ESRF	**Experiment title:** Development of internal structure during in-situ stretching of single carbon fibres	**Experiment number**: ME-187
Beamline:	**Date of experiment**: from: 3.5.2001 to: 6.5.2001	**Date of report**: 25.8.2001
Shifts:	**Local contact(s)**: Dr. Christian RIEKEL	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

H. Peterlik and D. Loidl**

Institute of Materials Physics, University of Vienna, Boltzmanng, 5, A-1090 Vienna, Austria

*O. Paris**

Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, and Metal Physics Institute, University of Leoben, Jahnstr. 12, A-8700 Leoben, Austria

*M. Müller**

Institute for Experimental and Appl. Physics, University of Kiel, Leibnizstraße 19, D-24098 Kiel, Germany

Report:

Carbon fibres combine high tensile strength and high tensile modulus with low weight. The outstanding mechanical properties are even improved up to temperatures of 2000°C. Thus, carbon fibers are the ideal reinforcing material for light weight structures, e.g. in aerospace applications. The Young's modulus of the fibers is mainly determined by the preferred orientation of the hexagonal carbon layers with respect to the fiber axis, the strength is a function not only of flaws, but also of the axial as well as the cross-sectional orientation of the basic structural units /1-3/.

In a recent experiment (HS827) we were able to show that information on the internal structure of single carbon fibers can be obtained by scanning microbeam X-ray diffraction /4/, and we developed a model to distinguish between random and radial-folded cross-sectional fiber textures by evaluating the intensities of the 002-reflection and the 10-band across the fiber cross-section /5/. Quantitative results were obtained on PAN- and pitch-based carbon fibers /6/.

In the present experiment (ME-187) the structural changes of the single fibers were measured during in-situ tension tests using the stretching cell developed by ID13. The fibers were bathed in a beam of about 10 microns diameter, as provided by a collimator optics. X-ray diffraction images of the fibers were measured by a two-dimensional position-sensitive detector (MAR-CCD). 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 (002-reflection and 10-band) signals in a wide range of scattering vectors from q = 0.02 $Å^{-1}$ – 4$Å^{-1}$ ($q = 4 \pi \sin(\theta) / \lambda$, where 2θ is the scattering angle).

In the in-situ tension tests, the force was incremented in steps and then stopped to perform a scan to get information on the structure along the length of the fibers. In a first evaluation, the fibers appeared to be very homogeneous along their length and no significant differences in the orientation of the reflections could be found. The procedure of loading was repeated until the fiber fractured.

The background resulting from air scattering was subtracted from all images, and the azimuthal width as well as the radial position of the reflections were evaluated. As an example, Fig.1a shows the azimuthal width (half-width at half-maximum, hwhm) of the 002 reflection of the four tested PAN-fibers (as-received, and heat treatment temperatures of 1800^{o}C, 2100^{o}C and 2400^{o}C) in dependence on the load. Fig.1b shows the same for the four pitch-based fibers (different production procedures). The azimuthal width decreases with increasing stress, indicating an increase of preferred orientation of the carbon layers /1/, and additionally an obvious non-linearity in the unwrinkling during loading can be observed. Whereas the orientation increases significantly, the lattice parameter c in the stacking direction of the carbon layers remains nearly constant.

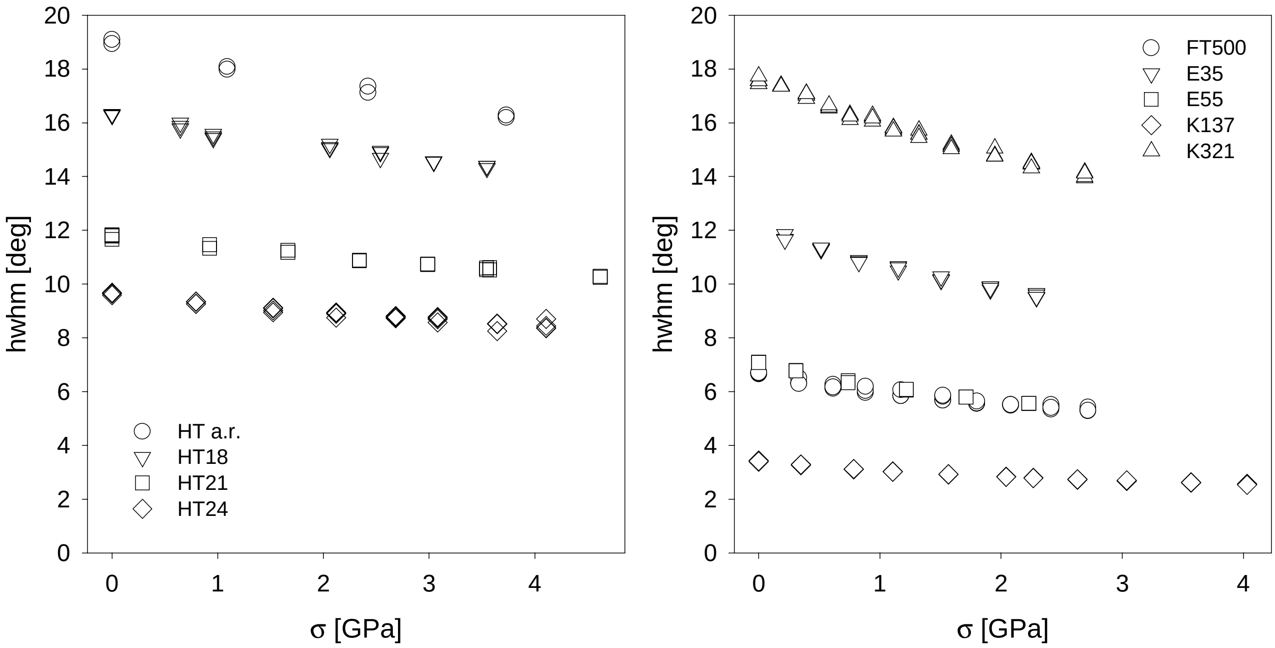

Fig.1a (left picture): azimuthal half-width at half maximum of the 002-reflection in dependence on the load of PAN-based fibers with different heat treatment temperatures. Fig.1b (right picture): azimuthal half-width at half maximum of the 002-reflection in dependence on the load of pitch-based fibers with different processing procedures.

From the evaluation of the 002-reflection and the corresponding data from the 10-band, the following results were obtained:

1) The degree of preferred orientation of the carbon filaments increases with increasing tension stress for PAN- and pitch-based fibers.
2) The strain of the fiber is composed of both, the increase of the lattice-parameter, determined from the 10-band in vertical direction, and the unwrinkling of the carbon filaments and thus the increase in their preferred orientation, determined from the decrease of the half-width of the 002-reflection.
3) From our experiment, quantitative results on the part of the respective mechanism were obtained for both types of fibers.

[1] W.Ruland. Appl. Polym. Symp. 1969;9:293-301.
[2] M.J.Endo. Mater. Sci. 1988;23:598-605.
[3] Y.Huang Y, R.J.Young. J. Mater. Sci. 1994; 29:4027-4036.
[4] O.Paris, D.Loidl, H.Peterlik, M.Müller, H.Lichtenegger, P.Fratzl. J.Appl.Cryst. 33 (2000) 695-699.
[5] O.Paris, D.Loidl, M.Müller, H.Lichtenegger, H.Peterlik. J.Appl.Cryst. 34 (2001) 473-479.
[6] O.Paris, D.Loidl, and H.Peterlik. Carbon, in print.