Keysight Technologies
Tensile Stress-Strain Response of Small-diameter Electrospun Fibers
Introduction

In recent years, electrospinning has become a method of choice for producing ultra-thin high-strength fibers due to its versatility, ease of use, ability to align structures and control fiber sizes [1]. This process is also highly suitable for dispersing different constituent materials into fiber structures. Hence, electrospinning has found increasing importance in the field of fabricating advanced nanomaterials for various applications, such as tissue engineering, filtration, nanofiber composites, etc [2]. In many of these applications, it is extremely important to understand the mechanical properties of the fibers; for example, cell proliferation and growth get affected significantly by the elastic modulus of the constituents in a tissue scaffold.

Given the multitude of applications, there has been increasing interest in measuring the tensile properties of single electrospun fibers. The electrospinning process produces fiber diameters anywhere between a couple of hundred nanometers to a few microns. At the same time it is also argued that electrospinning results in unique intrinsic structures within the fiber geometry. For example, the nanofibers can be stronger than their bulk counterparts because of the ordered arrangement of polymeric chains within the fiber geometry [3–5]. Although a number of recent articles have discussed the processing parameters for electrospinning and potential applications [6] for these polymeric nanofibers, there has been a lack of information on tensile deformation behavior of fibers with such small diameters.

The most obvious reason behind this small amount of information on tensile properties of nanofibers is due to the difficulty of accurately measuring the low loads required for their deformation. There have been a few attempts to address this issue by measuring the mechanical properties of these nanofibers by measuring the deformation of electrospun non-woven fabrics. However, the deformation in non-wovens depends significantly on fiber size distribution, individual fiber orientation, fiber-fiber interaction and entanglement [7]. Hence, it becomes difficult to obtain consistent data because of the large variation in these variables. Recent work has shown that by proper design of the collector it is possible to align fibers, which has found great importance in engineering applications such as tissue engineering, nanocomposites and electronic devices. Moreover, the alignment also helps in handling individual fibers for measurement of their tensile properties. Among a few other work on tensile behavior of single electrospun fibers [4, 5, 8], Tan et al. has reported tensile measurement of aligned individual polycaprolactone (PCL) fibers using the Keysight Technologies, Inc. UTM T150 [4].

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The Keysight UTM T150 (Figure 1) has been specifically designed to address the need for accurate measurement of very low loads and displacements to characterize the tensile properties of small diameter fibers. Some important technical specifications of the instrument can be found in Table 1. To date, the capabilities of the UTM T150 has been successfully utilized to characterize ultra-thin polymeric fibers [4], spider silk [9, 10], lyocell fibers [11], basalt fiber, polypropylene, tungsten [12] and copper wires [13]. The UTM T150 offers high load and displacement resolution to measure the tensile behavior of thin fibers of a wide range of materials. In this study, we demonstrate the capability of the UTM T150 to measure tensile behavior of small diameter PCL fibers synthesized under slightly different processing conditions.

**Materials and Experimental Details**

Thin PCL fibers have been produced by electrospinning a polymer solution consisting of 15 wt% PCL polymer and 85 wt% N, N-Dimethylformamide:chloroform. A schematic of the electrospinning process is shown in Figure 2a. Proper alignment of the fibers is achieved by collecting the fibers across a thick paper-based frame, placed on top of two parallel conductive strips (Figure 2b). The electrospinning process to produce PCL nanofibers can be found in earlier literature [2]. Once the aligned fibers are collected, individual fibers were separated on templates as shown in Figure 3a. These templates consist of parallel card strips where the two ends of the fiber are glued. The sides of the templates are formed using sticky tapes. This template design facilitates proper handling of the fibers before mounting them on the UTM T150, and ease of releasing the fibers for testing. Figure 3b shows a picture of a mounted template in the UTM T150. After the template is properly secured in the grips, the sides of the templates are carefully cut using a hot-blade along the dotted lines shown in Figure 3b. This reduces any unaccounted force on the extremely thin fibers that may be resulted from cutting the sides of the template using a pair of scissors. The diameters of the PCL fibers were measured by an Olympus LEXT Confocal Microscope. The gage lengths of the fibers were determined as the distance between the two glued ends using a caliper. The quasistatic tensile measurements using the UTM T150 were performed in the load-cell mode at a low strain rate of $1 \times 10^{-3} /s$. The engineering stress and strain were calculated from the load on specimen and specimen extension data.

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum Load</td>
<td>500mN (50.8gm)</td>
</tr>
<tr>
<td>Load Resolution</td>
<td>50nN (5.1µgm)</td>
</tr>
<tr>
<td>Maximum Actuating Transducer Displacement</td>
<td>±1mm</td>
</tr>
<tr>
<td>Dynamic Displacement Resolution</td>
<td>&lt; 0.1nm</td>
</tr>
<tr>
<td>Maximum Crosshead Extension</td>
<td>200mm</td>
</tr>
<tr>
<td>Extension Resolution</td>
<td>35nm</td>
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<tr>
<td>Extension Rate</td>
<td>0.5µm/s to 5mm/s</td>
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</table>

Table 1. Specifications for the Keysight UTM T150.
Results and Discussion

The engineering stress-strain curves for electrospun PCL fibers of four different diameters are plotted in Figure 4 (next page). The specimen diameter, gage length, Young's modulus, tensile strength and failure load values are listed in Table 2 (next page). It is important to note the extremely small loads, required for the tensile deformation of these electrospun ultra-thin fibers.

As it is hard to handle these extremely small diameter fibers, only a few PCL fibers of different diameters have been tested during the present study. The tensile strength of PCL fibers increased significantly with decreasing diameter. This may be due to some of the reasons discussed below. The Young's modulus also shows similar trend with decreasing fiber diameter.

Although the tensile strength of the current PCL fibers are of the same order of magnitude compared to earlier report [4], it is difficult to compare quantitatively as properties of polymer change with molecular weight and different processing parameters. However, the tensile strengths of these thin fibers are almost one order of magnitude higher than fibers of larger diameters made from other processing routes [14].

The electrospun fibers of semi-crystalline polymers usually show a structural hierarchy, where some polymer chains form crystalline lamellae and others form an amorphous phase [2]. The amount of lamellar structure determines the crystallinity of the fibers. It has been shown earlier that the electrospun fibers has a “core-shell” type morphology, where the shell consists of oriented layered structure and the core is made of randomly oriented chains [2]. As the fiber diameter decreases, the random coils in the core get oriented along the fiber axis. This in turn increases the tensile strength and Young’s modulus of finer fibers [3]. This core-shell morphology may also explain the small fluctuations observed in the tensile stress-strain curves for the electrospun fibers (Figure 4). The shear force during the electrospinning process also aligns the polymer chains along the fiber length. It is evident that more work is needed to understand the exact effects of crystallinity and molecular orientation on the tensile behavior of electrospun small diameter polymer fibers.

The tensile behavior of electrospun PCL fibers, as shown in Figure 4 (next page), is similar to the previous study [4]. The ductile nature of the curves may be related to the low glass transition temperature (T_g, approximately -60°C) [4]. The tests were conducted at room temperature (above T_g) that may have increased the mobility of the molecular chains allowing easier disentanglement. Hence, this may cause chains to move past each other at low loads. The low T_g also causes annealing of the fibers if kept for a long time at room temperature, and this annealing may affect the tensile properties. So, it can be another important research area to study the behavior of electrospun PCL fibers after different periods of annealing. The draw ratio during the electrospinning process is also known to affect the tensile properties of the electrospun PCL fibers.
Table 2. Results from tensile deformation of PCL electrospun fibers.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Diameter (µm)</th>
<th>Gage Length (mm)</th>
<th>Young’s Modulus (MPa)</th>
<th>Tensile Strength (MPa)</th>
<th>Failure Load (µN)</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>1.5</td>
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<td>5.1</td>
<td>119</td>
<td>13</td>
<td>120</td>
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<td>75</td>
<td>6</td>
<td>90</td>
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</table>

Figure 4. Tensile stress-strain response of electrospun PCL fibers of different diameters.
Conclusions

The tensile stress-strain behavior of small diameter electrospun fibers of PCL was characterized using the Keysight UTM T150. The variation in tensile strength and Young's modulus can be related to the fiber diameter, however more work is needed to address other important factors related to the processing parameters, molecular orientation, crystallinity and annealing time. The tensile measurements of single small diameter fibers have important implications in various applications such as tissue engineering, nanofiber reinforced composites, filtration and filler reinforced fiber systems.

Acknowledgements

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References


References (cont.)


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