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Rapid Preparation of a Polymer Fiber and a Free-Standing Polymer Film for Cross-Sectional Microtomy

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Introduction

Polymer films and fibers can readily be imaged in a "top-view" mode by depositing the sample directly on a transmission electron microscopy (TEM) support grid. Sample preparation of thin polymer films and fibers for cross-sectional views in the electron microscope, however, is a major challenge. Owing to their small dimensions, the films or fibers cannot be mounted alone, because they will not remain immobilized in the microtome, even at low temperatures. Brittle polymers complicate sample preparation even further because they tend to break due to the mechanical stresses exerted on them by the microtome sample holder and the knife. Such sections often jump off the knife-edge or the sample film, thus preventing a systematic collection of thin sections. Similar arguments apply for cross-sectional atomic force microscopy (AFM).

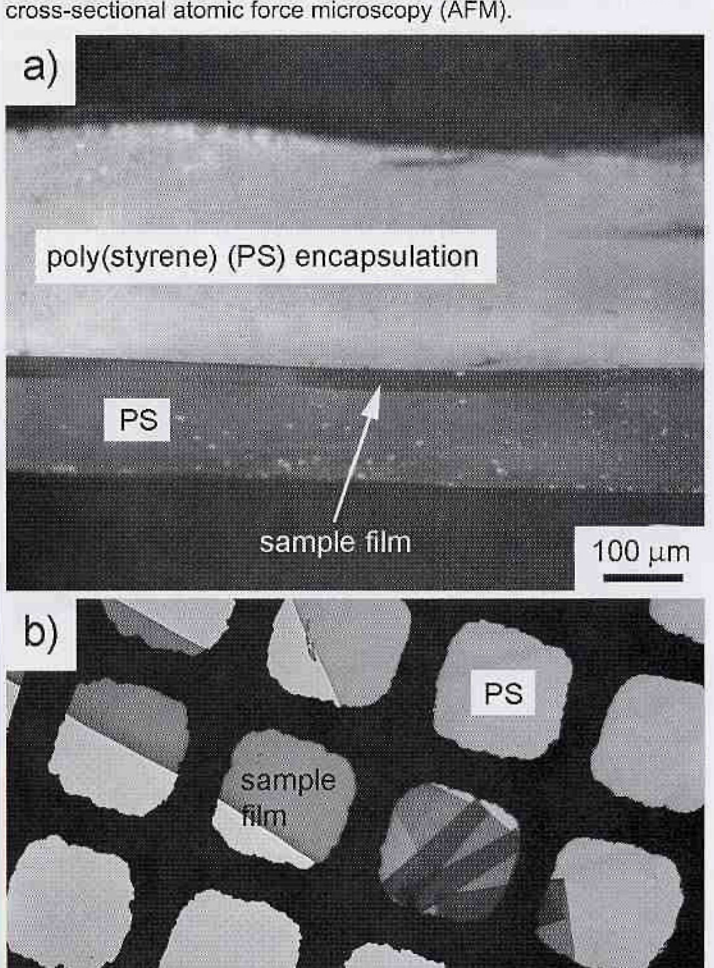

Fig. 1: (a) Optical micrograph of an encapsulated sample. (b) Low magnification TEM image of a cross-section.

We recently needed to obtain cross-sections of ~ 75 μm thick, brittle poly(imide) (PI) films with glass transition temperatures (T_g's) above 400°C for a TEM investigation.[1] This obviously requires embedding to immobilize and protect the films in the microtome and to enable thin sectioning of sensitive samples. For various reasons, we did not want to use epoxies for embedding. In the following, we describe how we obtained thin sections and block faces suitable for TEM and AFM imaging, respectively.

Materials and Methods

Sample preparation. We molded 300 to 500 μm thick poly(styrene) (PS, M_w ~ 250,000 g/mol, Aldrich) slabs; we then deposited a small piece of the sample film or fiber between two layers of the PS. These PS/sample/PS trilayer pellets were molded at 107°C and 1000 psi for about 5 minutes and allowed to cool to room temperature after removal from the hot-press; the whole procedure takes *ca.* 15 minutes. The encapsulated specimens were microtomed with a diamond knife at room temperature. The sections were floated on water and deposited on TEM support grids; the block face was used for AFM imaging.

Microscopy. Optical micrographs were obtained with an Olympus BH2 with a Polaroid camera. TEM experiments were made on a JEOL 2010F electron microscope operated at 200 kV and equipped with a Princeton Gamma Tech (PGT) X-ray energy dispersive spectrometer. X-ray spectra were acquired using the "Qual" mode of the IMIX software from PGT; data analysis and background subtraction was performed with the IMIX software. AFM images of the block face of a compression-molded and microtomed pellet were acquired in non-contact mode under ambient conditions using a DI Dimension 3000 SPM. After microtomy, the pellet was mounted directly onto the sample stage using adhesive tape. No further sample preparations were necessary.

Results and Discussion

Figure 1a is an optical micrograph of the block face of an encapsulated poly(imide) film after microtomy. It shows that the sample film in the PS still has sharp edges, indicating that no mixing between PS and sample occurs during compression molding. Molding at higher temperatures or higher pressures leads to a partial mixing evident from the disappearance of the sample edges and a slightly yellow color (the samples are yellow) of the regions adjacent to the actual sample location. The microtome binoculars, similar to the optical microscope, are sufficient to distinguish the sample from the PS during cutting; this allows for the observation of the effect of cutting conditions on sample preparation. A cutting speed of 0.2 mm/s gives the best sections. At higher cutting speeds, the films occasionally jump off the edge of the diamond knife and cannot be collected in an orderly fashion.

Figure 1b is a low magnification TEM image showing a small stripe (the presumed sample) and a large area (the presumed PS). Figure 1b demonstrates that the sample and the PS can be distinguished even after cutting and section deposition on the TEM support grid, because the sample cross-sectional thickness is much smaller than the PS. Occasionally, the sample and the PS separate during floating on water. If this happens, it usually is clear which one is the sample because it is ~ 75 μm across as compared to ~ 300 μm of the PS; the separation of the sample from the PS therefore enables picking up only the sample, which in turn facilitates the identification of the sample in the microscope and avoids the investigation of the PS.

In the current case, both the PS and the sample are amorphous and therefore electron diffraction[2] is not suitable for sample

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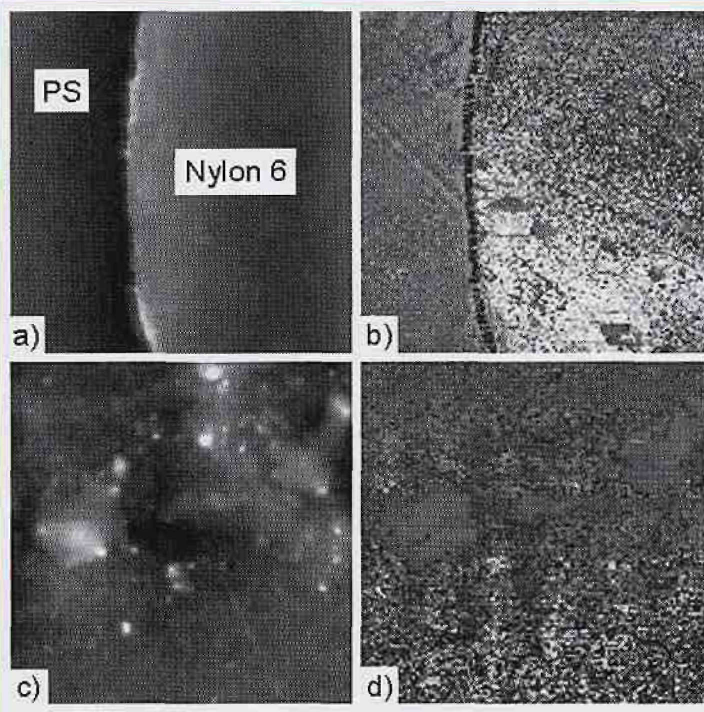

Fig. 2: AFM images of 2 areas of a nylon 6 fiber cross-section. AFM height (a) and phase (b) contrast images of a 22.7 × 22.7 μm area of the interface between the fiber and the embedding material. Height (c) and phase (d) contrast images of a 10 × 10 μm area of the interior of the same nylon 6 fiber.

identification in the electron microscope. We applied X-ray energy dispersive spectroscopy (XEDS) to corroborate that we properly identified the poly(imide) film, which contains C, F, N, O, and Al. The XEDS spectrum (not shown) of the stripe in Figure 1b shows distinct F *Kα*, N *Kα*, O *Kα*, and Al *Kα* lines, whereas the spectrum of the large piece on the right only shows an intense C *Kα* line. We thus conclude that our preliminary identification of the sample in the TEM is correct.

In contrast to the TEM, the AFM is only sensitive to surface features. Figure 2 shows that our sample preparation method is also capable of exposing the interior of small solids such as fibers to the AFM, which is important in light of the growing interest in micro- and nanoscale structures. Figures 2a and b are height and phase images, respectively, of the boundary between a nylon 6 fiber and the PS encapsulation. The fiber/PS interface appears as a vertical line through the center of the images. Figures 2c and d are height and phase images taken on the nylon 6 fiber. The images demonstrate that samples prepared using our technique can be characterized with AFM.

In conclusion, compression molding thin fibers and free-standing polymer films into a transparent thermoplastic that is easy to microtome is a viable procedure to obtain cross-sectional specimens. While the experimental conditions may have to be modified for each system, the procedure is robust, easy, inexpensive, and yields samples that are ready for microtomy in about 15 minutes. ■

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