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Report:

Many of the outstanding mechanical properties of carbon fibers are attributed to their internal structure, e.g., to a preferred orientation of the carbon layers and to possible skin-core effects. In order to obtain information about the structural arrangement of carbon layers and pores within the cross section of single carbon fibers, simultaneous X-ray microbeam small-angle scattering (SAXS) and wide-angle diffraction (WAXD) experiments were performed at the microfocus beamline (ID13) at the ESRF.

The monochromatic X-ray beam (wavelength λ=0.948 Å) was focussed by an ellipsoidal mirror and a glass capillary to a full width at half maximum (FWHM) of 3.6 μm at the specimen position. The exact beam profile was determined by scanning a circular pinhole (Pt-Ir, diameter 20 μm) through the beam. The experiments were performed with the capillary, the specimen, and a two-dimensional position-sensitive detector (MAR-CCD) positioned in one line. A sample-to-detector distance of 45 mm, together with the high pixel resolution of the detector (pixel size 64.5 x 64.5 μm^2) and the use of a small beam stop (0.3 mm diameter) allowed the simultaneous recording of the SAXS and WAXD signals in a range of scattering vectors from q = 0.1 $Å^{-1}$ – 5$Å^{-1}$ ($q = 4 \pi \sin(\theta) / \lambda$, where 2θ is the scattering angle).

Eight different carbon fibers (circular cross section, diameters between 7 μm and 12 μm) were examined in two different scattering geometries:

i) the usual fiber geometry with the fiber axis perpendicular to the X-ray beam. One-dimensional scans (step size 0.5 μm) were performed perpendicular to the fiber axis, acquiring a 2D scattering pattern for every scanning step.
ii) a transverse geometry with the fiber axis parallel to the X-ray beam. For this experiments, the fibers were embedded into resin and cut to a length of 3-5 μm. One-dimensional scans along two perpendicular directions of the fiber cross section were carried out. For one of the fibers, also two dimensional mesh scan was performed.
Fig. 1 shows a map of SAXS patterns from a 2D mesh-scan trough the cross section of a fiber, the X-ray beam being parallel to the fiber axis. The elliptical shape of the SAXS patterns is consistent with the presence of shelf like pores (where the longest pore dimension is known to point into axial fiber direction). The orientation of these elliptical SAXS patterns indicates that the longer side of the pore cross section points into radial fiber direction. Since shape and orientation of the pores must be connected with the arrangement of the carbon layers, a radial arrangement of carbon layers within the fiber cross section is directly obvious from Fig. 1.

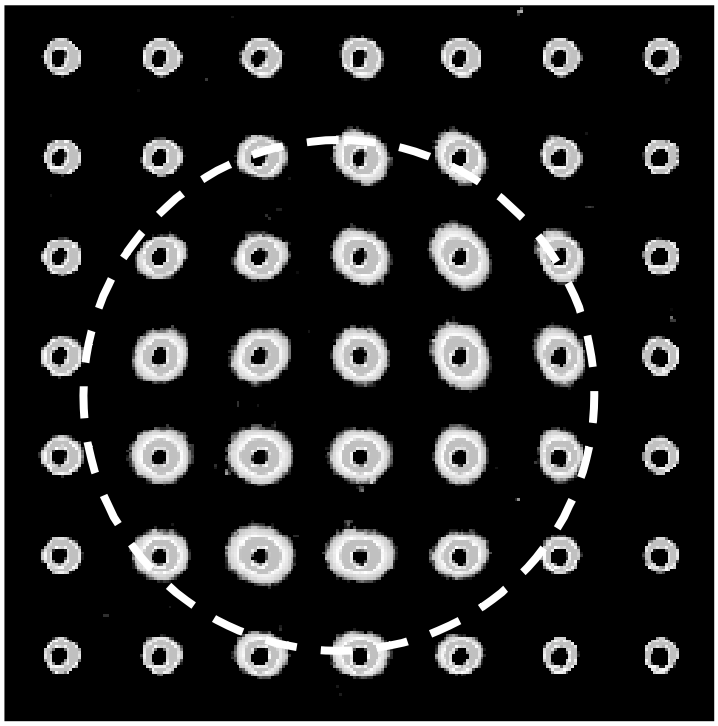

Figure 1: Map of SAXS patterns combined according to a mesh scan over the cross section of the MPP-based fiber *FT500*. The fiber axis was parallel to the direction of the X-ray beam and the step size was 2 μm. Every "pixel" of the mesh scan corresponds to a single scattering pattern with a wave vector transfer: $-0.3\ Å^{-1} < q < 0.3\ Å^{-1}$, horizontally and vertically. The dashed line indicates the border of the fiber. The signals outside the dashed line are mainly due to scattering contributions from the resin and from the guard pinhole.

The preliminary evaluation of the data indicates that in fibers based on polyacrylnitrile (PAN), the internal arrangement of carbon layers and pores is random within the fiber cross section, whereas for fibers based on a mesophase pitch (MPP), pronounced non random structures were found [1]. In particular, this information could also be derived from the standard fiber geometry experiments by taking advantage of the known X-ray beam profile [1]. Since fiber geometry experiments allow the investigation of single fibers in a non destructive way, this opens the possibility to perform in situ tension experiments on single carbon fibers in future experiments to correlate structural changes with the mechanical behavior.
[1] The internal structure of single carbon fibers investigated by simultaneous small- and wide-angle X-ray scattering, O. Paris, D. Loidl, H. Peterlik, M. Müller, H. Lichtenegger and P. Fratzl, to be published in the SAS99 proceedings, *J. Appl. Cryst.* (2000).