ESRF	**Experiment title:** Wet and dry wood, two very different composite materials: Mechanical investigations on the single cell level	**Experiment number**: SC-2011
Beamline: ID13	**Date of experiment**: from: 30.06.06 to: 04.07.06	**Date of report**: 19.09.06
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Report:

The semi-crystalline biopolymer *cellulose* consists of fibrous nanocrystals, the so-called *microfibrils*, embedded in a softer matrix of disordered material [1]. The main constituent of the wood cell wall is cellulose with cellulose, other polysaccharides (hemicelluloses) and lignin as the embedding matrix [2]. An additional structural parameter of the hierarchically organised biomaterial wood is the so-called *microfibril angle* (MFA), which is defined as the angle of the helical arrangement of the cellulose microfibrils with respect to the longitudinal axis of the wood cell [3].

The understanding of the mechanical properties of wood cell walls has to be based on their composite nature. Upon stretching, the cellulose microfibrils may *rotate* towards the longitudinal fibre axis so that the MFA (depending on its actual value) decreases for larger MFA [4] or that the microfibril orientation distribution is narrowed [5]. 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* [6].

Water drastically influences the mechanical properties of wood fibres since it penetrates the disordered matrix but not the crystalline microfibrils [7]. The Young's modulus is reduced as the matrix becomes softer. In the experiment reported here we could unfortunately only investigate **dry** wood fibres. Due to a failure of the storage ring, we could not carry out the second part of the experiment where **wet** wood fibres were planned to be investigated.

We extracted single fibres with the help of fine tweezers in an optical microscope and glued them into small plastic frames. These films were afterwards mounted within a specially designed mechanical holder, which permits easy handling of the samples and ensures that there is no mechanical damage possible before the measurement starts. Then the polymer films were cut apart using a soldering gun.

The sample holder loaded with the fibre was transferred into our humid stretching environment (HUSTEN) that allows the simultaneous acquisition of X-ray diffraction patterns and of force-elongation data within a humidity controlled atmosphere. HUSTEN has been described in detail previously [6].

We used the so-called scanning set-up of ID13 with a microbeam produced by compound refractive lenses. To be able to vary the position at the sample that was illuminated by the X-ray beam the whole sample environment was placed on top of a *xyz* translation table that was driven by the ID13 experimental control PC running spec©. The movements of this table were induced via TCP/IP connection between the ID13 PC in listener mode and the experiment controlling PC running a G code (bacon v0.5).
The whole experiment was observed with a high-resolution CCD Camera through a telecentric lens system looking via a heated mirror onto the sample, almost along the X-ray beam direction. A series of micrographs is shown in Fig. 1. Comparison of two subsequent images allows determination of local strain and clear identification of fibre rupture. The latter was particularly important as mechanical problems, probably mainly due to friction, did not allow obtaining quantitative force data, as is illustrated in Fig. 2 (black points).
During the tensile test experiment with a strain rate of 0.55 %/min (0.1 µm/s) the fibre was scanned through the beam over a range of 80 µm (10 steps), acquisition time for a 2D diffraction pattern on was 1 s with 1 s between subsequent exposures. Even though the fibres were mounted as straight as possible (alignment under the microscope) there is still a lateral shift of the order of 16 µm upon stretching (Fig. 3). In the further analysis, this was accounted for by always looking at corresponding diffraction patterns in the lateral scans.
Data analysis is still in progress. First results are shown in Fig. 2 (red circles): The *d*-spacing of the meridional 004 cellulose reflection, containing information on the length of the monoclinic unit cell in fibre = molecule direction, increases linearly with increasing macroscopic strain. There is about a factor of eight 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.

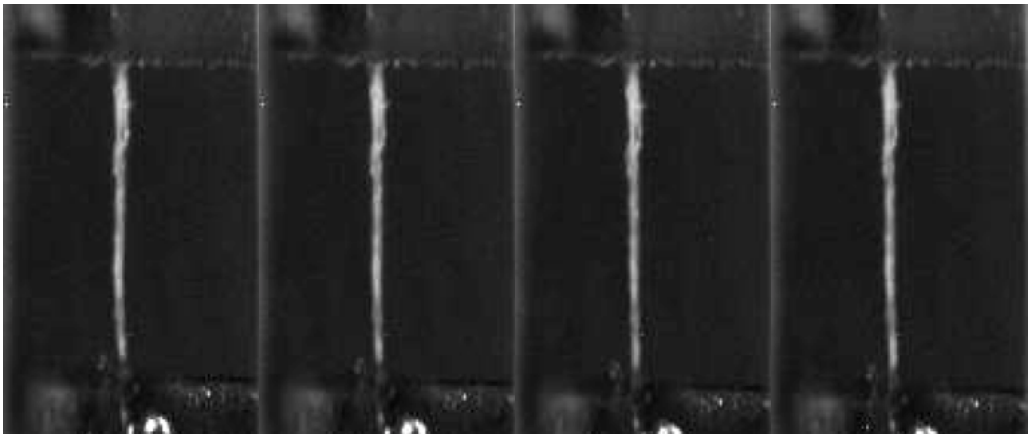

Fig. 1: High-resolution images of a single pine wood cell. Strain increases from left to right. Initial fibre length was 1.1 mm.

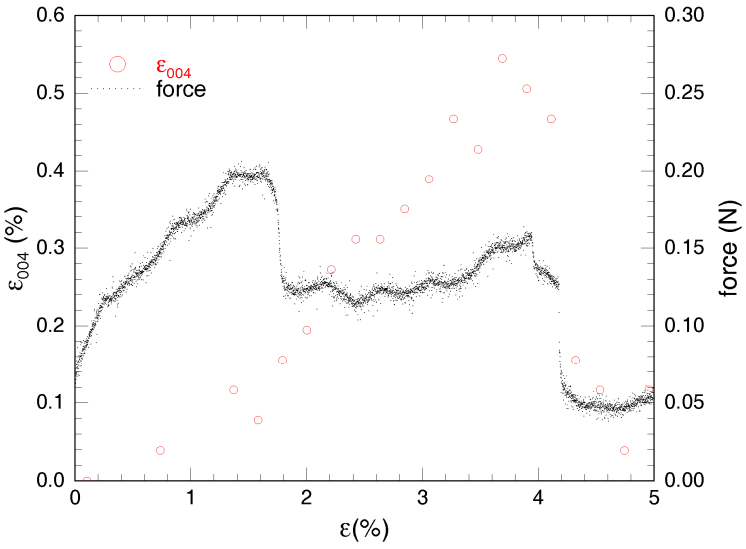

Fig. 2: Force-strain curve of a single fibre of pine earlywood (MFA 10°) shown in black. The obvious oscillation is an artefact of the stretching device (friction). The two steep drops of the force can be associated with the failure of part of the wood cell, as visible in microscope images recorded with a high-resolution CCD camera with a telecentric lens (Fig. 1). The red circles show the crystal strain inside the microfibrils as calculated from the shift of the cellulose 004 reflection positions with increasing strain. The part of the cell probed with the X-ray microbeam is only affected by the second breakage, indicating a local effect.

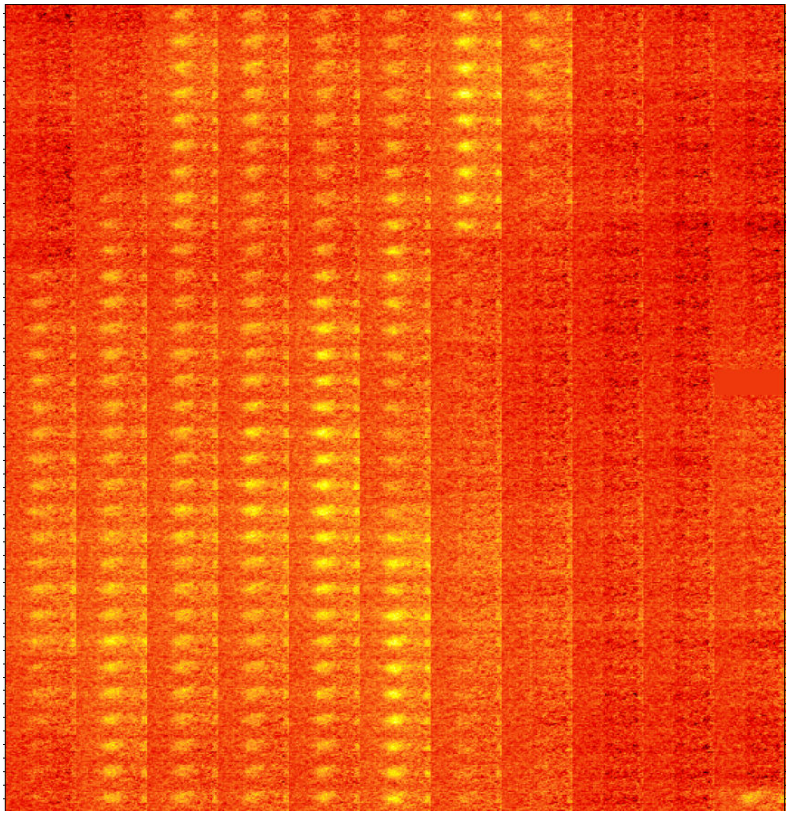

Fig. 3: Cellulose 002 reflection of a single pine wood cell under tensile stress. Time and thus strain increase from top to bottom. The single wood fibre was not completely straight at the start of the stretching experiment and thus moves laterally when it is straightened (scan step size 8 µm). Eventually, the fibre broke and moved again.

References

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