



	Experiment title: Structural defects and tensile properties of native and industrial flax fibres	Experiment number: SC-568
Beamline: ID13	Date of experiment: from: 05 Feb. 1999 to: 07 Feb. 1999	Date of report: February 28, 2001
Shifts: 6	Local contact(s): Martin Müller	<i>Received at ESRF:</i>
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Report:

Flax fibres essentially consist of cellulose. These cellulose chain-molecules are organised in small crystallites (microfibrils) as well as disordered regions. Thereby natural cellulose fibres can be regarded as a composite material. To determine the mechanical and structural properties of flax fibres diffraction patterns of different regions of a single fibre under stress were recorded.

The ID13 piezo stretching cell was used with a 0.5 N force sensor. Single flax fibres (industrially processed and bleached, diameter typically 10 to 20 microns) were glued with super-glue to the jaws of this tensile testing device. At zero strain, the length of the fibre was 3.3 mm.

In this experiment, the ID13 scanning set-up with focusing glass capillary was used. Beam size at sample position was about 3 microns. Diffraction images were recorded on a MAR Research CCD detector (2048 × 2048 pixels, 16 bit, 64.45 × 64.45 microns pixel size). Strain and force on the fibre were monitored in situ.

Two flax samples broke at relatively low strain. The reason was probably beam damage: The fibres were scanned through the beam several times in order to get a mesh of diffraction images across the fibre. With a third sample, this was avoided by scanning it in coarser mesh with 9 times 3 points diagonally across the fibre (edge - centre - edge). The strain of the fibre could be increased stepwise to about 1% when the fibre eventually broke at the end of the scan. Fig. 1 shows the stress-strain curve of the single fibre (lower line).

The diffraction images were further processed using fit2d. The meridional cellulose 004 reflections as well as the equatorial 200 reflections were integrated in both radial and azimuthal direction. Radial scans through the 004 reflections reveal the strain of the crystalline cellulose microfibrils in fibre (c) direction. Assuming that the stress is isotropically distributed within the composite material cellulose, a second stress-strain curve – this time for the crystalline regions – can be deduced (upper curve in Fig. 1). The elastic modulus of the microfibrils with respect to the fibre is greater by a factor of 3.5.

Surprisingly, also the equatorial lattice constants increase with strain. A possible explanation might be radiation damage: Intermolecular hydrogen bonds are destroyed and the chain-chain distance of the cellulose chains within a crystallite therefore increases. Such an effect has already been observed for electron radiation [1].

Native flax fibres are known for their characteristic “knee” defects with local variation of the cellulose orientation. As industrial flax is mechanically processed, additional structural defects are induced. The investigated fibre shows a strong variation of the degree of orientation f_c (Herman’s orientation function [2]) between 0.970 and 0.987 (Figure 2). Upon stretching, already well oriented regions ($f_c \leq 0.985$) do not undergo any changes of orientation whereas those regions with a low value of f_c become better oriented. This ordering is certainly responsible for part of the difference of elastic moduli of fibre and microfibrils.

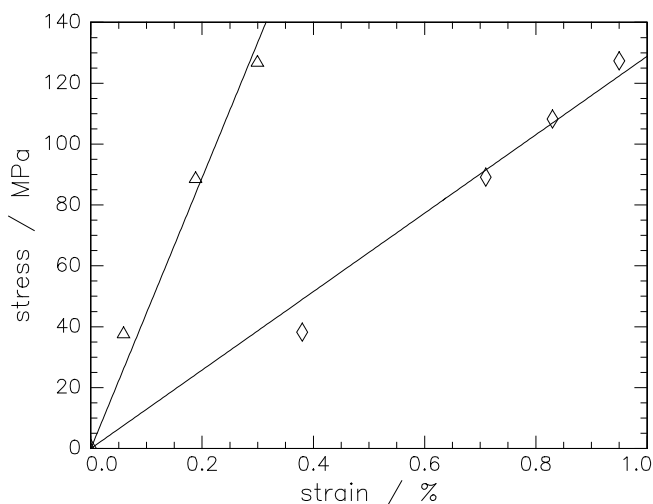


Fig. 1: Stress-strain curves of a single flaxfibre (◇), and of cellulose microfibrils in the fibre (△).

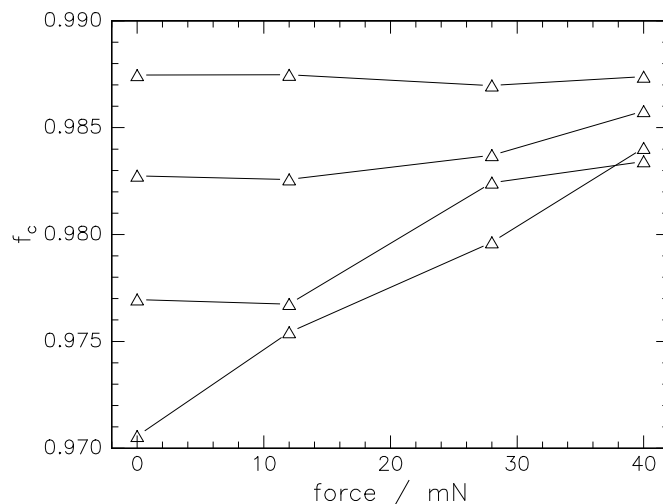


Fig. 2: Change of the degree of orientation f_c of cellulose microfibrils under tension.

[1] J. F. Revol; J. Mater. Sci. Lett. **4**, 1347 (1985)

[2] R. S. Stein, G. L. Wilkes; In: I. M. Ward, editor. Structure and properties of oriented polymers, London: Applied Science Publishers, 1975. p. 57