofiber bundles was characterized using a TA MDSC 2920 differential scanning calorimetry (DSC). The DSC furnace was purged with nitrogen during the measurement. The procedures are as follows: the samples (ca. 5–10 mg) were heated from room temperature to 300 °C at a heating rate of 10 °C/min.

3. Results and discussion

The macroscopically PA66 nanofiber bundles with a length of approximately 3 cm are successfully electrospun (see digital photos in Fig. 1b and c). The SEM images of electrospun bundles are shown in Fig. 2, in which the diameter of single fiber in bundle (ca. 140 nm) is generally in nanoscale regardless of the pin's rotating speed. As shown in Fig. 2a–c, apart from a small amount of randomly arranged nanofibers in PA66-0, most nanofibers align along the bundle's axis. With respect to PA66-300 (see Fig. 2d–f), only a few nanofibers align along the bundle's axis while most nanofibers arrange disorderly. This indicates that the rotating collector pin shows a significant influence on the arrangement of nanofibers in bundle.

As mentioned above, the arrangement of nanofibers in bundle is substantially influenced by the rotating pin. A question therefore arouses: will such different arrangement change the tensile property