Time-Resolved USAXS Studies of Polymers During Straining

A. Almendarez Camarillo¹, N. Stribeck¹, S. V. Roth², M. Dommach², R. Döhrmann²

¹Institute of Technical and Macromolecular Chemistry, University of Hamburg, Bundesstr. 45, 20146 Hamburg, Germany
²HASYLAB at DESY, Notkestr. 85, 22603 Hamburg, Germany

For the first time a commercial extensometer (Zwicki Z1.0/TH1S, Zwick GmbH, Ulm) was fully integrated into beamline BW4 and used for the straining of polymer samples (SBS multiblock copolymers, Polyester–PUR foams, and highly oriented PVDF). The straining stage was mounted at a sample-to-detector distance of 7542 mm. An exposure of 2 min was required in order to record USAXS patterns with a good S/N–ratio on the mar-CCD detector operated at 2048 × 2048 pixel resolution in multi-read modus.

Figure 1 shows the machine on the beamline (left) and the BW4-control booth with screens displaying the stress-strain curve, the sample on a video monitor screen with fiducial marks and a cross-hair cursor indicating the penetration point of the beam, and the current USAXS pattern on the control screen of the mar-CCD detector.

The extensometer is equipped with load cell and transducer. The corresponding signals are fed into two monitor channels of the BW4 data-acquisition system. Thus current stress and crosshead position are automatically stored with each USAXS image. The true elongation can be computed from the distance between fiducial marks monitored by a tiny video camera (Conrad Electronik, Kamera-Modul 2) that is able to display a full-screen focused image of a 7 mm × 10 mm spot on the sample. Video images of the sample can be grabbed for documentation and data evaluation.

For PVDF the maximum elongation is 18%. The material is undergoing homogeneous straining. After $\sigma_{max}$ is crossed in the stress–strain diagram, the nanostructure is changing considerably and a meridional streak is growing. Quantitative evaluation of the data is in progress.

Figure 1: Left photo: Zwicki extensometer integrated in beamline BW4 with elongated polymer sample. Right photo: Remote control screens. Left: Stress-strain curve and Zwicki control. Middle: Monitored polymer sample with fiducial marks (distance: 3 mm) and cross-hair indicating the penetration point of the beam. Right: USAXS pattern on the mar-CCD detector screen
The polyester–PUR foams exhibit the typical characteristics of a rubber. All sample show a strong central and diffuse scattering that cannot be evaluated because of the relatively short distance chosen. Only one of the materials shows a discrete scattering.

The materials with the most spectacular USAXS are the polystyrene-b-polybutadiene-b-polystyrene multiblock copolymers. As had been found out some years ago, for these materials the sample-to-detector distance chosen now is the optimum. We expect interesting information on the nanostructure evolution during straining and appreciate to have the actual stress-strain curves available for the first time.

**Acknowledgements.** We acknowledge HASYLAB, Hamburg, for provision of the synchrotron radiation facilities at beamline BW4 in the frame of project II-04-039. Support of this study concerning the extensometer, manpower, and a financial grant by the Deutsche Forschungsgemeinschaft (DFG STR501/4-1) is gratefully acknowledged.