Quasi-static and Dynamic Properties of Technical Fibers

Application Note

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Figure 1. The Agilent T150 Tensile Tester in action.

Introduction

Natural and synthetic fibers are used in many products including fabric, insulation, and composite materials. Often, the mechanical properties of the fibers dictate the performance and longevity of the products in which they are used. Generally, it is not sufficient to assume that a fiber will have the same strength as a larger specimen of the same material. This is especially true for metals, because strength depends directly on grain size, and grain size depends on geometric constraints. So for its size, a thin metal wire will generally be stronger than a large specimen of the same material, because the wire has smaller grains. Some polymers also manifest size-dependent strengthening mechanisms [1]. Therefore, the ability to measure the mechanical properties of fibers is essential for their successful incorporation into products. This article describes experimental method and results for three prototypical fibers: a basalt glass fiber, a fine tungsten wire, and polypropylene.

The patented Agilent T150 Tensile Tester (Figure 1) has been specially designed to facilitate fiber testing [2]. It has been used to test ultra-fine polymeric fiber [1], spider silk [3-10], and lyocell fibers [11]. The system is illustrated schematically in Figure 2.
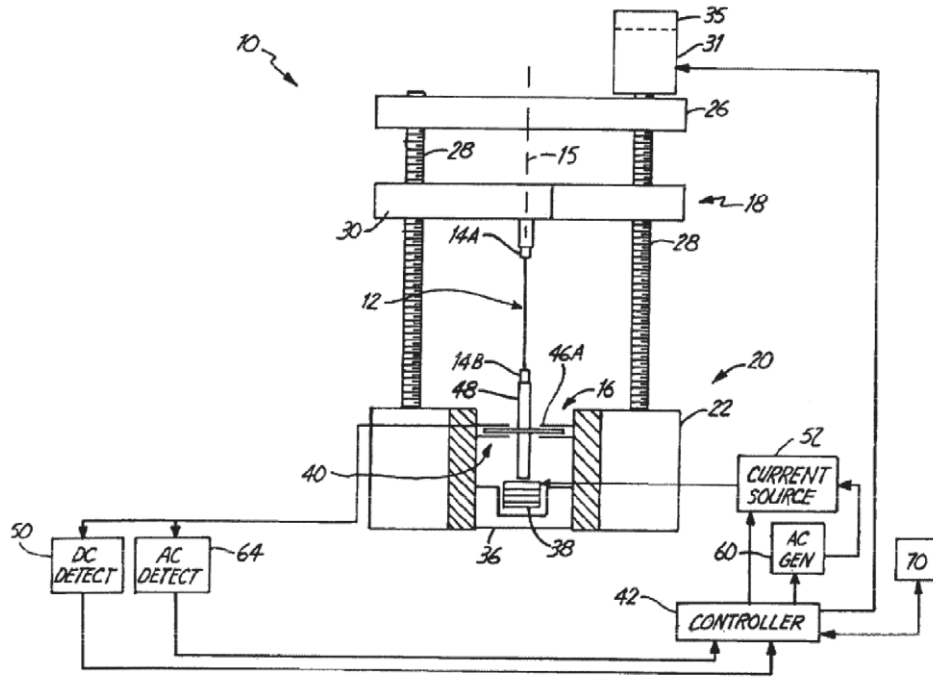
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Figure 2. Schematic representation of the T150 Tensile Tester.

The T150 performs a tensile test in the following manner. At the start of the test, the lower grip (14B) is in its target position. To extend the fiber, the screw-driven crosshead (30) moves up, bringing the upper grip (14A) with it. This motion produces a small perturbation in the position of the lower grip (14B), and this perturbation is sensed by the capacitive gage (16). Meanwhile, an electromagnetic reaction force is applied to the lower grip (14B) by passing current through a conducting coil (38) that sits within an annular magnet (36). By means of a feedback loop that senses the position of the lower grip, the electromagnetic reaction force is increased in order to maintain the lower grip in its target position. Thus, the electromagnetic force, P, required to keep the lower grip in its target position is the tensile force in the fiber. Fiber extension, ΔL, is measured by a device (35) which tracks the number of turns of the screw that drives the crosshead.

Quasi-static analysis

Following conventional tensile analysis, the engineering stress in the fiber is calculated as

$$\sigma = P/A,$$

where A is the cross-sectional area of the fiber. The engineering strain is calculated as

$$\epsilon = \Delta L/L,$$

Where L is the original length of the fiber, and ΔL is the change in length. A plot of stress vs. strain is a useful way to identify key properties of the material. The Young's modulus, E, may be obtained from the slope of the linear part of the stress-strain curve, as this regime manifests elastic deformation.

The yield stress, σ_y, is the stress at which the material begins to deform permanently. Conventionally, this is determined as the stress at which a line passing through the σ-ϵ curve and having a slope of E intersects the strain axis at 0.2%.

Dynamic analysis

In addition to the quasi-static test described in the previous paragraph, the CDA option for the T150 adds the unique ability to determine the properties of a fiber dynamically. This is accomplished by superimposing an AC current through the coil (38). This causes an oscillating force of amplitude F_0 on the lower grip, and the lower grip responds to this force. It oscillates with amplitude z_0, and this response lags the force oscillation by a phase angle, ϕ. Logistically, the response of the lower grip is sensed by monitoring the AC output of the capacitive gage (16) with a frequency-specific amplifier. Dynamic analysis of this system as a simple-harmonic oscillator reveals that the stiffness of the fiber can be calculated at any point during the test as

$$S = \frac{F_0}{z_0}\cos\phi - \left.\frac{F_0}{z_0}\cos\phi\right|_{slack-removal}.$$

Eq. 1

That is, the fiber stiffness is calculated as the real part of the amplitude ratio, less the value of that same parameter prior to engaging the fiber. If the deformation caused by this oscillation is elastic, then we can use the fact that

$$E = \frac{P}{\Delta L}\frac{L}{A} = S\frac{L}{A}$$

Eq. 2

to derive an expression for the dynamic determination of Young's modulus. We combine Eq. 1 and Eq. 2 to determine the Young's modulus at any point during the test as

$$E = \frac{L}{A}\left(\frac{F_0}{z_0}\cos\phi - \frac{F_0}{z_0}\cos\phi\bigg|_{slack-removal} \right),$$

Eq. 3

where A is the instantaneous cross-sectional area, not the initial area. The relevance of this capability is demonstrated for the three prototypical fibers selected for testing. Hereafter, the Young's modulus calculated according to Eq. 3 is called the "storage modulus" to differentiate it from Young's modulus calculated from the slope of the stress-strain curve.

Experimental method

Individual fibers were mounted across a card-stock template as shown in Figure 3. The ends of the fibers were secured with cyanoacrylate. The sample was then mounted in the T150 as shown in Figure 4. (Note: Although the T150 comes with a variety of grips, these "template grips" are the most frequently used.) After mounting the template, the sides of the template were cut away to expose the fiber to the test. The standard test method "UTM-Bionix Standard Toecomp CDA" was used to test all fibers, because it returns Young's modulus as determined by the slope of the stress-strain curve and also by means of Eq. 3. Ten fibers of each type were tested. Fibers were extended to the point of failure. Table 1 summarizes test inputs.
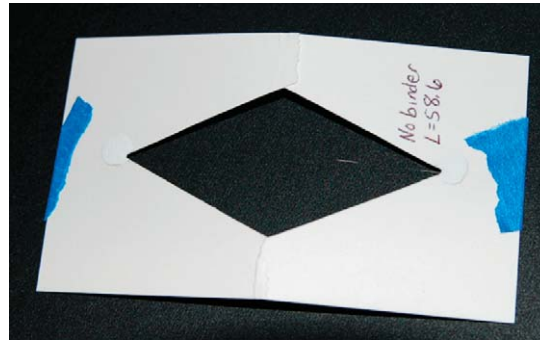

Figure 3. Single fiber mounted on cardstock. Fiber ends are secured with cyanoacrylate.
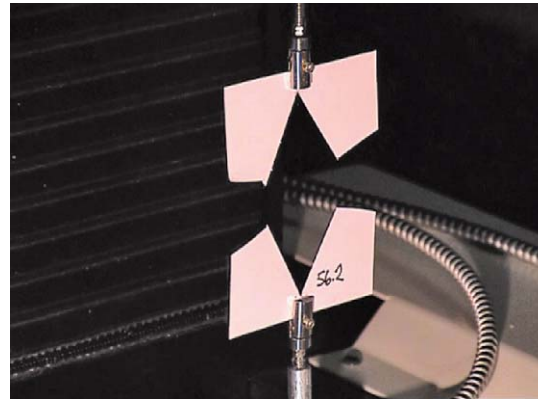

Figure 4. Sample mounted in T150 using template grips. Card has been cut to release sample for testing.

Material	Nominal E, GPa	Ref	Diameter, microns	Strain rate, 1/s	F_0, mN	f, Hz*
Basalt glass	89	12	11-16	1.0e-4	4	20
Tungsten	411	13	12.5	1.0e-4	4	20
Polypropylene	1.5-2.0	14	90-130	2.7e-3	4	20

Table 1. Summary of tests.

* Frequency in Hertz, obtained by the relationship $f = \omega/2\pi$.

Uncertainty analysis reveals that the uncertainty in the fiber diameter is the dominant source of uncertainty in the calculation of Young's modulus. The uncertainty in Young's modulus goes as twice the uncertainty in fiber diameter. In this work, fiber diameter was measured using a Mitutoyo micrometer with a resolution of 0.5 micron. The thinnest fibers tested had a diameter on the order of 10 microns. For these fibers, the 5% uncertainty in diameter (0.5/10) manifests as a 10% uncertainty in modulus. Therefore, substantially lower uncertainty in modulus would be obtained by measuring fiber diameter in a scanning-electron microscope.

Material	E^a, GPa	$s(E)^b$, GPa	$\sigma_y{}^c$, MPa	$s(\sigma_y)^d$, MPa	$\sigma_{UT}{}^e$, MPa	$s(\sigma_{UT})^f$, MPa
Basalt glass	88.9	5.2	N/A	N/A	2262	324
Tungsten	403	6.3	3660	50.0	3930	110
Polypropylene	1.44	0.14	20.59	1.89	27.11	3.10

Table 2. Summary of semi-static results.

[a] Young's modulus from the slope of the σ-ϵ curve in the elastic regime
[b] Standard deviation in Young's modulus over 10 tests
[c] Yield stress at 0.2% offset
[d] Standard deviation in yield stress over 10 tests
[e] Ultimate tensile strength (engineering)
[f] Standard deviation in ultimate tensile strength over 10 tests

Results and Discussion

Tables 2 and 3 summarize the results for this testing. The comparison between Young's modulus as measured quasi-statically and dynamically is particularly interesting and will be discussed for each fiber type.

Material	E^a, GPa	$s(E)^b$, GPa	Strain, % ^{c}
Basalt glass	91.6	5.3	0.5
Tungsten	418	2.2	0.5
Polypropylene	1.32	0.06	15
Polypropylene	8.12	0.49	400

Table 3. Summary of dynamic results.

[a] Young's modulus from Eq. 3, cited at a particular strain
[b] Standard deviation in Young's modulus over 10 tests
[c] Strain value for citing Young's modulus

Basalt glass
Balsalt is a naturally occurring volcanic rock; it has a nominal elastic modulus of 89 GPa. Basalt fiber is created by melting the rock at 1400 C° and extruding the molten rock through small nozzles to create continuous filaments. Basalt fiber has a number of industrial applications. It can be used to produce fiberglass. As a woven textile, it is used in the aerospace and automotive industries as a fire retardant. It is also used as a strengthening fiber in composites [12].

The basalt fibers we tested had a range of diameters between 11 and 16 microns. Figure 5 shows the results for a typical basalt fiber. The blue trace is the stress-strain curve; the Young's modulus derived from the slope of this curve is 90.6 GPa. The fact that it is linear tells us that the deformation is elastic up to the point of fracture. This
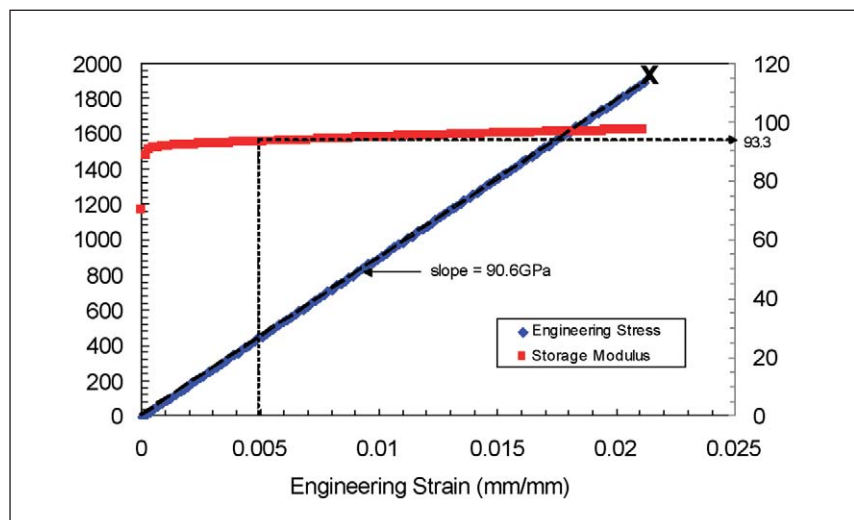

Figure 5. Stress and storage modulus for a single basalt (glass) fiber.
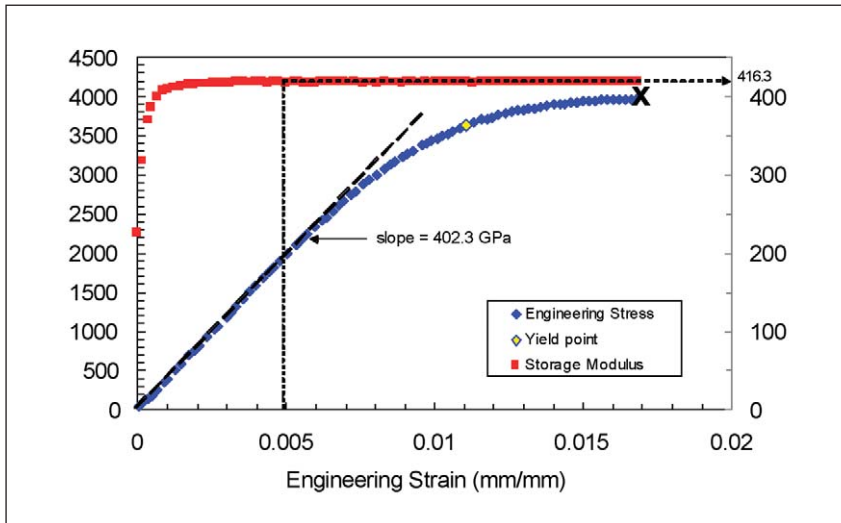
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Figure 6. Stress and storage modulus for a single tungsten wire.

elasticity has several implications. First, it is inappropriate to define a yield point; only the stress (or strain) at fracture is interesting. Second, because the deformation is completely elastic, we expect the Young's modulus to agree well with the storage modulus. The red trace of Figure 5 shows storage modulus as a function of strain. Rather arbitrarily, we pick off the value of storage modulus at 0.5% strain: 93.3 GPa. Averaged over ten tests, the Young's modulus and storage modulus were 88.9 GPa and 91.6 GPa, respectively. This behavior is typical of glass.

For this material, the storage modulus increases slightly as a function of strain. This is an artifact. The decrease in cross-sectional area is calculated assuming substantially plastic deformation; this calculation is consistent with standard analysis for tensile testing. However, for this particular material, it would be better to calculate the reduction in area by assuming elastic deformation and using the Poisson's ratio (~0.2).

Tungsten

Tungsten is an elemental metal. It is isotropic and has a nominal Young's modulus of 411 GPa [13]. Although it has many uses, at this scale, it is used almost exclusively as an electrical conductor. However, electrical conductors must still behave themselves mechanically.

The high-purity, hardened tungsten wire we tested had an extremely uniform cross section of 12.5 microns; all ten samples were taken from the same wire. The blue trace of Figure 6 shows the stress-strain curve for one sample; the Young's modulus derived from the slope of this curve is 402.3 GPa. The departure from linear-elastic behavior is gradual, thus demonstrating the need for a threshold offset for determining yield stress. Because the material is hardened, fracture quickly follows yield.

The red trace of Figure 6 shows the storage modulus as a function of strain. After a brief ramp-up, the value is constant, even for stresses greater than

the yield stress. From this information, we can conclude that the superimposed oscillation does indeed produce only linear-elastic deformation. Again, we pick off the value of storage modulus at 0.5% strain: 416.3 GPa. Averaged over ten tests, the Young's modulus and storage modulus were 403 GPa and 418 GPa, respectively.

Because this wire had been hardened, the Young's modulus and storage modulus agree well. However, this is not the case for more ductile metal wires. For example, the first test of a copper wire will give a Young's modulus that is substantially lower than the storage modulus (and the true value). This is because there is really no part of the stress-strain curve that is truly linear. That is, plasticity initiates at very small stresses and inevitably influences the quasi-static determination of Young's modulus by decreasing the slope of the stress-strain curve. By contrast, the storage modulus is unaffected by plasticity, because the deformation caused by the oscillation is sufficiently

small. As the wire is worked by stretching it and relaxing it, the Young's modulus gradually approaches the storage modulus. Therefore, for thin wires of soft metal, it is generally better to determine Young's modulus dynamically. But for this hardened tungsten, the advantage of dynamic testing is marginal.

Polypropylene

Polypropylene is a thermoplastic polymer; literature values for Young's modulus range from 1.5 GPa to 2 GPa. The widespread use of this material is best conveyed by its recycling code, "5". Practical uses for polypropylene that require knowledge of mechanical properties include: ropes, packaging material, dielectrics, and medical tools [14].

The polypropylene fibers we tested had a range of diameters between 90 and 130 microns. Figure 7a shows a typical stress-strain curve at low strains. The Young's modulus derived from the slope of this curve is 1.44 GPa and this agrees well with the storage modulus at 15% strain: 1.41 GPa. However, Figure 7b shows the same information over the entirety of the test and reveals that this small-strain modulus is a woefully inadequate characterization of the elastic behavior of this material. From this plot, it is clear that for polypropylene, Young's modulus is not a single value, but rather a strong function of strain. Polypropylene stiffens as it deforms. At the maximum strain, the storage modulus is nearly ten times the small-strain Young's modulus! Polypropylene is used precisely because of its extreme extensibility, so this is valuable information for engineers working with this material!

The mechanism for this stiffening is the stretching of long polymer chains. The phenomenon is similar to what happens when stretching a coiled spring of low stiffness. At first, the coil stretches easily, because the means for elastically accommodating the
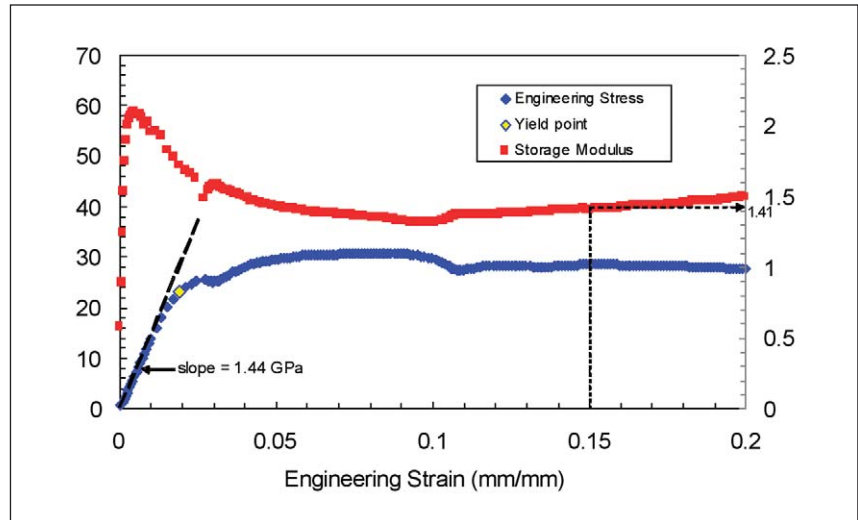

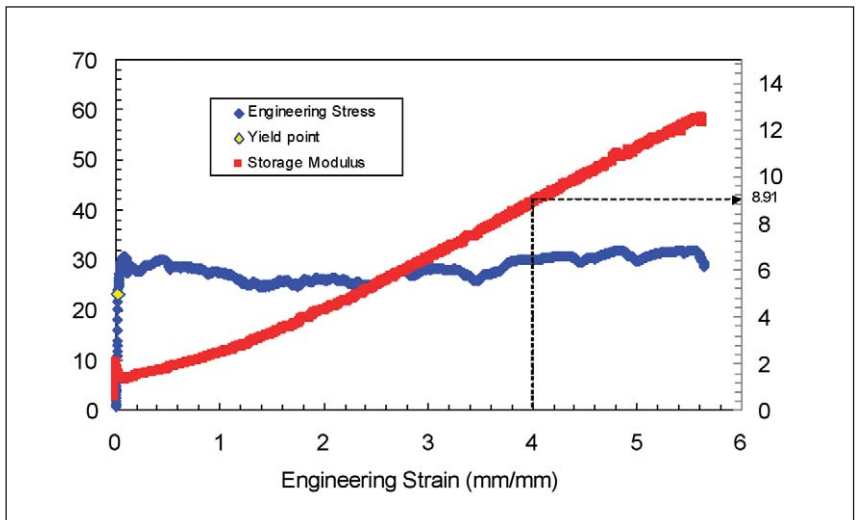Figure 7a. Stress and storage modulus for a single polypropylene fiber, up to 20% strain.

Figure 7b. Stress and storage modulus for entire test on the same fiber as Figure 7a.

deformation is separation of the coils. As stretching continues, however, the means for elastically accommodating the deformation gradually changes. Just prior to the onset of plasticity, elastic deformation is accommodated by elastic deformation of the material comprising the coil, and this material is generally much stiffer than the coil itself. Thus the coil gradually stiffens as it is extended. Polypropylene stiffens in tension by much the same mechanism

– polymer chains unravel and align as they are pulled taut. (This mechanism is also the explanation for the "jaggedness" of the stress-strain curve in Figure 7b.) For polypropylene and other polymers, dynamic determination of Young's modulus as a function of strain is an essential aspect of materials characterization. This feature is what has made the T150 the singular choice for scientists working with spider silk [3-10].

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Conclusions

The Agilent T150 Tensile Tester is the only commercially available instrument in the world that allows dynamic characterization of Young's modulus as a continuous function of strain. For the basalt fiber, the storage modulus agreed well (within 3%) of the value obtained from the slope of the stress-strain curve, because the material was linear elastic to the point of failure. Other glass fibers should be expected to behave similarly. Similar agreement was achieved for the hardened tungsten wire, although such agreement is not typical for more ductile metals, for which plasticity can unduly affect the slope of the stress-strain curve from the outset of the test. Therefore, for thin metal wires, storage modulus is generally a better indicator of the true Young's modulus. Finally, for the polypropylene, the single quasi-static value of Young's modulus was simply an inadequate description of the material. Dynamic assessment of Young's modulus revealed the parameter to be an increasing function of strain, due to the extension and alignment of polymer molecules. Generally, polymer (and biological) fibers should be expected to manifest this type of behavior to some extent. Therefore, dynamic assessment of Young's modulus is necessary for characterizing such materials.

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