Keysight Technologies
Statistical Variation and Strain-Rate Sensitivity of the Mechanical Properties of Individual PET Fibers
Application Note

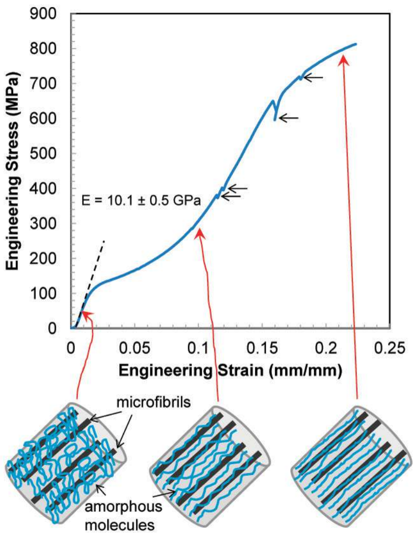

Figure 1. Typical engineering stress-strain curve of an individual PET fiber. The schematics outline the evolution in the molecular structure of amorphous polymer chains during different regimes of deformation.

Introduction

Poly(ethylene terephthalate), or PET (also known as polyester), is a thermoplastic polymer used extensively as synthetic fiber. PET fibers are popular because of their improved wrinkle resistance, durability and high color retention [1, 2]. Most characteristic physical properties of PET fibers are attributed to the presence of benzene rings, which also lead to high stiffness of the polymer chains [1].

The polymer chains in one individual PET fiber can be distributed in crystalline, oriented semi-crystalline and non-crystalline (amorphous) regions [3]. A PET fiber consists of microfibrils aligned along the fiber axis. These microfibrils, in turn, consist of crystalline and amorphous regions, and connected to other microfibrils by another kind of amorphous phase, known as mesamorphous phase. The different regions observed in the tensile stress-strain curve (Figure 1) can be explained by the deformation of the different microstructural regions mentioned above. During the initial deformation, the amorphous regions within the microfibrils align themselves in the similar orientation as the mesamorphous phase. The stress-strain curve goes through another point of inflexion when the applied load starts to strain the bonds in both amorphous and crystalline phases [3]. The final part of the curve represents slippage between microfibrils. It is important to note here that the changes in elastic properties of a PET fiber during these different deformation regimes were significant and have been measured previously using continuous dynamic analysis (CDA) [4].

During the deformation of the amorphous regions, stress concentration in the surface layer of the fiber results in surface cracks and crazing in the PET fibers (horizontal arrows in Figure 1). The propagation of one or few of these cracks through the sample is a stochastic process depending on the distribution of crystalline and amorphous regions in the core of the fiber. Hence, it is not only important to characterize the mechanical properties of individual PET fibers, but also to understand the statistical variability in their response that can be extrapolated to the underlying molecular structure and processing conditions.

Failure in materials is often a stochastic process because of the distributions of defects and molecular structure, and it has been successfully shown to follow a Weibull statistical distribution of the form [5]

$$P_f = 1 - exp\left[-\left(\frac{\sigma}{\sigma_0}\right)^m\right] \tag{1}$$

where, P_f is the probability of failure of a fiber at a stress less than or equal to σ, σ_0 is the characteristic strength, and is the Weibull modulus that describes the variability of the failure strength. A high Weibull modulus represents a more uniform distribution, and hence better reliability. Rearrangement of the Weibull distribution (Eqn. 1) provides

$$\ln\ln\left(\frac{1}{1-P_f}\right) = m\, ln\sigma - m\, ln\sigma_0 \qquad (2)$$

and hence the characteristic strength and Weibull modulus both can be obtained from a plot of $\ln\ln(1/1\text{-}P_f)$ versus lnσ (known as Weibull plot). The slope of the linear fit should produce the Weibull modulus and the intercept should produce the characteristic strength. In this current application note, the statistical variation in the tensile strength of individual PET fibers is analyzed with respect to a Weibull distribution to shed more light on the variability of the manufacturing process.

Another important parameter that also affects the yield strength and tensile strength of a fiber is strain-rate, or the rate at which the material is being deformed. The relationship between strength, σ, and strain-rate, $\dot{\epsilon}$, can be represented as [5]

$$\sigma = A\dot{\varepsilon}^n \qquad (3)$$

where, is a constant depending on material and environmental parameters, and is the strain-rate exponent that can be determined from the slope of the plot of $\ln\sigma$ versus $\ln\dot{\epsilon}$. A low value of strain-rate exponent indicates less influence of deformation rate on the mechanical strength. This work addresses the influence of strain-rate on the yield strength and tensile strength of individual PET fibers.

Experimental Details

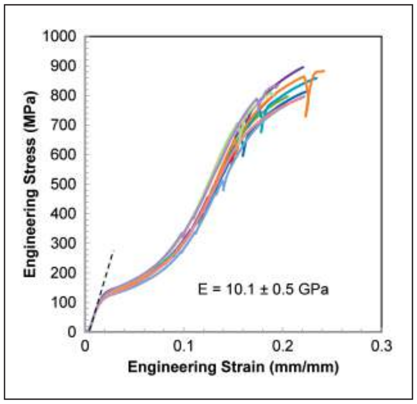

Figure 2. Engineering stress-strain curves obtained from quasi-static tensile tests on seven different PET fibers. Note the variation in tensile strength values.

Quasi-static tensile tests on commercially available PET fibers were performed using the Keysight Technologies, Inc. T150 UTM. The diameter of the individual PET fibers (also known as filaments) was uniform at 17 µm, as measured from optical microscopy. The Keysight T150 – because of its unique nano-mechanical actuating transducer (NMAT) design – is capable of measuring small forces needed for failure of micro and nano fibers with very high precision. Hence, the quasi-static measurements not only generated accurate tensile strength values, but also provided us with the complete stress-strain response with information about Young's modulus and yield strength. Each PET fiber was carefully mounted on thick paper-based templates and the gage lengths were measured using a caliper. The surface morphology of one of the fibers was observed in an SEM after the tensile test to determine the failure process.

For determination of the statistical variation in tensile strength, seven tests were performed at a constant strain rate of 1 x 10^{-3} per second. Once all the tensile strength values were determined, they were ranked 1 through 7 and arranged in ascending order to perform the Weibull analysis. The failure probability, P_f, for each tensile strength was calculated as

$$P_{f,i} = \frac{i}{N+1};\ i=1,2,\ldots,7;\ N=7 \qquad (4)$$

The data were then plotted and the Weibull modulus was determined from Eqn. 2.

For determination of the strain-rate exponent, three tensile tests were performed at strain-rates of 1 x 10^{-1}, 1 x 10^{-2} and 1 x 10^{-4} per second, each. The previous set of tests used for determining Weibull modulus, were also used as the dataset for the strain-rate of 1 x 10^{-3} per second.

Results and Discussion

The engineering stress-strain curves from the seven tests performed at 1 x 10^{-3} per second strain-rate are shown in Figure 2. The general shape of the curve is consistent with previously observed deformation behavior of PET fiber [3, 4]. The Young's modulus measured from the initial linear regime of the stress-strain curve is 10.1 ± 0.5 GPa, and has little variation from one fiber to another. However, a clear distribution of tensile strength is visible. Figures 3a and 3b show the surface morphology near the failure point in one of the fibers. As shown in Figure 3a (horizontal arrows), the failure in these PET fibers occur due to formation of cracks on the surface. The morphology also indicates that the fiber most-likely possess a core-shell structure, where the cracks initiate in the shell. This behavior is similar to most ceramic fibers where

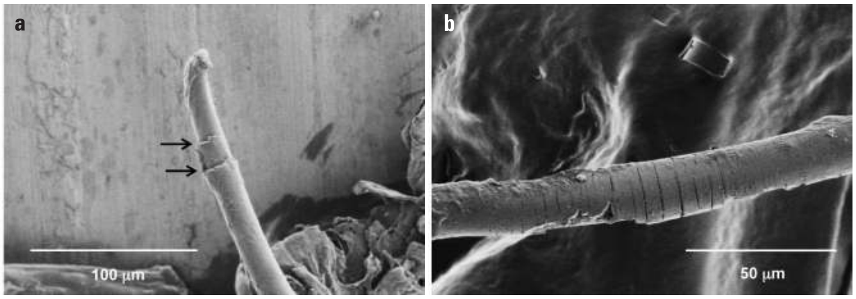

Figure 3. (a) SEM micrograph showing cracks near the failure point of the PET fiber. (b) Fiber surface showing parallel cracks perpendicular to the loading axis, indicating surface crack nucleation and crazing during deformation.

Rank, i	Tensile Strength, σ (MPa)	Probability of Failure, $P_{f,i}$	ln(σ)	ln ln (1/1-$P_{f,i}$)
1	797	0.125	6.680855	-2.01341868
2	805	0.25	6.690842	-1.24589932
3	812	0.375	6.6995	-0.75501486
4	836	0.5	6.728629	-0.36651292
5	859	0.625	6.755769	-0.01935689
6	883	0.75	6.783325	0.32663426
7	895	0.875	6.796824	0.732099368

Table 1. Weibull analysis of tensile strength of individual PET fiber.

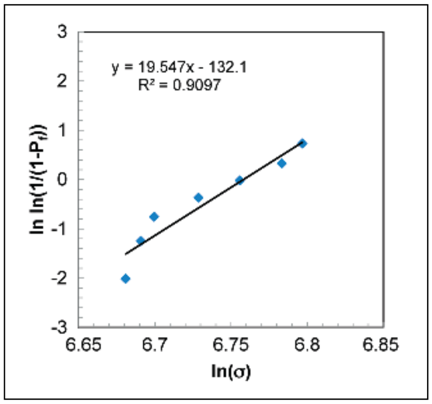

Figure 4. Weibull plot showing distribution of tensile strength of PET fibers.

any surface defect can act as a crack nucleation site. However, the origin of the cracks in these PET fibers may be slightly different. A closer look at a nearby location on the fiber (Figure 3b) demonstrates clear signs of formation of an array of cracks perpendicular to the tensile axis. This indicates that these cracks can be linked to crazing (damage accumulation and shear band formation ahead of crack tip) in the fiber. Similar to the formation of cracks in ceramic fibers, these cracks in the PET fibers are also stochastic in nature resulting in the variation of tensile strength values observed in Figure 2.

The statistical analysis is performed according to the Weibull distribution, and the calculated values are listed in Table 1.

Figure 4 shows the Weibull plot for PET fibers. The linear fit of the data points result in a Weibull modulus, m, of 19, and a characteristic strength, σ_0, of 861 MPa. It is interesting to note here that, in comparison, typical Weibull modulus for technical ceramic fibers is about 5–10, and for metals it can be as high as 90–100 [5]. In other words, when the failure is more ductile the material behaves more uniformly compared to the situations when crack forms. The Weibull modulus is an important quality control parameter for fibers, and the origin of the uniformity (or, lack thereof) can be traced back to the manufacturing parameters. Depending on the application requirements, the process may be tuned to produce fibers with certain uniformity in defect distribution.

The stress-strain curves from three different strain-rates were plotted together in Figure 5. While Figure 5a shows a more overlapping behavior in terms of tensile strength, the magnified low-strain region, shown in Figure 5b, exhibits clear differences in yield strength. Note that there is no variation in the Young's modulus of PET fibers due to different strain-rates. The logarithms of the yield strength and tensile strength were plotted against the logarithm of strain rate in Figures 6a and 6b, respectively. It is evident that even if the yield strength of PET fiber is strain-rate sensitive, there is almost no significant strain-rate sensitivity for the tensile strength.

The reason behind such a behavior lies in the evolution of the molecular

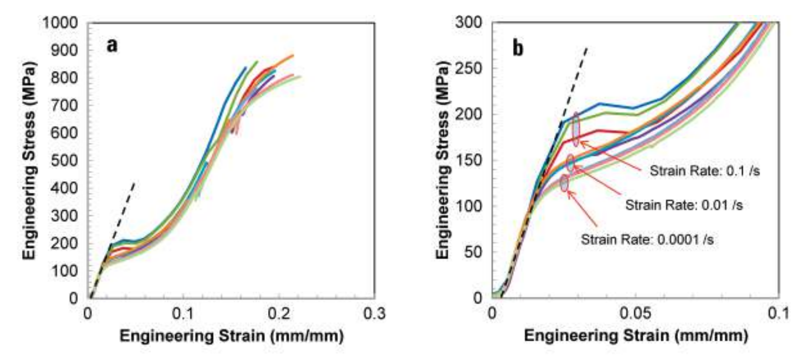

Figure 5. (a) Engineering stress-strain curves for PET fibers tested at three different strain-rates. (b) Magnified view of the curves shown in a, indicating significant strain-rate sensitivity of the yield strength.

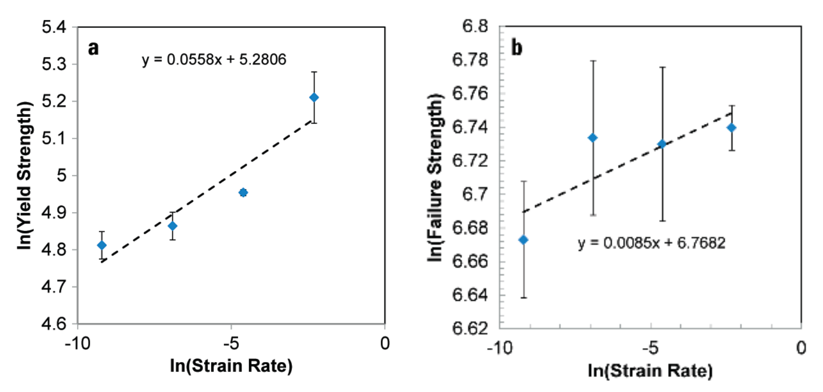

Figure 6. Strain-rate sensitivity of (a) yield strength, and (b) failure strength of PET fiber.

structure during different deformation regimes. The yield happens when the amorphous polymer chains between the crystalline microfibrils start to orient along the axis of deformation [3]. Hence, the faster the deformation, the higher is the time lag for orientation of amorphous molecules resulting in higher yield strength. The strain-rate exponent for the yield strength, calculated from Figure 6a, is about 0.05 and is very similar to other semi-crystalline polymers [6]. On the other hand, the statistical variation in tensile strength for each strain-rate is high, and the strain-rate exponent of 0.008 is not significant. This statistical variation may be due to the variability in crack nucleation on the fiber surface as described by the Weibull analysis, discussed above.

Conclusions

The statistical variation in tensile strength of individual PET fibers is analyzed according to the Weibull distribution function. A Weibull modulus suggests that the distribution of crack nucleating defects in PET fibers is more uniform than most technical ceramic fibers – but the failure is not as uniform as ductile metal fibers. Because of the high variation in defect distribution, there is no significant strain-rate sensitivity for the tensile strength of the fibers. However before the nucleation of the cracks, the yield strength, representing orientation of amorphous molecules along the fiber axis, shows strain-rate sensitivity similar to other semi-crystalline polymers.

References

1. http://web.utk.edu/~mse/Textiles/Polyester%20fiber.htm.
2. http://en.wikipedia.org/wiki/Polyethylene_terephthalate.
3. Lechat, C., et al., *Mechanical behaviour of polyethylene terephthalate & polyethylene naphthalate fibres under cyclic loading.* Journal of Materials Science, 2006. 41(6): p. 1745.
4. Basu, S., *Tensile deformation of fibers used in textile industry.* Keysight Technologies Application Note, 2014.
5. Meyers, M.A. and K.K. Chawla, *Mechanical behavior of materials* 1999: Prentice-Hall.
6. Goble, D.L. and E.G. Wolff, *Strain-rate sensitivity index of thermoplastics.* J. Mater. Sci., 1993. 28: p. 5986–5994.

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