ESRF	**Experiment title:** Micoscopic yield mechanisms in wet wood: single wood fibres stretched *in situ*	**Experiment number**: SC-2247
Beamline: ID13	**Date of experiment**: from: 10.09.07 to: 13.09.07	**Date of report**: 02.10.07
Shifts: 9	**Local contact(s)**: Richard Davies	*Received at ESRF:*

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Preliminary Report:

The understanding of the mechanical properties of wood cell walls has to be based on their composite nature (crystalline cellulose microfibrils embedded in an amorphous matrix). Upon stretching, the cellulose microfibrils may *rotate* towards the longitudinal fibre axis so that the helix angle, the microfibril angle MFA (depending on its actual value) decreases for larger MFA [1] or that the total microfibril orientation distribution is narrowed [2]. The cellulose crystals may also be *stretched*, visible in a change of the lattice spacing in fibre direction. These two mechanisms are well reflected in X-ray fibre diffraction diagrams acquired *in situ* [3,4]. Water drastically influences the mechanical properties of wood fibres since it penetrates the disordered matrix but not the crystalline microfibrils [5].

In a previous experiment only dry single wood fibres could be investigated as part of the beamtime was lost due to a storage ring failure [4]. In the experiment reported here we used a new improved set-up for stretching in an atmosphere with controlled humidity. Data analysis is still in progress; only preliminary results are reported here.

A piezo stretching device for the simultaneous acquisition of X-ray diffraction patterns and of force-elongation data of single fibres was developed for the experiment at ID13 (Fig. 1). Constant humidity is ensured by encapsulating the device (with Kapton windows). The integrated basin was filled with silica gel (relative humidity 8 %) or water (RH 92 %). The ambient relative humidity in the air-conditioned hutch was 42 % (open cell operation). We extracted single fibres with the help of fine tweezers in an optical microscope and glued them into small pre-fabricated plastic frames. Typical fibre length was 2-3 mm with a free length of 1.1 mm in the frames.

We used the so-called scanning set-up of ID13 with a microbeam (about 1 μm diameter at the sample position) produced by KB mirrors. The whole experiment was observed from the side with a high-resolution CCD Camera through a telecentric lens system. Comparison of two subsequent images will allow for determination of local strain.

The tensile test experiment was carried out stepwise (0.5 µm steps, corresponding to strain steps of 0.045 %; see Fig. 2). The sample was allowed to relax for 10 s, and then a scan across the fibre (5 µm step size, 10 steps) was carried out. Acquisition time for a 2D diffraction pattern on was 1 s with 0.88 s between subsequent exposures.

Data analysis is still in progress. First results for dry wood are shown in Figs. 2 and 3 (red triangles): The *d*-spacing of the meridional 004 cellulose reflection, containing information on the length of the monoclinic unit cell in fibre = molecule direction, increases with increasing macroscopic strain. There is about a factor of seven between the microfibril strain ε_{004} and the macroscopic strain. The disordered matrix thus has to account for most of the extension of the wood cell upon tensile stress. It also allows a reorientation of the microfibrils, visible in the reduction of the azimuthal width of the equatorial cellulose 200 reflection.

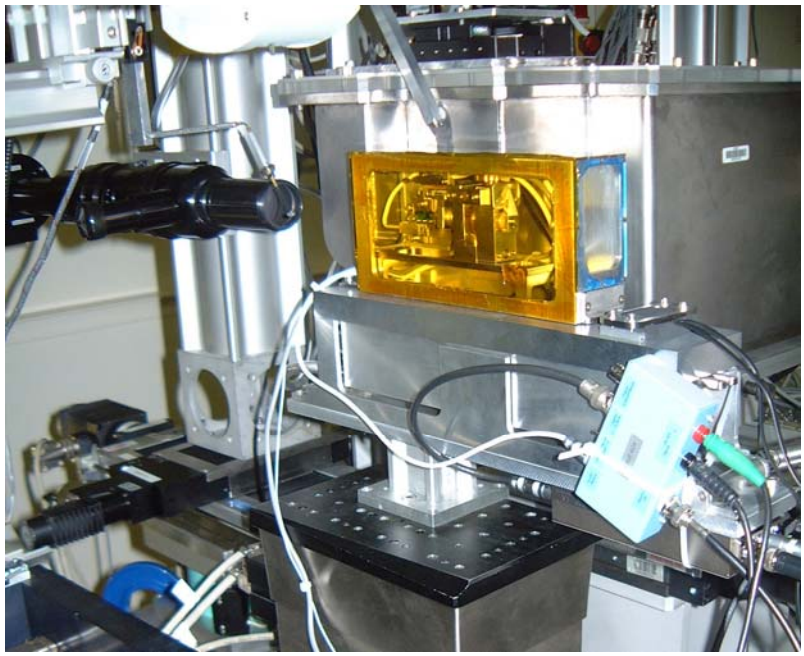


Fig. 1: Stretching set-up with humidity control and sample observation telescope as installed on ID13.

Fig. 2: Force-time curve of a single fibre of pine earlywood (MFA 10°, 10 % RH) shown in black. Each step corresponds to a strain increase of 0.045 %, the fibre breaks at a strain of ε_{max} = 1.30 %. Force relaxation on the steps is clearly visible. The red triangles show the crystal strain inside the microfibrils as calculated from the shift of the meridional cellulose 004 reflection position with increasing strain.

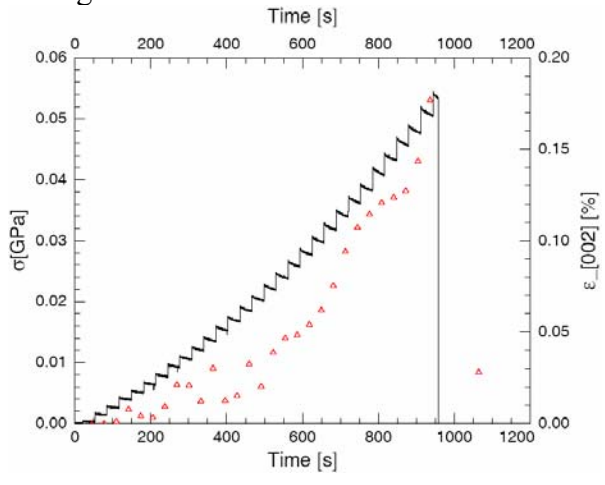
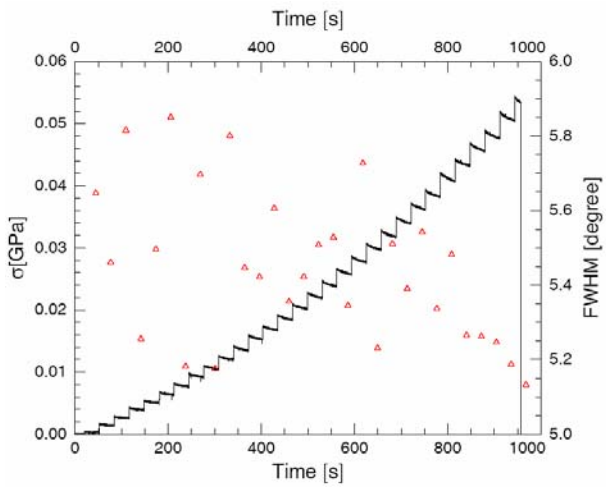


Fig. 3: Same force-time curve as in Fig. 2 shown in black. The red triangles show the width (FWHM) of the equatorial cellulose 200 reflection positions with increasing strain. Obviously the orientation of the cellulose microfibrils improves with increasing strain (or stress).



First test experiments were carried out on *silkworm silk* fibres. In the semicrsystalline biopolymer similar mechanisms as in cellulose are to be expected. The deformation of the fibroin crystal is very important (up to 1.4 %). However, the experiment suffered from beam damage leading to premature rupture of the fibre. Future experiment will have to be carried out without time-consuming scans across the fibre (secondary beam damage!) but rather with a constant strain rate and diffraction from different points on the sample. In contrast to the local structure variation in wood fibres, silkworm silk should be sufficiently homogeneous to allow for such a scanning mode.

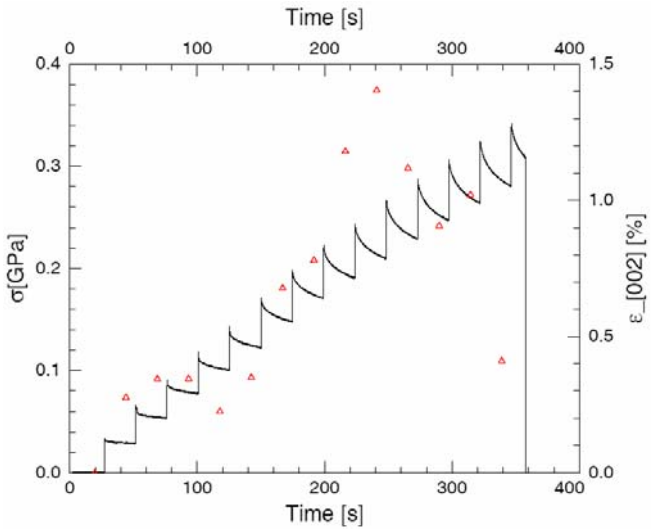


Fig. 4: Force-time curve of a single silkworm silk fibre (10 % RH) shown in black. The fibre breaks at a strain of ε_{max} = 5.90 %. Force relaxation on the steps is clearly visible. The red triangles show the crystal strain inside the microfibrils as calculated from the shift of the meridional fibroin 002 reflection position with increasing strain.

References

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[2] K. Kölln, I. Grotkopp, M. Burghammer, S. V. Roth, S. S. Funari, M. Dommach, M. Müller. *J. Synchr. Rad.* **12**, 739-744 (2005)

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[4] ESRF Experimental report **SC-2011**

[5] M. Ioelovitsch, M. Gordeev. *Acta Polym*. **45**, 121-123 (1994)