Keysight Technologies
Mechanical Properties Measurement on Individual Composite Micro-fibers
Application Brief

Bicomponent micro-fibers, consisting of nylon 6 nano-islands in a PET sea, were characterized for their mechanical properties under tensile loading. It is a challenging problem to accurately measure the mechanical properties of individual micro-fibers using conventional mechanical testing instruments because of the small forces required to deform these materials. Deciphering the fundamental properties of these small-diameter fibers, especially with a composite architecture, by testing multi-fiber yarns is also complicated due to different types of fiber-fiber interactions.

In the present study, we used the Keysight Technologies, Inc. T150 tensile tester, which is capable of measuring changes in small amounts of force over a wide range of deformation strain. Due to its high resolution force transducer (also known as nano-mechanical actuating transducer, or NMAT) and the high-resolution extension capability, the Keysight T150 can measure accurate quasi-static stress-strain behavior of micro- and nano-fibers. Many of these studies have been documented in published literature. Besides the quasi-static deformation, the unique design of the NMAT also allows measurement of dynamic mechanical properties of the fiber specimen as a continuous function of strain. For most polymeric fibers, molecular chains undergo significant alignment and/or crystallization processes, which are known to influence their mechanical properties. However, the continuous dynamic analysis (CDA) using the T150, for the first time, can quantify these changes in dynamic storage and loss modulus, and characterize the evolution of mechanical properties with increasing strain.

A schematic of the composite micro-fibers, with the island-in-sea (I/S) architecture, is shown in Figure 1. Three different sets of nylon-PET IS fibers – NP3, NP4 and NP5 – were characterized during this study. The difference between the NP3 and NP5 fibers was the molecular weight of the PET sea (matrix), NP3 having higher molecular weight PET compared to NP5. On the other hand, the NP4 and NP5 fibers differed in the amount of nylon 6 content – NP4 containing greater amount of nylon islands compared to NP5. The tensile tests were carried out at a constant strain-rate of 1 x 10^{-3}/s. The CDA measurements were performed at a constant frequency of 20 Hz for the complete deformation range.

Figure 2 shows comparative true stress – true strain plots for the NP3 and NP5 fibers which differ in the molecular weight of PET (sea). Among the two components in these fibers, PET has higher stiffness compared to nylon 6. Hence, it is reasonable to infer that resistance to initial deformation mostly

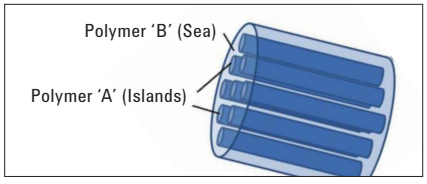

Figure 1. Schematic of a composite micro-fiber with island-in-sea (IS) architecture. Here the polymer 'A' is nylon 6, and the polymer 'B' is PET.

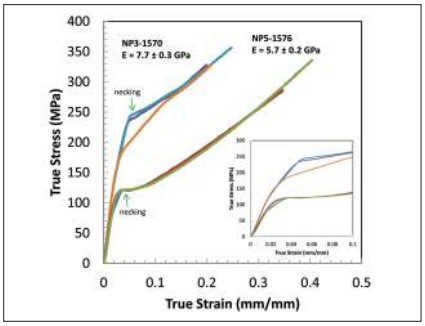

Figure 2. Tensile stress-strain behavior for NP3 and NP5 fibers. The Young's modulus, E, was measured from the slope of very initial linear elastic region. The inset magnifies the small-strain deformation of fibers.

affected by the PET matrix. The NP3 fibers exhibit a higher Young's modulus calculated from the initial slope of the stress-strain curves. As evident from the magnified low-strain region, shown in the inset, the longer PET molecules in NP3 fibers undergo more unfolding resulting in a slight decrease in the stress-strain slope after the initial linear elastic regime. This phenomenon is not observed for the NP5 fibers that contain lower molecular weight PET. The higher molecular weight PET in NP3 also withstands necking (arrows in Fig. 1) until a higher stress. Beyond the necking, the amorphous regions in the PET matrix, along with the finer fibrils of nylon 6 islands, start to align more along the fiber axis. This process causes the linear increase in the stress-strain behavior. Due to further interlocking between longer PET chains, the failure strain in the NP3 fibers is smaller compared to that of the NP5 fibers.

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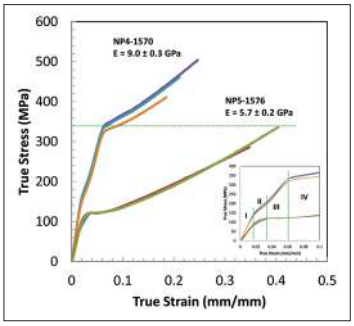

Figure 3. Tensile stress-strain behavior for NP4 and NP5 fibers. The Young's modulus, E, was measured from the slope of very initial linear elastic region. The inset magnifies the small-strain deformation for both types of fibers.

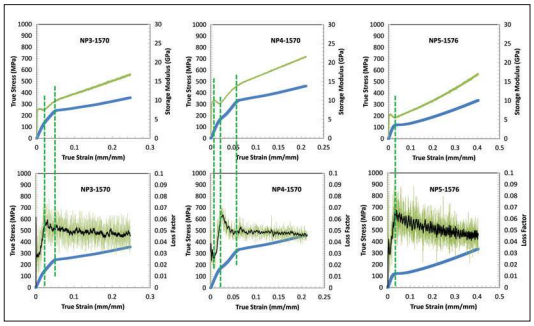

Figure 4. Continuous dynamic analysis results for NP3, NP4 and NP5 fibers. The top and bottom rows, plot the evolution of storage modulus and loss factor, respectively, along with the simultaneously-measured stress-strain curves.

The comparative true stress – true strain curves for the NP4 and NP5 fibers are shown in Figure 3. NP4 has a larger number of nylon 6 islands compared to NP5. The measured Young's modulus for the NP4 fibers is significantly higher than that for NP5, and approaches that of pure PET fibers (~ 10 GPa). This is a little surprising given that the NP4 has higher nylon 6 content; however, the higher modulus suggests that the PET molecules are probably more aligned along the fiber axis. Although it cannot be ruled out at this moment, another reason behind the higher modulus may be that the higher nylon content in these fibers causes the islands to be discontinuous along the length of the fiber, and the PET sea is the dominant structural component during the initial deformation of these fibers. This hypothesis can only be solved with a three-dimensional probing of the specimen. The different regimes of deformation in the NP4 fibers are clearly evident in the magnified plot showed in the inset. Regimes I, II and III are very similar to the deformation of pure PET fibers. Beyond necking (arrows in Fig. 3), the increasing molecular alignment causes a linear increase in the stress as a function of strain. There is a slight difference in slope between NP4 and NP5 fibers during this region, which corresponds to the difference in the nylon content and may also to the interfacial characteristics between the nylon 6 and PET. The higher nylon 6 content in the NP4 specimen also resulted in higher failure stresses compared to NP3 and NP5 fibers (dotted horizontal line in Fig. 3).

The different regimes of deformation for these bicomponent I/S micro-fibers, as observed in the stress-strain curves, can be linked to the evolution of molecular chain structure and their arrangement during the deformation process. Therefore, it is extremely important to ask the question – "how these changes in structure affects the viscoelastic properties of the fibers during their deformation?". Such information will be instrumental in designing any load bearing component using these bicomponent micro-fibers. Conventional tensile tests and dynamic mechanical analysis only measures the elastic properties at a very small applied strain. However, with the unique transducer design for the Keysight T150, an extremely small harmonic force oscillation was applied during the complete tensile deformation. The small harmonic oscillations always stay within the linear viscoelastic regime, making it possible to measure dynamic storage and loss modulus, even when the quasi-static deformation is nonlinear.

Figure 4 shows the CDA results for the NP3, NP4 and NP5 fibers. The quantitative evolution of dynamic storage modulus (top row) and the dynamic loss factor (bottom row) with the stress-strain behavior are clearly evident for all the fibers. The dotted vertical lines delineate how the viscoelastic properties evolve during the changes observed in the stress-strain curves in Figs. 2 and 3. Both the storage modulus and loss factor stays almost stable during the initial linear elastic regime (at extremely small strains). After that, during the unfolding of the polymer chains, the loss factor increases. Once the chain alignment increases with increasing strain the dynamic storage modulus increases significantly, and almost doubles before the fiber breaks. Due to the same process, the loss factor keeps decreasing. These changes agree with the expected behavior, and now for the first time we have evidence and a quantitative understanding of the evolution of viscoelastic properties of these bicomponent fibers.

In summary, the tensile behavior of bicomponent micro-fibers has been successfully measured. The CDA quantitatively determined the evolution of elastic properties as a continuous function of strain, which can be correlated to the structural changes of the polymer chains within the fiber during deformation. These experiments prove the versatility of the Keysight T150 in probing molecular behavior of polymeric materials under deformation.

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