Keysight Technologies

Tensile Testing of Fibers using Keysight T150 UTM

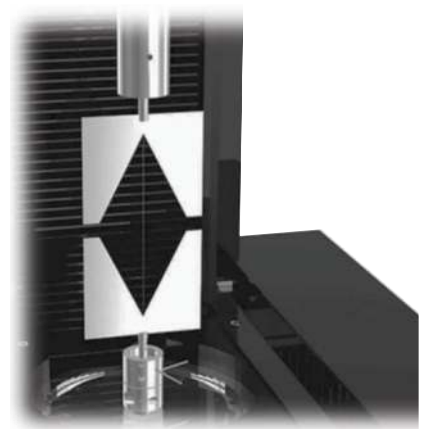
Quasi-static Tensile Test

Application Note

Introduction

Fibers for advanced applications, such as bulletproof vests, biomedical scaffolds, microelectronics, etc. require a good combination of strength, stiffness and toughness. However, characterizing these properties for micro/nano-fibers is challenging because of their low stiffness. Moreover, because of the fiber drawing process, most of the chemical bonds in a fiber are oriented along the fiber axis. This gives rise to a big difference in mechanical properties along the fiber axis compared to the perpendicular direction and, hence, it is not reasonable to interpret fiber properties from their bulk counterparts. Such differences are especially evident in fibers with smaller diameters (few hundred nanometers to a few 10's of microns).

For example, one of the popular methods to fabricate ultra-thin nano/micro-fibers is electrospinning. Despite the importance, most of the studies on mechanical properties have been limited to characterizing the fibermats instead of individual fibers. However, in a recent study, Chen et al. clearly shows that the mechanical behavior of collagen-chitosan fibermats is significantly different from that of the corresponding individual fibers 1. In recent years, multiple studies, including the above reference, have shown ways to design the electrospinning setup to collect aligned nano/micro-fibers those can be easily transferred to templates for tensile testing using the Keysight Technologies, Inc. T150 UTM.



Tensile Test of Thin Fibers: Challenges and The T150 Advantage

The most effective method for mechanical characterization of fibers is uniaxial tensile test. During a tensile test, a fiber specimen is pulled at a specified rate and the resulting reaction force is measured. As we know the initial sample dimensions (fiber length and fiber diameter), several key engineering parameters can be calculated using the measured force and displacement. Some of these parameters are: ultimate tensile strength, Young's modulus, yield stress, yield strain and failure strain. As more and more fibers, especially micro and nano-fibers are being proposed for structural applications, the tensile parameters are important for better simulation and design of structures using these fibers.

As the fiber diameter decreases, the absolute stiffness of a fiber drops significantly, and it becomes challenging to measure the tensile parameters using conventional test equipment. The mass and inertia of the grips and the driveshaft

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for a conventional tensile test system are much greater than the small-diameter fiber samples. Hence, although one may accommodate a fiber sample in a conventional tensile testing system, the accuracy of the results is doubtful. One way researchers try to avoid the challenges is by interpreting the properties of thin fibers from a larger sample of same material, or a yarn (bundle of multiple fibers) or woven/nonwoven fibermat. However, this is not a convincing characterization method because of the differences in molecular alignment, and various elastic and non-elastic interactions between fibers. The force range and the gripping arrangement of the Keysight T150 UTM are more suitable for measuring these small-diameter fiber samples. There are several critical components behind the accurate measurements using a nanomechanical tensile tester like the T150 UTM. These components include a patented nano-mechanical actuating transducer (NMAT) — shown in Figure 1 — that can measure small changes in force to provide a uniquely large dynamic force range (0.0005 to 500 mN) for tensile measurements. A precise extension axis (35 nm resolution) also enables characterization of small diameter fibers at a wide range of strain rates. In addition, the Keysight NanoSuite control software for the T150 not only facilitates precise test control and analysis of data, but also adds the capability to add new test protocols for novel characterization of

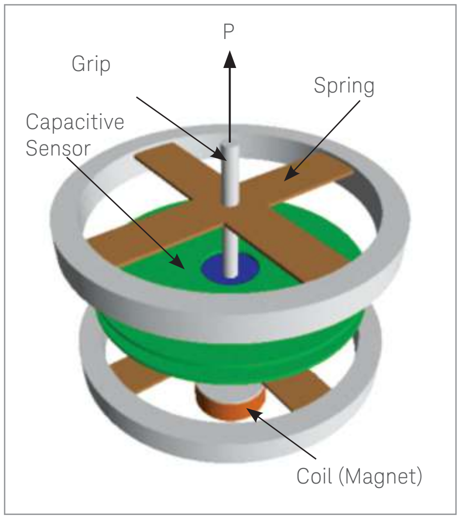

Figure 1. Schematic of nanomechanical actuating transducer (NMAT).

nano/micro-fiber materials. The technical specifications for the T150 UTM can be found in Table 1.

Applications

Polymer Fibers

Figure 2 shows an engineering stress vs. engineering strain curve for a PET fiber, obtained by extending the fiber at a specified strain rate. In this case, the large extension range (up to 200 mm) of the T150 UTM makes it possible to characterize the fibers over the complete range of strain, through breaking. This is especially useful for thin polymeric fibers, which requires small forces for deformation but can extend to large strains. A stress-strain curve for a polymer fiber, such as PET (Figure 2), is extremely important for understanding its mechanical deformation [2-4]. A polymer fiber consists of many polymer chains, and their physical properties govern the properties of the fiber. During a tensile test, extension in a polymer fiber is a result of elastic extension of the polymer chains, and elastic and non-elastic rotation of the chain axis towards the fiber axis. Hence, a polymer fiber with small orientation distribution of the polymer chains exhibits high modulus. However, the strength of a polymer fiber also depends on the nature of overlap between the chains, since the strength of the physical hydrogen or van der Waals bonds between the chains are much weaker compared to the covalent bonds within the chains.

Spider Silk

Spider silk is known to be a wonderful material for many structural and biomedical applications. Different protein glands in a spider produce a variety of silk with diversified arrangement of protein molecules. Hence, it provides researchers a complete toolkit to study the protein molecules and their arrangements that gives rise to the unique combination of high strength and high ductility [5]. However, mechanical testing of the individual silk strands

Maximum load	500 mN (50.8 gm)
Load resolution	50 nN (5.1 µgm)
Maximum actuating transducer displacement	± 1 mm
Displacement resolution	< 0.1 nm
Dynamic displacement resolution	< 0.001 nm
Maximum crosshead extension	200 mm
Extension resolution	35 nm
Extension rate	0.5 µm/s to 5 mm/s
Dynamic frequency range (sample dependent)	0.1 Hz to 2.5 kHz

Table 1. Technical specifications for the Keysight T150 UTM.

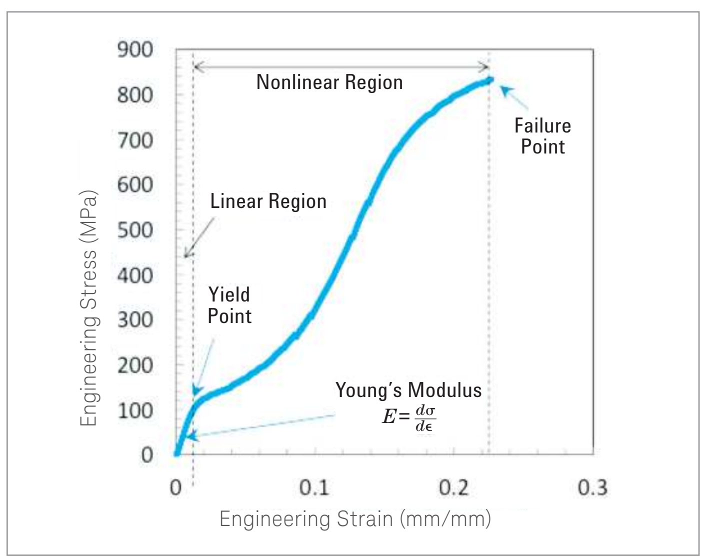

Figure 2. Engineering stress-strain curve for a PET fiber (monofilament) of 17 µm diameter. Note the precise stress and strain measurements for the complete range of strain.

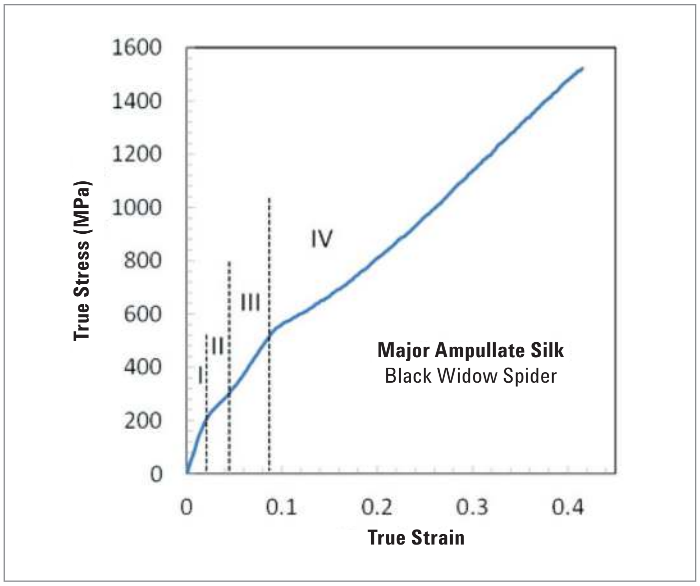

Figure 3. Typical tensile stress-strain behavior of major ampullate silk (diameter 1.8 µm) from Black Widow spider (collaboration with Jeff Yarger and Nik Chawla, Arizona State University).

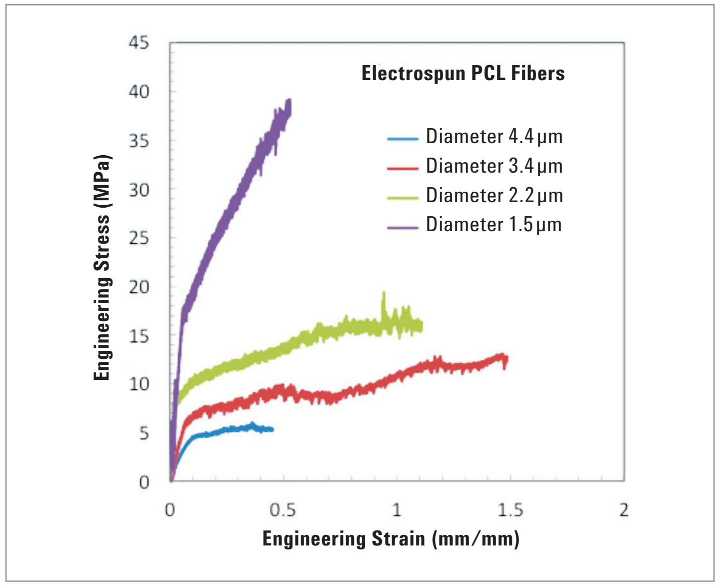

Figure 4. Engineering stress-strain curves for electrospun PCL fibers, showing variation of mechanical properties with fiber diameter (collaboration with Frank Ko, University of British Columbia).

in tension is challenging due to their fine diameter and high compliance. The large dynamic range of the Keysight T150 UTM enables a complete stress-strain analysis of individual spider silk strands with very high resolution [6-8]. Figure 3 is showing a typical true stress vs. true strain curve for a major ampullate silk strand from black widow spider (Latrodectus Hesperus). The failure in this thin fiber (1.8 µm diameter) happened only at 2.5 mN force. More interestingly, the high force and displacement resolution of the T150 enabled the precise measurement of the complete stress-strain behavior, which exhibits four distinct regimes. Regime I corresponds to the linear elastic response due to homogeneous stretching of amorphous molecules. The amorphous protein chains unfold during Regime II causing a decrease in the slope of the stress-strain curve. In Regime III, the amorphous chains align with the crystalline β-sheets resulting in hardening. When the aligned molecular chains starts to slip past each other in Regime IV it causes a decrease in slope, and finally results in failure of the fiber.

Electrospun Fibers

In recent years, electrospinning has become a method of choice to produce nano/micro fibers for applications in filters, biomedical scaffolds, etc.[9-11] Researchers are also trying to mimic the structures of natural fibers, like spider silk, to produce artificial fibers of similar properties. In all these applications, further development of materials and fiber architecture depends on fundamental understanding of mechanical properties. Figure 4 shows the tensile response of thin electrospun poly-caprolactone (PCL) fibers as a function of diameter. A decreasing fiber diameter affects the crystallinity and arrangement of the molecules within an electrospun fiber, usually resulting in an enhancement of Young's modulus and tensile strength[1,11,12]. The ultra-high force resolution of the T150 UTM enables measurement of µN level forces required for the deformation of these thin fibers, and hence accurate measurement of Young's modulus. Figure 4 accurately quantifies the increase in modulus and tensile strength with fiber diameter for electrospun PCL fibers.

Summary

The Keysight T150 UTM is a specifically designed instrument to measure the tensile properties of wide range of fibers with small cross-sectional diameters. The ASTM compliant nanomechanical actuating transducer provides a unique combination of force and displacement resolution, along with a large extension range. These capabilities make it suitable for characterizing thin polymeric monofilaments, metal microwires, spider silk, and various electrospun nano/micro-fibers.

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