ESRF	**Experiment title:** Cellulose microfibril orientation and the mechanical properties of single conifer tracheids	**Experiment number**: ME-550
Beamline: ID13	**Date of experiment**: from: 16.7.2004 to: 20.7.2004	**Date of report**: 1.3.2004
Shifts: 12	**Local contact(s)**: Dr. Stephan V. Roth	*Received at ESRF:*

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Report:

Wood is by nature a remarkably complex composite material. When its structure and mechanical properties are studied, one faces the problem of magnitude. To really understand the material, one needs to study its basic building units, single cells, or at most very small pieces with a limited number of cells. This imposes severe challenges on the instrumentation (micrometer size beam with sufficient intensity, sample movement control to the same level of accuracy, etc.). Thus the microdiffraction beamline ID13 is in many ways an ideal site for such studies.

The aim of the experiment was to (1) continue the determination of the cellulose microfibril orientation distribution of single tracheid wall through a bordered pit, (2) determine the mechanical properties of the same material on a sufficiently small scale. Part (1) was a continuation of the experiment ME-270, which was conducted in November 2001 (see Experimental Report). Instead of the capillary optics with beam diameter 2 μm, a 5 μm pinhole collimated beam (λ = 0.96 Å) was available for the experiment. The larger beam size accounted for less pronounced radiation damage than that observed during ME-270. Thus longer exposure times, up to 150 seconds per exposure, could be used.

Part (1) of the experiment was carried out with the same procedure as during experiment ME-270. Samples were attached to thin glass capillaries by epoxy glue, the capillaries were then mounted on a Huber goniometer head. The samples were moved horizontally across the x-ray beam with 4 μm steps by using a translation stage. From the diffraction patterns of cellulose the reflection *200* was analysed.

Due to the small size of bordered pits in Norway spruce (*Picea abies* [L.] Karst.), the measurements through pits were done with samples of Scotch pine (*Pinus sylvestris* [L.]). Unfortunately these measurements were not very successful, most likely due to wrinkling of the samples during preparation. A tracheid of silver birch (*Betula pendula* Roth) was measured as well, to test the feasibility of single tracheid measurements on a hardwood species. Since the pits in the walls of birch tracheids are very small, only the orientation distribution of the double cell wall could be measured. However, the microfibril orientation distribution of a

single cell wall could be obtained from the double cell wall intensity profile by fitting (see Figure 1). To account for the asymmetry of the profile, the complete fit consisted of a sum of three pairs of Gaussian functions, mirrored with respect to the orientation parallel to cell axis (0 degrees). In Figure 1, half of the fit is shown, corresponding to the distribution in one cell wall. The shape of this distribution is quite similar to the ones obtained from single Norway spruce tracheid walls during ME-270 (an example is shown in Figure 2), although the wood species is completely different.

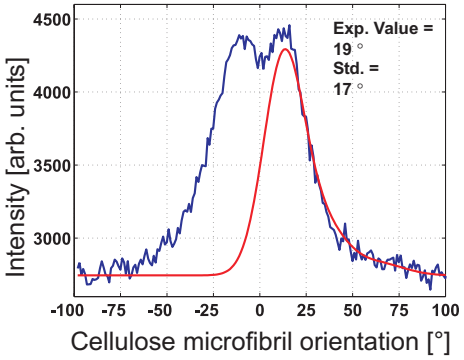

Figure 1. The cellulose microfibril orientation distribution of a silver birch tracheid. Blue line, measurement data; Red line, MFA distribution of a single cell wall.

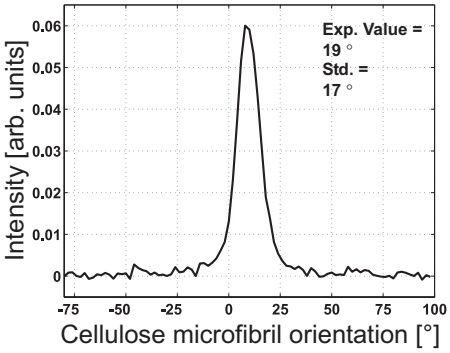

Figure 2. The cellulose microfibril orientation distribution of a macerated Norway spruce tracheid, obtained during the experiment ME-270.

In part (2) the piezoelectric stretching cell available at ID13 was used as the sample stage. The samples were stretched with a constant rate of 0.2 μm/sec and diffraction patterns of crystalline cellulose were recorded *in situ*. Time resolution of the experiment was 21 seconds (exposure time 15 seconds per frame, dead time of the detector 6 sec.). From the diffraction patterns the reflections *200* and *004* were studied. Observing the changes in the profile of *004* make it possible to deduce the extent of deformation of the crystalline cellulose unit cell along the cellulose chains, whereas the *200* gives information on the perpendicular direction.

In order to measure the mechanical properties of wood in its native chemical composition, small pieces of wood were used instead of macerated tracheids. Samples were made of normally grown Norway spruce from southern Finland. The dimensions of the samples were approximately 8mm (length) * 0.2mm (width)* 75 μm (thickness). A typical set of measurement data is shown in figures 3 — 5. Analysis of the reflection *004* revealed changes in its position with respect to the scattering angle, also the FWHM (full width at half maximum) of the reflection changed. The changes were not large but they appeared in every analyzed dataset and correspond well to the stress/strain curves. Strain causes elongation of the unit cell, recovery to the initial length takes place immediately after fracture of the sample. Further analysis of the stress/strain curves and the respective diffraction patterns, especially considering the reflection *200*, is in progress.

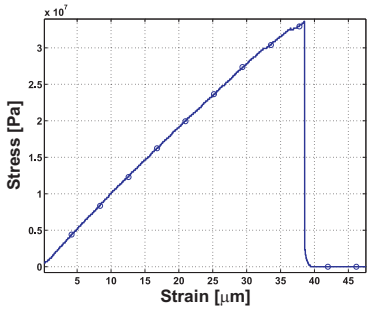

Figure 3. Stress/strain curve of a Norway spruce sample. The circles point out where one measured frame (diffraction pattern) ends. The first frame measured in tension is number 2.

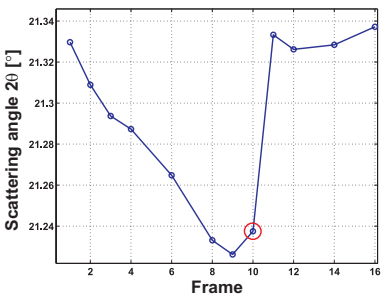

Figure 4. The change in scattering angle of reflection 004. Frame number denotes the position in the stress-strain curve. Approximate position of fracture is indicated by the red circle.

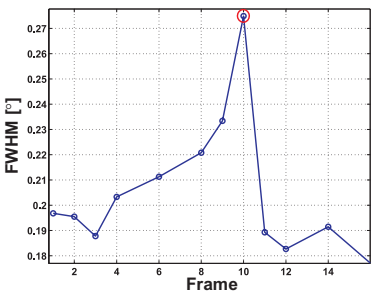

Figure 5. The FWHM of reflection 004 in the same direction as in Figure 4. Frame numbers and position of fracture as in Figure 4.