doi:10.1017/S1431927618004713
Microsc. Microanal. 24 (Suppl 1), 2018

A Unique Approach for Preparing Cross-Sectional EM Study of Textile Fibers

Wen-An Chiou[1], Sz-Chian Liou[1], Jiancun Rao[1] and Timothy K. Maugel[2]

1. AIM Lab, Maryland NanoCenter, University of Maryland, College Park, MD 20742, USA
2. Laboratory for Biological Ultrastructure, Univ. of Maryland, College Park, MD 20742, USA

Textiles are made up of fibers that are arranged in different ways to create the desired strength, durability, appearance and texture. They are heavily treated with chemicals before the new fibers are spun. The textile surface modification improved the flexural behavior of the materials. Examining fibers of textile under the microscope has been an important practice of the textile industry over the years. Among the microscopy techniques, optical and SEM have been the most popularly used to date. Despite the study of cross-sectional samples provides valuable information of the materials, publication of textile fibers especially their cross-sectional TEM/SEM has been scarce. This study presents the results of a cross-sectional EM study of textile fibers including discussion of different sample preparation techniques.

Due to the delicate nature of textile fibers, conventional techniques using grinding and polishing followed by ion milling for preparing cross-sectional TEM/SEM specimen is not suitable. Borrowing the sample preparation method of biological materials, a few textile fibers were picked up from a bundle of fibers and embedded in low viscosity epoxy resin. Curing was done at 70 ^{o}C for overnight. The embedded block was trimmed and TEM thin sections were cut perpendicular to the fiber's longitudinal axis with an ultramicrotome (Reichert Ultracut E) using a diamond knife. Section thickness ranged between 70 to 90 nm. Thin sections were collected on 200 mesh hexagonal Cu grids and coated with a very thin (~10 nm) C film for stabilization. Ultrathin specimens were examined in JEM 2100 LaB6 TEM (at 100 and 200 kV).

A focused ion beam (FIB) technique was also applied for preparing cross-sectional view of the same sample for comparison. To minimize potential ion beam damage, cryo-FIB was used. A few textile fibers were mounted on special Au dishes (~3 mm in diameter) using cryo-compatible glue (OCT compound, Tissue-Tek®). The mounted samples were submerged into a LN_2 bath using Leica EM VCM device. The sample dishes were then mounted to cryo-stage within the LN_2 bath and subsequently transferred to a vacuum chamber (Leica EM ACE 600) using Leica EM ACE 600 cryo-shuttle. Cryo-fracturing was performed using a special device inside the vacuum chamber at near LN_2 temperature. To prevent charging, the fractured surface was cryo-etched at -100 °C for 3 min., and then coated with 10 nm thick AuPd layer at -150 °C before transferring to a Tescan FIB/SEM (GAIA) with the cryo-shuttle. The sample was kept at a temperature between -150 °C to -160 ^{o}C at all times while carrying out SEM observation and FIB milling of the sample.

As shown in all figures, the textile fibers were composed mainly of bundles of two fibers. The surface of these fibers was coated and/or treated with a very thin layer of fiber-like materials that might contain nanotubes (Fig. 1). The amounts of coatings and pre-treatment of fibers were probably different on two fibers as reflected on their surface morphology, which in turn also affects the surface property, e.g., degree of wettability. TEM images of cross-sectional specimen prepared by ultramicrotoming depict detail of the internal microstructure of the fiber (Fig. 2). Among those two fibers (in a bundle), the larger one appears to contain higher electron density carbonaceous fiber-like materials whereas the small fiber interior is less dense but contains a few relative large, dense and very dark materials across the fiber without specific pattern. At higher magnification, a very thin film (with small needle-like materials) surrounding the fiber can be seen (arrows in Figs 2a and 2c). This thin layer corresponds to the thin coating that we have observed in SEM. Cross-sectional SEM image was impossible due to the severe ion beam and fiber interaction despite milling at cryo-temperature (Fig. 3). The milling rate of those two

fibers was quite different. The larger fiber had higher milling rate and tended to have more charging phenomena while imaging as compared with the small fiber. This may reflect differences in the physical properties between those fibers; however, determining whether these are due to external coating and treatment and/or the difference between internal structures requires further research.

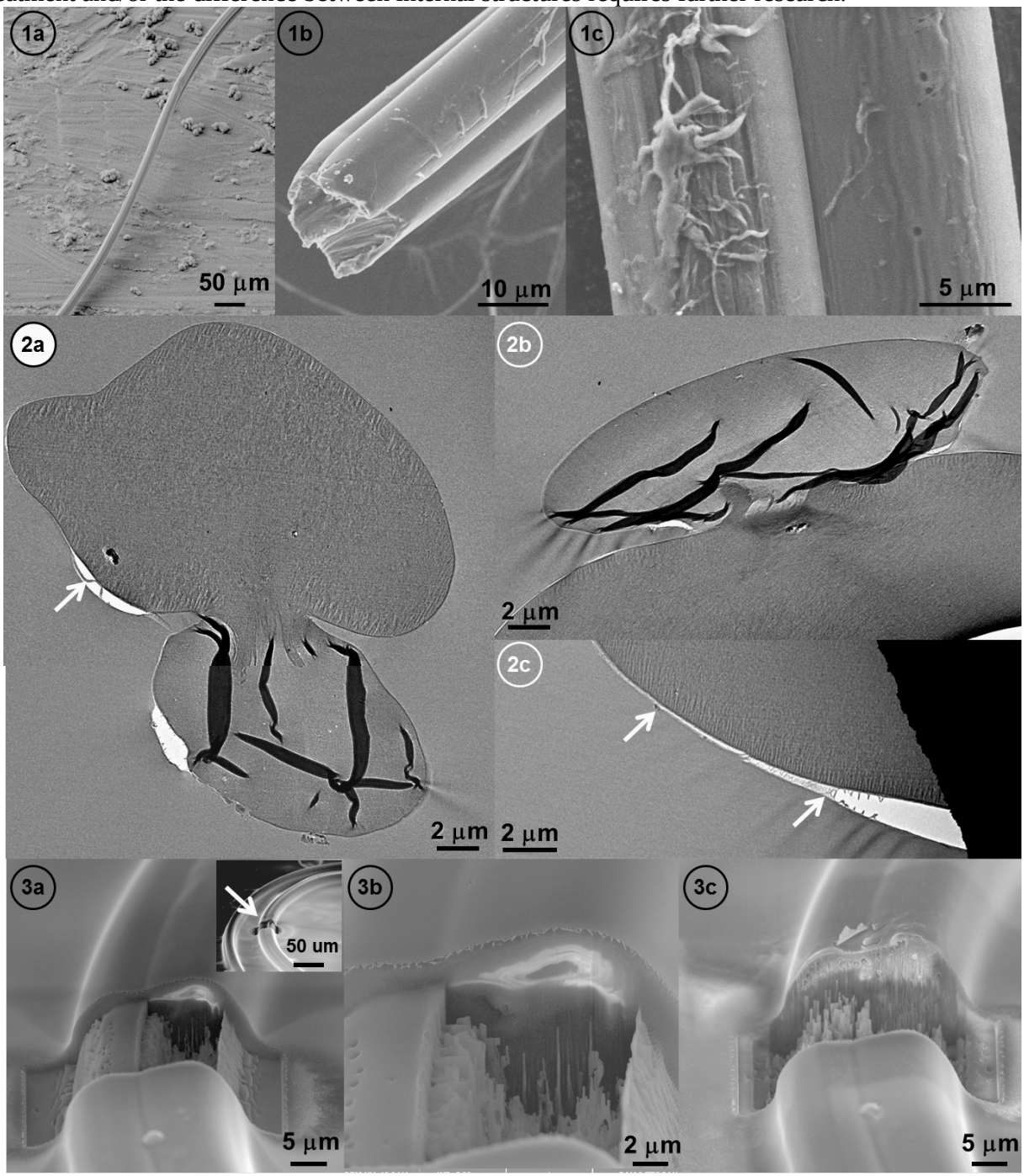

Fig. 1. SEM micrographs show textile fibers at different magnifications. Note the surface morphology.

Fig. 2. Cross-sectional TEM images of ultra-microtomed fibers reveal the detail internal microstructure. Note the distinguished difference between large and small fibers. Arrows: thin film on surface.

Fig. 3. Cross-sectional Cryo-SEM images of textile fibers reveal differential ion milling rate between large and small fibers. Fibers were damaged easily and quickly by Ga ions despite at LN2 temp.

https://doi.org/10.1017/S1431927618004713 Published online by Cambridge University Press