Scanning Microbeam X-ray Scattering of Fibers Analyzed by One-dimensional Tomography

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Experimental Setup. Scanning-microbeam SAXS experiments are carried out at HASYLAB, Hamburg, beamline BW4. The incident primary beam (wavelength λ=0.13 nm) is focused by means of a stack of Be-lenses [1, 2] yielding a beam cross-section at the sample of 40 μm integral width and 39 μm height as measured by a knife edge. The strands are linearly scanned through the beam with a step size of 50 μm. The distance between sample and detector is 1910 mm. Each scattering pattern is exposed for 40 s using a 2D marccd 165 detector (mar research, Norderstedt, Germany). A low-noise machine background pattern is exposed for 3 min. The absorption of the primary beam is measured by monitoring the beam intensity before and after the sample. A simple check for fiber symmetry has been carried out by performing a second scan after rotating the fiber by 90$^{\mathrm{o}}$ about its axis and comparing the results.

Summary of Scientific Results. The investigation of structure gradients in polymer fibers or pipes by the X-ray microbeam scanning technique is put on its theoretical fundament. The inverse Abel transform desmears measured data in X-Ray scattering fiber computer-tomography (XSF-CT). [3] Fast, low noise algorithms from one-dimensional tomography are available. They are applicable to scan data in which the X-ray absorption, the small-angle X-ray scattering (SAXS) or the wide-angle X-ray diffraction (WAXD) is measured. The method is demonstrated by application to SAXS scan data from polymer fibers [4]. The resulting sequence of image-space SAXS patterns is reflecting the nanostructure variation along the fiber radius. We notice restricted visibility of scattering features within the series of patterns. The reason is violation of local fiber symmetry (LFS) in the irradiated volume elements (voxels). For its theoretical treatment a set of elementary topologies (tangential grain, radial grain) is introduced. Systematic aberrations (ultra-reconstruction, infra-reconstruction) generated by tomographic reconstruction of affected series are described. A concept for handling and utilization of these aberrations for nanostructure analysis is devised. Precursors of polymer microfibrillar-reinforced composites (MFC) containing poly(ether)-*block*-amide (PEBA) and poly(ethylene terephthalate) (PET) with varying cold-draw ratio are studied. We compare results from a direct analysis of the smeared measured patterns to results obtained after tomographic reconstruction and fathom the power of reconstruction methods. Ideas for advanced practical applications of the XSF-CT method are discussed.

Example. Figure 1 shows selected measured SAXS patterns from the microbeam scan (left) and the tomographically reconstructed patterns (right) – all patterns close to the center of the fiber. The reconstructed patterns show that the isotropic ring feature of the PEBA block copolymer phase is not present in the center of the fiber, whereas the horizontal streak is only changing its shape. The latter is related to the reinforcing PET microfibrils in the composite. The reconstructed pattern from the center collects all the structure with tangential grain [4] of the voxels along its path.

Conclusion. The method of fiber tomography permits to reconstruct the structure gradient along the radius of fibers, strands and pipes from one microbeam scan. It is by a factor of 100 faster

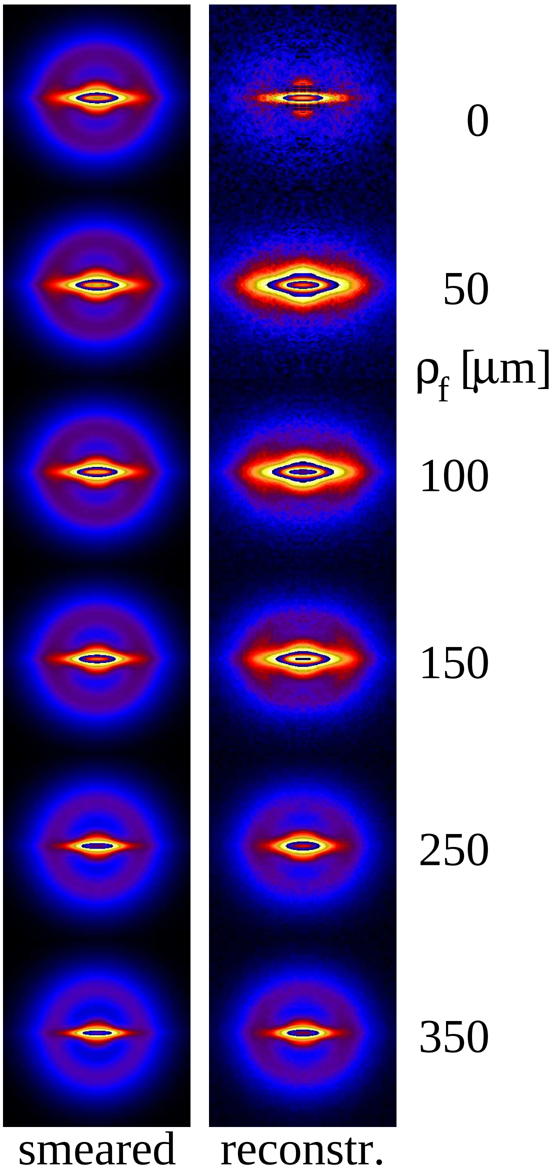

Figure 1: Cold-drawn ($\lambda_d \approx 3$) MFC in a scanning microbeam experiment. Measured scattering intensity $\{I\}(s_{12}, s_3, \rho_f)$ (left column) and reconstructed scattering $I(s_{12}, s_3, \rho_f)$ (right) for short distances ρ_f from the fiber axis. The patterns display the range -0.1 nm$^{-1} \leq s_{12}, s_3 \leq$0.1 nm^{-1} in uniform logarithmic scale

than the general tomography [5] – both what the beamtime and the reconstruction effort is concerned. We expect that it will become very useful for the study of industrial filaments, as soon as a microbeam with 1 μm diameter will be available at PETRA III.

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