

Keysight Technologies

Evolution of Dynamic Mechanical Properties of Nylon-PET Island-in-the-Sea Bicomponent Fibers

Application Note

Introduction

A bicomponent fiber is a single fiber that contains two different polymer components within the same filament. Sometimes it is also known as a 'conjugate' fiber¹. The concept of producing bicomponent fibers is not very new as Dupont introduced it in mid 1960s. In the beginning simple bicomponent fibers were produced with either a sheath-core or a side-by-side morphology. With the advent of new technologies in recent years, a wide variety of bicomponent fibers with complex morphology is being produced to address a wider range of performance features. One major reason behind the increasing research interest in this area is that bicomponent fibers with finely tailored properties can now be produced to fit specific design needs².

One interesting structure for novel bicomponent fiber is known as island-in-the-sea (IS). In the cross-section, areas of one polymer (islands) are in the matrix of another polymer (sea). Figure 1a shows a schematic of the spinning process of IS bicomponent fibers from two separate polymer melts³. The schematic of the IS fiber is shown in Figure 1b. The number of islands in the earlier version of IS fibers were limited, however with innova-

tions in new spinning technologies, IS fibers are now being produced with thousands of islands in a few microns diameter cross-section². In the present study, we focus on individual bicomponent fibers with IS cross-sectional geometry, where the islands are Nylon and the sea (or, the matrix) is PET. Addition of PET to nylon results in increase in modulus of the fiber, reduction in moisture regain, improvement in texturing ability, and a lustrous appearance¹. Besides using them as composite bicomponent fibers, the IS fibers are also an attractive way to produce micro and nano fibers, after removing the sea component by dissolving it in a solvent. The micro and nano fibers produced by this method are used in synthetic leather, ultra-high filtration media, artificial arteries, among other specialty applications.

Tensile strength and elastic modulus of a fiber are important parameters for most applications. However, our knowledge about the complete deformation behavior of these novel bicomponent fibers is still limited. In this work, we studied the mechanical response of the composite Nylon-PET IS bicomponent fibers. We also address the evolution of the mechanical properties of these composite fibers as a continuous function of strain.

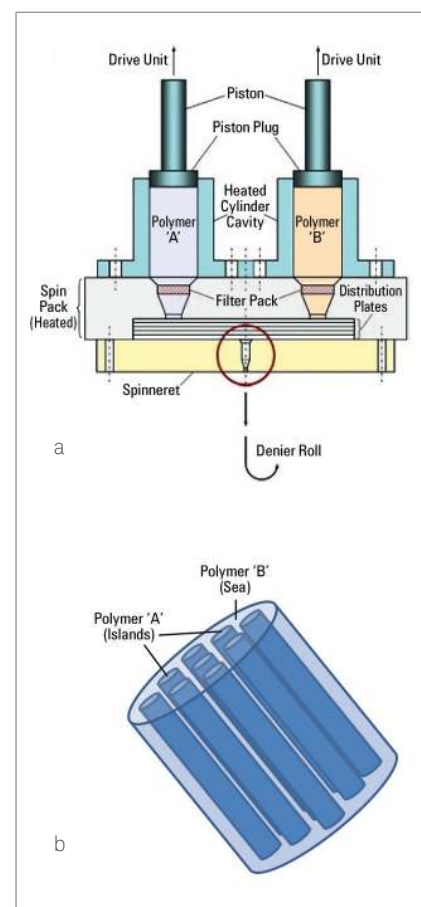


Figure 1. (a) Schematic of spinning two polymer melts together to form bicomponent fibers. (b) Schematic of an island-in-the-sea (IS) fiber, where the cross-section consists of islands of one polymer embedded in a matrix (or, sea) of another polymer.

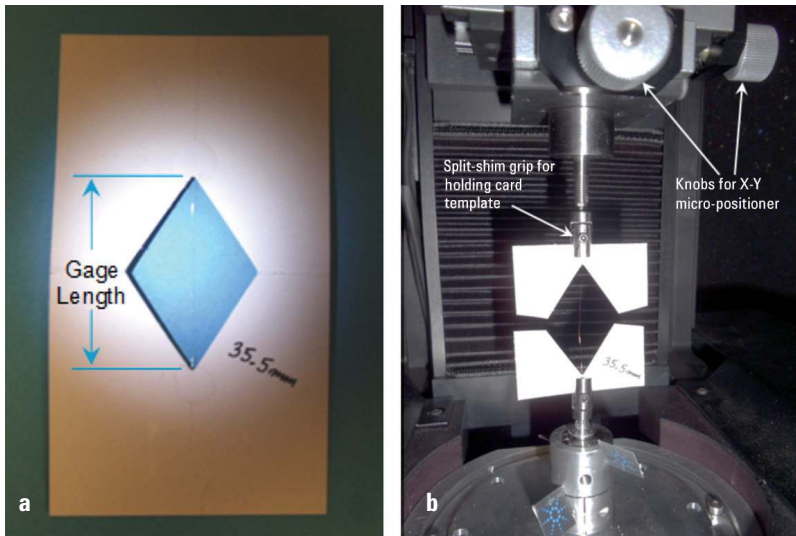


Figure 2. (a) Individual bicomponent IS fiber mounted on a card template. (b) The card template with a single fiber specimen is mounted on the Keysight T150. The sides of the template are clipped to release the fiber for the tensile test.

Continuous Dynamic Analysis

It has been recognized that the molecular structure of a polymer evolves with their deformation. For example, in semi-crystalline polymers, the amorphous polymer chains tend to align themselves along the fiber axis as it is stretched. This alignment of polymer chains results in significant changes in the polymer structure (and in some cases increase in crystallinity), which in turn cause significant changes to the elastic properties of the fiber. However, most of our understanding of the elastic mechanical properties, such as storage and loss moduli, of a polymer fiber is limited to the very small strain (mostly, up to 1%) deformation regime.

To address this lack of understanding of the evolution of elastic properties of polymer fibers during tensile deformation, a novel test methodology called continuous dynamic analysis (CDA) has been introduced, using the nanomechanical actuating transducer technology in the Keysight Technologies T150 UTM. In addition to the quasi-static uniaxial loading, the CDA module applies a high frequency harmonic force to measure the dynamic storage and loss modulus of the fiber at each and every point of strain during a uniaxial tensile test. It is important to mention here that the harmonic force is much smaller compared to the quasi-static force on the fiber specimen. Hence, the CDA measurements do not affect the quasi-static stress-strain response.

Because of the viscoelastic nature of polymer fibers, the harmonic displacement (z_0) lags behind the harmonic force (F_0) by a phase angle ϕ . Hence, the dynamic storage modulus of the specimen is related to the harmonic force and displacement amplitudes and the phase angle by [4]:

$$E' = \frac{l}{A} \left(\frac{F_0}{z_0} \cos\phi \right) \quad (1)$$

where, l and A are the instantaneous gage length and fiber cross-sectional area, respectively.

Similarly, the dynamic loss modulus can be calculated from:

$$E'' = \frac{l}{A} \left(\frac{F_0}{z_0} \sin\phi \right) \quad (2)$$

And, the loss factor is represented by:

$$\tan\delta = \frac{E''}{E'} \quad (3)$$

As mentioned above, the CDA enables the measurement of these dynamic mechanical properties of the fiber at each and every point over the complete range of strain during one single uniaxial tensile test. The CDA method has been successfully employed to characterize the evolution of mechanical properties of synthetic polymer, metal and ceramic micro-fibers and spider silk⁴⁻⁶.

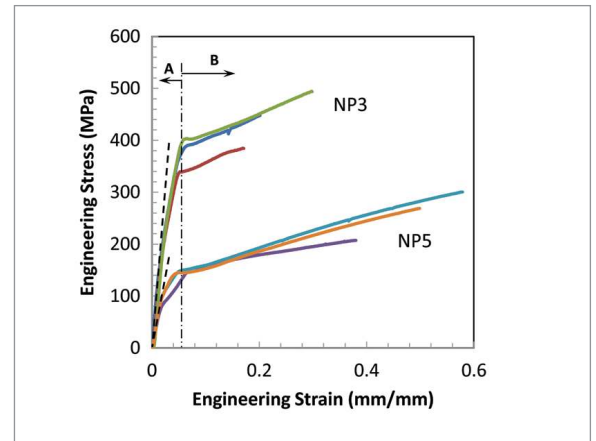


Figure 3. Engineering stress-strain curves for nylon-PET bicomponent IS fibers, with 85% nylon and 15% PET. The molecular weight of PET in NP5 fibers is higher than that in NP3 fibers. The dashed lines at the initial part of the curve indicate linear elastic deformation. Regime A is prior to formation of any neck in the fiber, whereas regime B indicate strain hardening in the necked region.

Experimental Details

Nylon-PET bicomponent IS fibers, with nylon islands in a PET matrix, were obtained from Hills Inc. (Melbourne, FL). The individual composite fibers contain 85% nylon and 15% PET. The diameter of the bicomponent IS fibers was about 10 μm . During this study, we characterized two different types of nylon-PET bicomponent fibers, namely NP3 and NP5, where the NP5 contains higher molecular weight PET compared to the NP3. Individual nylon-PET bicomponent IS fibers of each type were carefully mounted on thick paper-based card templates, as shown in Figure 2a. The gage section of each fiber specimen is measured using a caliper, and the diameter of the fibers was measured using an optical microscope. The card template was then mounted on the Keysight T150 nanomechanical UTM (Figure 2b) using the template-grips and the sides of the template were clipped to release the fiber for testing. The micro-positioner is used to ensure proper alignment of the fibers before testing. Three fiber samples each of NP3 and NP5 variety were characterized.

The NanoSuite (Keysight Technologies) test method named "UTM T150 Standard Toecomp CDA" was used for tensile testing of the individual bicomponent IS fibers, along with continuous dynamic analysis. All the tests were performed with a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$. The continuous dynamic analysis during each test was performed using a harmonic force of 4.5 mN at a frequency of 20 Hz.

Results and Discussion

The quasi-static engineering stress-strain curves for the individual NP3 and NP5 fibers are shown in Figure 3. Initially the fibers exhibit a linear elastic regime due to homogeneous elastic deformation (up to about 1% strain), before the engineering stress increases nonlinearly up to the yield point (regime A, as marked in Figure 3). The Young's modulus, measured from the slope of the initial linear elastic region (dashed lines in Figure 3), is 12 ± 1 GPa for NP3 fibers and 6 ± 1 GPa for the NP5 fibers. As noted above, the NP5 fibers contain PET of higher molecular weight compared to the NP3 fibers. As PET has high stiffness due to the presence of benzene ring in its molecule⁷, it is supposed to be the load bearing component in the IS fibers. Hence, the difference in the modulus for the NP3 and NP5 fibers can be attributed to the difference in PET molecular weight. The PET in the NP5 fibers contain more amorphous regions due to the high molecular weight, resulting in lower stiffness (or, modulus) of the fiber⁸. The difference in molecular weight also explains the lower yield points observed for the NP5 fibers.

After the yield (regime B, as marked in Figure 3), the small drop in engineering stress most probably related to formation of a neck that propagates across the gage section⁸. The necked region then strain hardens as seen from the increase in engineering stress until the fiber fractures. During this time, the amorphous molecules in the fiber also tend to align themselves along the fiber axis, resulting in the increase in stress^{7,9}. The fracture strain of low molecular weight NP3 fibers was significantly lower than higher molecular weight NP5 fibers. This high ductility of NP5 fibers can be attributed to longer amorphous PET molecules, causing larger number of entanglements that increases the stability of the neck up to a greater strain⁸. This engineering stress-strain response is in good agreement with previously reported results for PET fibers, confirming that the PET has the more dominant contribution towards the mechanical properties of the nylon-PET bicomponent IS fibers.

The CDA measurements on the NP3 fibers are shown in Figure 4. Figures 4a and 4b show the evolution of the storage modu-

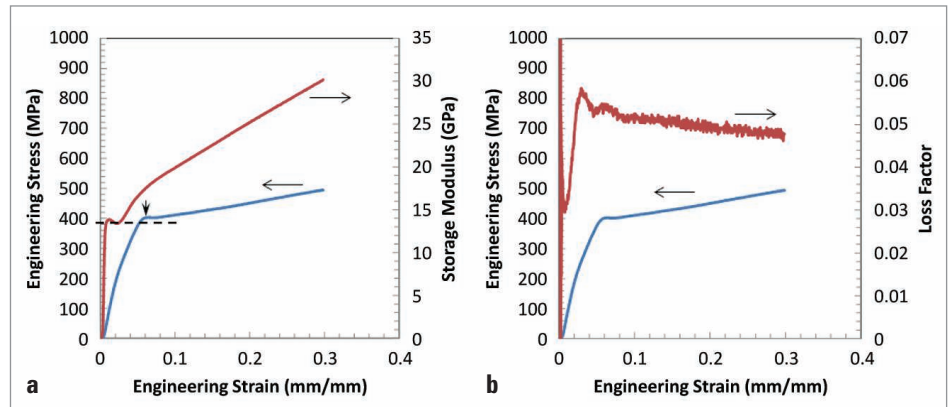


Figure 4. The evolution of dynamic storage modulus (a) and loss factor (b) in a NP3 fiber as a continuous function of strain.

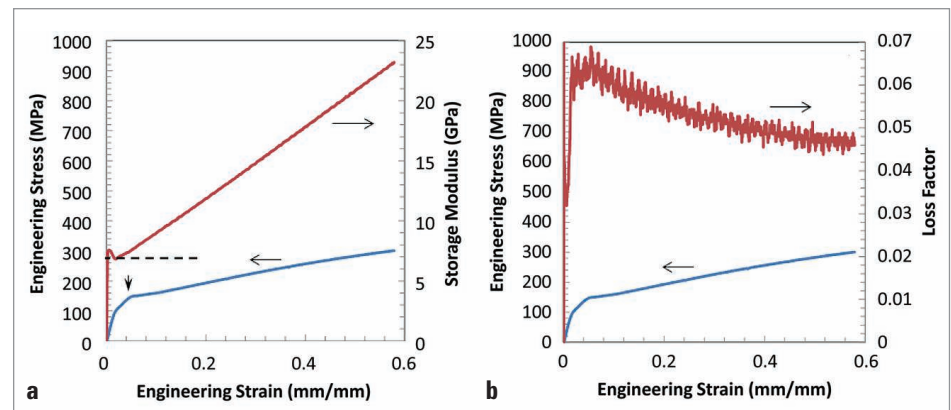


Figure 5. The evolution of dynamic storage modulus (a) and loss factor (b) in a NP5 fiber as a continuous function of strain.

lus and the loss factor, respectively, as a continuous function of strain. It is important to note here that the initial value of the dynamic storage modulus (dashed horizontal line in Figure 4a) matches quite well with the Young's modulus measured from the quasi-static stress-strain curves. This confirms the validity of our continuous dynamic analysis results. Figure 4a also shows that the storage modulus starts to increase at a slightly lower strain than the yield point (vertical arrow). Such a behavior implies that some amorphous PET chains start to align prior to macroscopic yield. Once the neck forms at the yield point the alignment procedure becomes more coherent and results in a linear increase in storage modulus until failure⁶. The CDA reveals that the storage modulus for the NP3 fiber increases significantly from about 12 GPa to 30 GPa during the deformation process. On the other hand, as the PET chains starts to align along the fi-

ber axis, the deformation process becomes less viscoelastic which shows excellent agreement with the decrease in loss factor with increasing strain (Figure 4b).

Figures 5a and 5b show the CDA results from the measurement on a NP5 nylon-PET bicomponent IS fiber. Similar to the NP3 fiber, the changes in dynamic storage modulus and loss factor can be correlated with molecular orientation in the PET component. Due to higher molecular weight, the initial storage modulus (dashed horizontal line) for the NP5 fiber is almost half of the modulus of NP3. However, at failure strain, the dynamic storage modulus for the two fibers becomes almost similar, even if the NP5 fiber withstands a larger strain before failure. As NP5 contains more amorphous molecules, it requires larger strain to reach similar molecular alignment as the NP3. Such behavior strongly suggests that the CDA is a potential tool

to relate molecular alignment in polymer fibers to their mechanical properties. Note that the contribution from nylon is not considered here as it has lower stiffness than PET and both the NP3 and NP5 fibers contained same amount of nylon. This comment notwithstanding, more experiments are needed to measure the influence of the nylon molecules on the mechanical properties, especially at high strains.

Summary and Conclusions

Mechanical properties of individual nylon-PET bicomponent IS fibers, with two different PET molecular weights, were characterized using the Keysight T150 UTM. The quasi-static stress-strain curves show significant difference between the two different types of fibers. Higher molecular weight NP5 fibers contain more amorphous PET molecules resulting in lower modulus and yield strength. However, the greater amount of amorphous molecules caused more entanglements resulting in larger failure strain of the higher molecular weight fibers. The CDA measurements show significant increase in dynamic storage modulus, and corresponding decrease in loss factor, of the fiber with increasing strain. The CDA not only provides fundamental information about the deformation process, but also becomes an important tool to relate molecular structure of polymer fibers with their mechanical properties.

References

1. Hegde, R.R., A. Dahiya, and M.G. Kamath, <http://web.utk.edu/~mse/Textiles/Bicomponent%20fibers.htm>.
2. Dugan, J.S., http://www.textileworld.com/Articles/2010/July/July_August_issue/Features/Specialty_Markets_Bicomponent_Fibers.html.
3. Chae, H.G., et al., *Carbon nanotube reinforced small diameter polyacrylonitrile based carbon*

fiber. Composites Science and Technology, 2009. 69(3-4): p. 406-413.

4. Hay, J., *Quasi-static and Dynamic Properties of Technical Fibers*. Keysight Technologies Application Note, 2014.
5. Blackledge, T.A., J.E. Swindeman, and C.Y. Hayashi, *Quasistatic and continuous dynamic characterization of the mechanical properties of silk from the cobweb of the black widow spider *Latrodectus hesperus**. The Journal of Experimental Biology, 2005. 208: p. 1937-1949.
6. Basu, S., *Tensile deformation of fibers used in textile industry*. Keysight Technologies Application Note, 2014.
7. Lechat, C., et al., *Mechanical behaviour of polyethylene terephthalate and polyethylene naphthalate fibres under cyclic loading*. Journal of Materials Science, 2006. 41(6): p. 1745.
8. Pawlak, A., et al., *Mechanical Properties of Poly(ethylene terephthalate) Modified with Functionalized Polymers*. Journal of Applied Polymer Science, 1999. 73: p. 203-219.
9. Basu, S., *Statistical Variation and Strain-Rate Sensitivity of the Mechanical Properties of Individual PET Fibers*. Keysight Technologies Application Note, 2014.

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