

## Formation of interfiber bonding in electrospun poly(etherimide) nanofiber web

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Electrospinning is a very useful technique to produce polymer nanofiber webs [1–10]. A pendant droplet of the polymer solution at the capillary tip is deformed into a conical shape under the electrostatic field. When the electrostatic forces overcome the surface tension, a charged jet is ejected. The jet moves towards a ground plate acting as a counter electrode. A thin polymer fiber is deposited on the counter electrode.

Electrospun polymer nanofiber webs have different morphologies depending on the specific polymer properties. Most electrospun nanofiber webs do not have interfiber bonding. If electrospun nanofiber webs have interfiber bonding, the web will have stronger physical properties. In the previous work [5], we prepared and characterized electrospun poly(etherimide) (PEI) nanofibers. The electrospun PEI nanofiber web does not have interfiber bonding. In this study, we treated thermally the electrospun PEI nanofiber web to improve the physical property by introduction of interfiber bonding. Changes of morphology and tensile property of the electrospun PEI web with the interfiber bonding were investigated.

ULTEM 1000 of General Electric Co. was used as poly(etherimide) (PEI). Its glass transition point is 225 °C. 1,1,2-Trichloroethane (TCE, bp = 110–115 °C) of Aldrich Co. was used as the solvent. PEI of 14 wt% was dissolved in TCE. Pasteur pipette with 1 mm of diameter was used as the solution reservoir. A drum shaped counter electrode with a diameter of 220 mm was located below the reservoir. The winding drum was rotated at speed of 30 rpm during the electrospinning. The fibers were collected on aluminum foil covering the tubular layer. The distance between the tip of the capillary and the counter electrode (tip-to-collector distance, TCD) was 150 mm and the applied voltage was 15 kV.

The electrospun nanofiber web was thermally treated at 80–240 °C for 1 h in a convection oven. The morphol-

ogy and diameter of PEI fiber were measured with scanning electron microscope (SEM) of Hitachi S-2350. The thermal properties and content of the solvent remained in the electrospun fiber web were analyzed with a thermogravimetric analyzer of TGA 2050 of TA Instrument. TGA analysis was performed at 30–400 °C with 20 °C/min under N<sub>2</sub> condition. The tensile properties of the electrospun fiber web was measured with the universal testing machine (Instron 4467). The deformation rate was 40 mm/min. The sample dimension was 20 × 50 mm (width and length) and its thickness was 50 μm. Eight samples of each type were tested and the results were averaged.

The diameter distribution of the electrospun PEI nanofibers was measured with SEM. One hundred PEI fibers were selected from ten spots of 80 × 60 μm<sup>2</sup>. The diameter distribution of the electrospun PEI nanofibers is 100–1200 nm and the most probable diameter range is 500–700 nm. Fig. 1 shows TGA curves of the raw PEI and the electrospun PEI fibers. The electrospun PEI fibers show fast weight reduction at 100–160 °C as shown in Fig. 1b. This is due to the evaporation of TCE

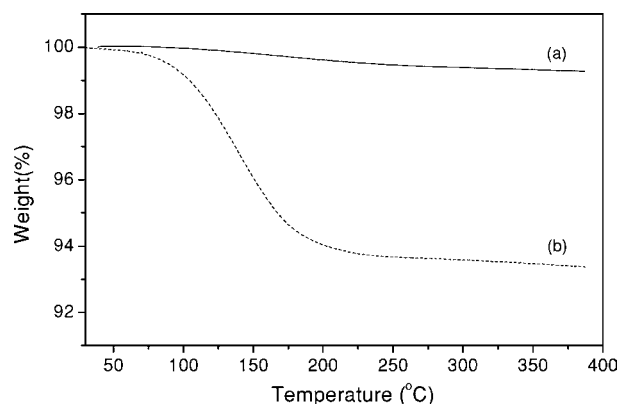


Figure 1 Thermogravimetric analysis (TGA) curves of the raw PEI (a) and the electrospun PEI fibers (b).

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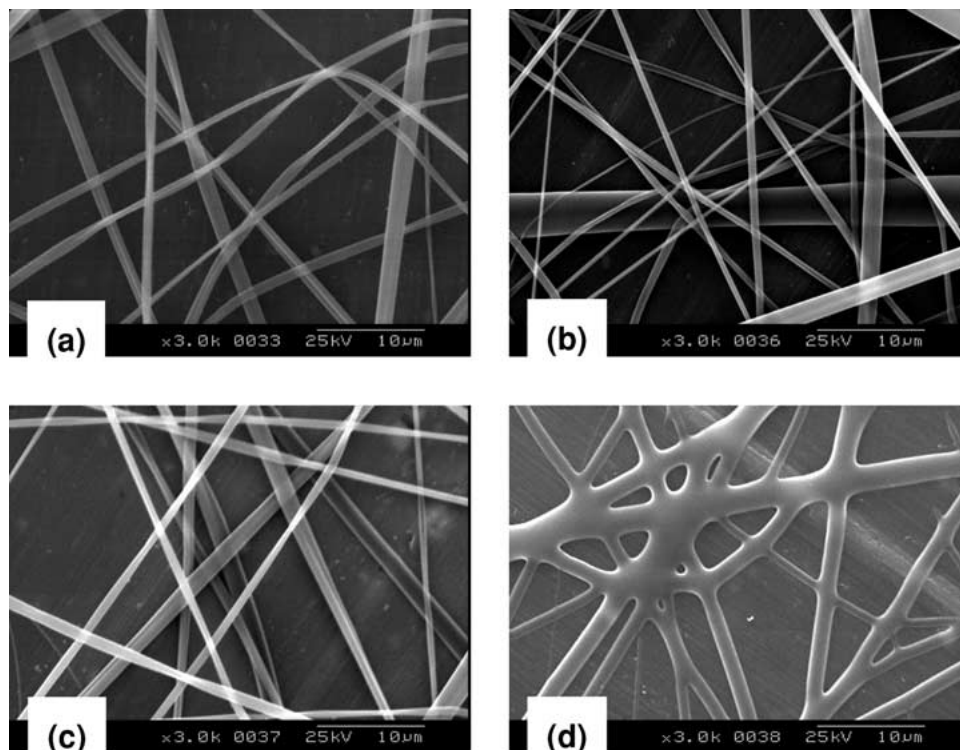


Figure 2 Scanning electron microscopy (SEM) images of electrospun PEI fibers after thermal treatment at 80 °C (a), 150 °C (b), 220 °C (c), and 240 °C (d).

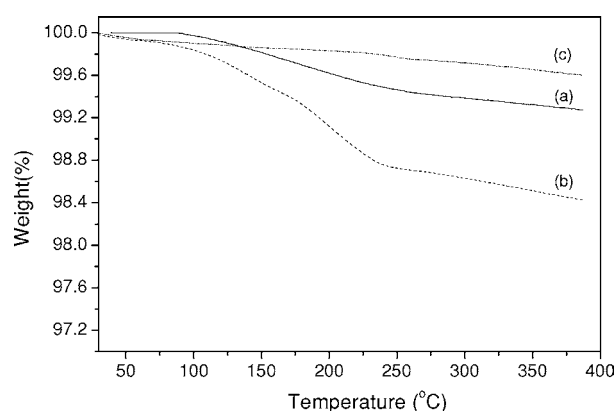


Figure 3 Thermogravimetric analysis (TGA) curves of the raw PEI (a), and the electrospun PEI fibers after thermal treatment at 150 °C (b) and 240 °C (c).

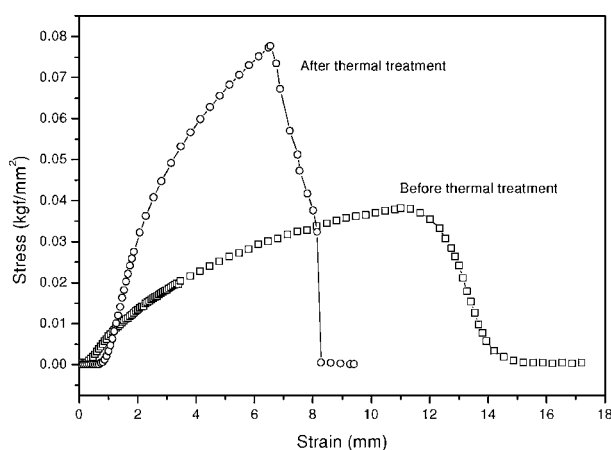


Figure 4 Stress-strain curves of the PEI nanofiber web before the thermal treatment (a) and the PEI nanofiber web treated at 240 °C (b).

remaining in the fibers. Content of TCE remained in the fibers was about 6 wt%.

The electrospun PEI fibers were treated at 80–240 °C to make interfiber bonding. Fig. 2 shows SEM images of the electrospun PEI fibers after thermal treatment. Morphologies of the fibers treated at 80–220 °C are not changed, but the fibers treated at 240 °C show clear interfiber bonding. The glass transition temperature of PEI is 225 °C and the typical temperature of the melt PEI is over 330 °C for the injection molding process. Thus, it can be suggested that the interfiber bonding is formed by softening the electrospun PEI fibers. Since the PEI nanofibers are ultra thin and its surface area is very high, they can be softened effectively and the interfiber bonding is formed. Fig. 3 shows TGA curves of the electrospun PEI fibers after the thermal treatment. The electrospun PEI fibers treated at 150 °C have some TCE as shown in Fig. 3b though the boiling point (110–115 °C) of TCE is much lower than 150 °C. The electrospun PEI fibers treated at 240 °C do not have the solvent and show better thermal stability than the raw PEI as shown in Fig. 3a and c. Weight reductions of the PEI fibers treated at 240 °C and the raw PEI are 0.4 and 0.7% at 800 °C, respectively.

TABLE I Tensile properties of the electrospun PEI nanofiber webs

Sample	Tensile strength (MPa) <sup>a</sup>	Elongation (%) <sup>b</sup>
Before thermal treatment	0.362 ± 0.047	20.6 ± 1.1
After thermal treatment at 240 °C	0.680 ± 0.044	14.3 ± 1.1

<sup>a</sup>Maximum stress.

<sup>b</sup>Strain at maximum stress.

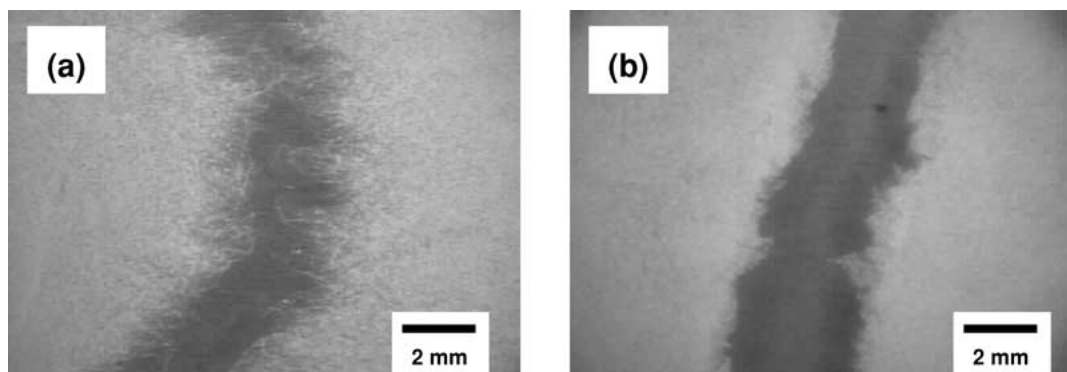


Figure 5 Photographs of the broken samples after the tensile test. (a) the PEI nanofiber web before the thermal treatment and (b) the PEI nanofiber web treated at 240 °C.

Tensile properties of the electrospun PEI nanofiber webs with and without the interfiber bonding were measured. The electrospun PEI fiber web with the interfiber bonding were prepared by the thermal treatment at 240 °C for 1 h in a convection oven. Fig. 4 shows the stress-strain curves of the representative PEI fiber webs with and without the interfiber bonding. For the untreated web, the stress increases smoothly to the maximum point and then also decreases smoothly. For the heat-treated web, however, the stress increases steeply to the maximum point and then drops rapidly. The steeper tensile behaviors of the web are due to the interfiber bonding. The interfiber bonding make the web rigid. The untreated web has longer elongation and weaker tensile strength compared with the jointed web. The tensile strength and elongation were listed in Table I. The tensile strength of the jointed web is about twice that of the untreated web, while the elongation of the jointed web is shorter than that of the untreated one by about 30%. Fig. 5 shows the broken sample after the tensile test. The untreated web shows lots of disentangled and elongated fibers, while the jointed web shows a dense and stiff fracture surface due to the interfiber bonding.

In summary, we can suggest that the interfiber bonding in the electrospun PEI fiber web makes its physical properties improve and is formed by thermal treatment above its glass transition temperature.

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