Preparation of Aligned Polyetherimide Fiber by Electrospinning

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ABSTRACT: Fifteen to 20 wt % polyetherimide (PEI) solutions with 1-methyl-2-pyrrolidinone (NMP) were prepared. The electrical conductivity and surface tension of the solutions were determined. The fiber spinning technique of electrospinning was optimized in order to prepare unidirectionally aligned, structurally oriented nanofiber tows. The morphology of the PEI fibers was investigated using field emission scanning electron microscopy (FESEM). The well-aligned fibers with diameters between 0.58 and 0.90 μm (FESEM) were collected by electrospinning 20 wt % PEI solutions with NMP in the range of 8–10 kV onto a target rotating with a surface velocity 9.8 m/s. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 109: 691–694, 2008

Key words: electrospinning; polyetherimide; alignment

INTRODUCTION

There has been a great deal of interest in the electrospinning technique to prepare a wide range of fibers with diameters ranging from several microns to nanometers in size.[1–3] The fiber electrospinning technique has been extensively explored as a simple method to prepare fibers from polymer solutions or melts.[3,4]

In a typical electrospinning process, an electrical potential is applied between a droplet of a polymer solution, or melt, held at the end of a capillary tube and a grounded target.[3] A charged jet of polymer solution is ejected as the applied electric field overcomes the surface tension of the droplet. The trajectory of the charged jet is controlled by the electric field.[3] The jet exhibits bending instabilities due to repulsive forces between the charges carried within the jet.[3] The jet extends through spiraling loops, as the loops increase in diameter, and the jet grows longer and thinner until it solidifies or collects on the target.[3,5] The fiber morphology is controlled by the experimental design and is dependent upon several parameters such as solution conductivity, solution surface tension, polymer content, polymer molecular weight, viscosity, and applied voltage.[3,6]

In the past several decades, electrospinning has been used to generate very fine fibers from a broad range of polymers, including engineering plastics, biopolymers, conducting polymers, and polymer blends. Recently it has been used to prepare nanofibers made from ceramics and composite materials.[3] The main feature of the electrospinning process is that it is a simple means to prepare continuous fibers with unusually large surface-to-volume ratios and pore structure surfaces.[3,6] Because of the chaotic oscillation of the electrospinning jet, a characteristic feature of the electospinning process, randomly oriented and isotropic structures in the form of nonwoven nanofiber mats or webs are often generated due to a lack of control over the forces driving fiber orientation and crystallization.[3] Meanwhile, obtaining continuously aligned nanofibers and high-volume production is very important for many areas such as fiber reinforcement and device manufacture.[1] Several techniques have been developed to align electrospun nanofibers, and some breakthroughs have been obtained.[1] The results are promising, but these methods need to be further improved for practical applications. The technique of using a rapidly rotating drum as the collector[1,3,7] can produce good alignment if the surface velocity is high. Some new techniques can produce well-aligned fibers, but only of limited length,[1,8,9] area,[1,10] and thickness.[1,11]

In the present work, the polyetherimide (PEI) fibers were prepared using both a stationary and a rotating grounded target under variable parameters. The effect of solution concentration and applied voltage on fiber diameter was examined. The influence of applied voltage and take-up speed on the alignment and molecular orientation of the generated fiber was also examined.

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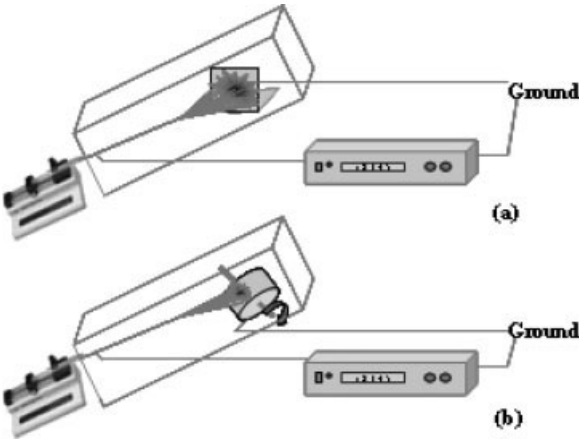

Figure 1 Electrospinning apparatus with a stationary grounded target (a) and a rotating grounded target (b).

EXPERIMENTAL

Materials

Polyetherimide (PEI, ULTEM 1000, melt flow rate 9 g/10 min) was supplied by General Electric Corporation (GE Plastics, Mt. Vernon, IN) and 1-methyl-2-pyrrolidinone (NMP) was obtained from Sigma Aldrich Co. (St. Louis, MO). The polymer was dried before use; PEI was dried between 90 and 100°C under vacuum for 2 h. The 15, 18, and 20 wt % solutions of PEI in NMP were prepared at room temperature under constant mixing.

Electrospinning set-up

The electrospinning apparatus consists of a high voltage power supply, a syringe infusion pump, and a grounded target. Polymer solution is loaded into the syringe and an electrode is clipped onto the needle. The needle, electrode, and grounded target are all enclosed to reduce the effect of air currents on the trajectory of the electrospun jet.[3] The flow rate of the solution to the needle tip is maintained, so that a pendant drop remains during electrospinning. All air bubbles are purged prior to electrospinning, and the solution is electrospun between 6 and 24 kV horizontally onto the target.[3]

TABLE I
The Viscosity, Surface Tension, and Conductivity of the PEI Solutions

PEI concentration (wt %)	Viscosity (cP, at 25°C)	Surface tension (mN/m)	Conductivity (μs/cm)
15	336	14.1	1.8
18	932	14.2	0.8
20	1808	14.7	0.7

The grounded target is between 9 and 14 cm from the charged capillary tip and may be either stationary or rotating[3,12,13] as described elsewhere (Fig. 1). The 4-in. diameter grounded wheel is rotated from 0 to 2300 rpm, producing surface velocities ranging from 0 to 12 m/s.

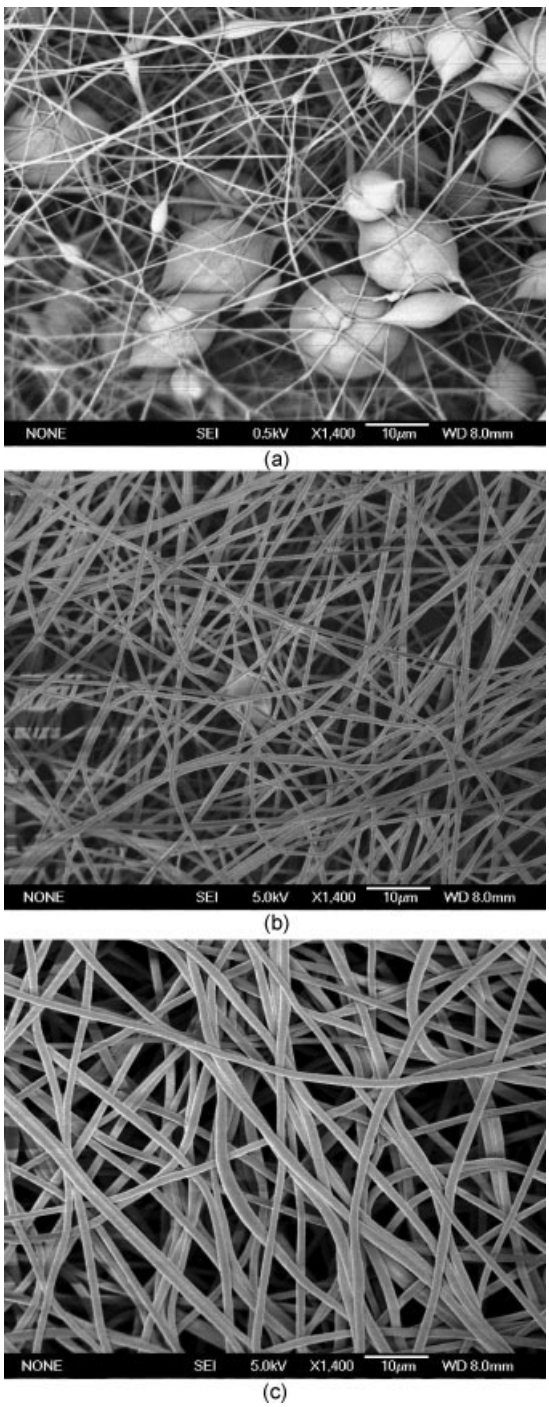

Figure 2 Field emission scanning electron microscopy (FESEM) images of PEI electrospun fibers from 15 wt % (a), 18 wt % (b), and 20 wt % (c) PEI solutions with NMP at 20 kV onto a stationary target.

TABLE II
Average Diameter of Electrospun PEI Fibers as Collected on a Stationary and Rotating Grounded Target with Increasing Applied Voltage, Distance of Needle to Grounded Target and Solution Concentration, Determined by FESEM

Grounded target	Applied voltage (kV)	Distance of needle to target (cm)	15 wt % PEI in NMP (μm)	18 wt % PEI in NMP (μm)	20 wt % PEI in NMP (μm)
Stationary	7	12	–	–	1.365 ± 0.210
	8	12	–	–	1.495 ± 0.250
	10	12	–	–	1.652 ± 0.207
	20	12	0.375 ± 0.175	0.710 ± 0.100	1.525 ± 0.525
Rotating 9.8 m/s	8	14	–	–	0.770 ± 0.130
	9	14	–	–	0.780 ± 0.070
	10	14	–	–	0.715 ± 0.135

Characterization

The electrical conductivity, surface tension, and viscosity of each solution were determined using a standard conductivity meter, surface tension meter, and viscometer. Morphologies such as diameter, surface, and shape of the PEI electrospun fibers were observed by field emission scanning electron microscopy (FESEM). Samples were mounted onto SEM plates, sputter-coated with gold, and examined using a JOEL JSM 6320FXV electron microscope to determine fiber diameters. Measured fiber diameters include a 5% random error.[3]

RESULTS AND DISCUSSION

The viscosity, surface tension, and conductivity of the 15–20 wt % PEI solutions is shown in Table I. The viscosity and surface tension of the solutions increased with increasing solution concentration. In particular, the viscosity of the 20 wt % PEI solution is much

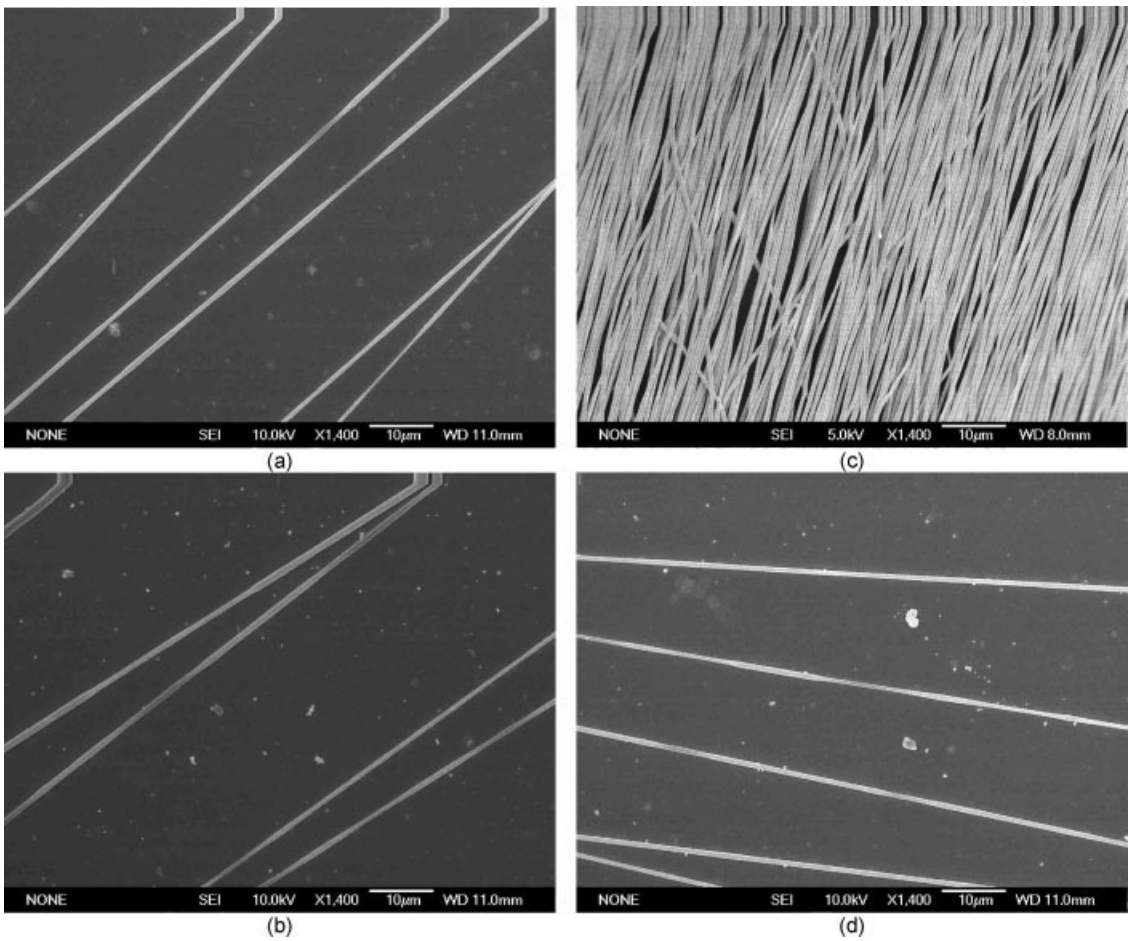

Figure 3 FESEM images of PEI electrospun fibers with an applied voltage of 8 kV (a), 9 kV (b,c), and 10 kV (d) onto a rotating target with a surface velocity of 9.8 m/s.

higher than that of the 18 wt % solution. The electrical conductivity of the solutions is very low.

The concentration, viscosity, conductivity, and surface tension of the solution as well as the applied voltage and distance between the charged electrode and the grounded target were adjusted in order to obtain a stable jet. The droplet of solution at the capillary tip gradually elongates from a hemispherical shape to a conical shape or Taylor cone[14] as the electric field was increased during the electrospinning process.

The standard electrospinning process produces isotropic, amorphous entangled PEI fibers (Fig. 2) because of a lack of control of the forces that drive orientation.[3] The morphologies of 18 and 20 wt % PEI fibers collected on a stationary grounded target were better than those of 15 wt %. Some beads were observed at 15 wt % electrospun fibers, but were not present at 20 wt % electrospun fibers (Fig. 2). Thus, the beads often observed during electrospinning were decreased with increasing the solution concentration.

The diameter of fibers collected onto a stationary grounded target increased as the solution concentration and the applied voltage were increased (Table II). The force pulling the polymer solution jet is increased by increasing the electric field if the distance between the charged capillary and the target are held constant; therefore, an increase in fiber diameter is expected.[3] The diameter of the fibers was increased because the volume percent of solid in the solution and viscosity were increased with the increase of the solution concentration.[3] The diameter deviation of 20 wt % PEI nanofibers collected on a rotating target at 9 kV is smaller than those at 8 or 10 kV.

Well-aligned fibers with diameters between 0.58 and 0.90 μm (FESEM) were collected from 20 wt % PEI solutions with NMP by electrospinning in the range of 8–10 kV onto a target rotating with a surface velocity 9.8 m/s (Table I, Fig. 3). The alignment of the collected fibers is induced by the rotating target and improves as the take-up velocity is increased.[1,3] The 20 wt % PEI solution was shown to have rapid solidification in the range of 16–24 kV on a rotating grounded target because of the fast evaporation of solvent.

CONCLUSION

The fiber electrospinning technique provides a novel way to prepare PEI fiber with diameters in the submicron to micron range. The optimum conditions having good morphologies such as no beading and good alignment of the fiber were observed. The well-aligned PEI continuous fibers were obtained in the range of applied voltage of 8–10 kV and take-up speed of rotating target of 9.8 m/s. It has been shown that the morphology of fiber becomes better at increasing solution concentration and take-up velocity. Electrospinning permits for the production of nondirectional isotropic mats, and unidirectionally oriented yarns using isotropic, continuous precursor fiber. This electrospinning method can be quite versatile and can be developed and optimized for different applications. The PEI is an amber-transparent, high-performance polymer that combines high strength and rigidity at elevated temperatures with long-term heat resistance. The PEI has excellent dimensional stability ($T_g \approx 210^\circ$C) combined with broad chemical resistance. And also, the PEI is inherently flame-resistant and low smoke generating (LOI $\approx$ 47). Therefore, the well-aligned PEI electrospun is expected to be useful for a variety of applications such as heat-resistant membranes, flame-retarding materials, and electronic applications.

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