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Report:

The cell wall of tracheid wood cells is a natural fibre composite of cellulose crystals (microfibrils) embedded in an amorphous matrix of lignin and other carbohydrates (hemicelluloses). Spatially, the cell wall is layered with a primary (P) and three secondary (S1, S2, S3) walls (Fig. 1). The S2 layer comprises about 80 % of the wall thickness and the cellulose content in the S layers is around 60 weight-%. The microfibrils of the S2 are well–aligned and arranged in a steep right–handed (Z–) helix around the lumen. The angle of the helix with respect to the longitudinal cell axis is defined as microfibril angle (MFA). The mechanical properties of a wood cell are, however, not only determined by the major contribution of the S2 but as well by the slow left–handed (S–) helices of the thinner S1 and S3 layers. Furthermore, there is evidence from electron microscopy studies of developing cell walls [1,2] or stained sections [3,4] that the change of microfibril orientation between neighbouring S layers is gradual. Such a helicoidal wall structure is mechanically much stronger than a simply layered system as it reduces the tendency to split axially and to fail to resist shear forces.

The analysis of experiments in standard geometry, i.e. with the beam perpendicular to the cell axis can only yield the microfibril orientation in the complete cell wall, but cannot attribute the distribution to certain wall layers [5]. By scanning through the walls of thin cross sections, the spatial MFA distribution can be resolved.

As the amorphous matrix does not contribute any crystal reflections, the scattering pattern of thin cross sections is the same as from a tilted fibre bundle. The curvature of the Ewald sphere and the tilted fibres lead to an asymmetric scattering pattern [6]. The azimuthal distribution of the scattering intensity can be used to calculate the fibre tilt angle, which is in this case equivalent with the MFA.

To determine details of the cell wall structure, the new KB mirror optics in the EH3, providing a beam size of 215(v) x 235(h) nm², was used. Scans through a cell wall (Fig. 2) were performed with a spatial resolution of

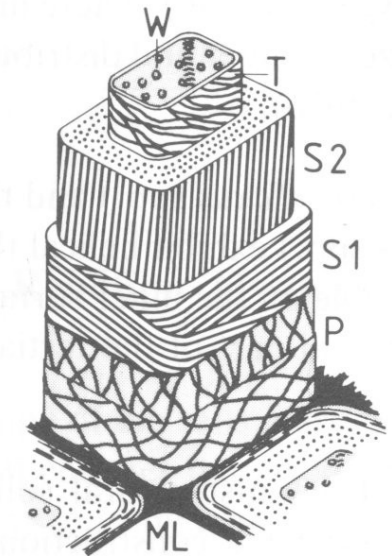

Figure 1: Schematic drawing of the layered structure in a softwood tracheid wall. The marks are explained in the text [7].

200nm. As a double cell wall has a thickness of about 5-6µm, each scan gave about 25-30 data points. The resolution is limited by the accuracy of the sample alignment. A tilt of the sample leads to the different cell wall layers not being strictly separated and thus the signal of different wall layers is superimposed in each diffraction pattern. The beamline design planned a hexapod for fine sample alignment. As this hexapod had not yet been delivered, the sample stage was temporary built from three independent goniometer cradles and 3 linear motors. Unfortunately, this design did not allow to rotate the sample without displacing it from the focal point. Thus, the alignment had to be performed by visual judgment without any chance for fine tuning online. This leads to difficulties in the data analysis which could be avoided in the future.

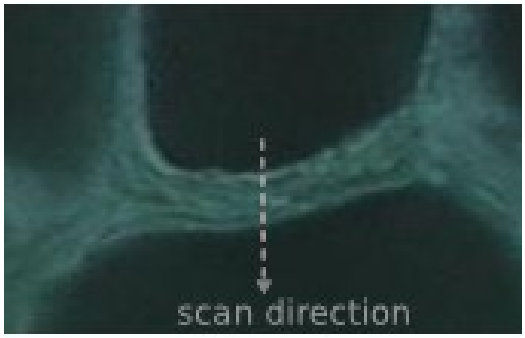

Figure 2: Online microscope image of sample; scan direction is indicated by the dashed line.

As most biological samples, cellulose is very sensitive for radiation damage. This was a problem at first, but by tweaking the parameters of exposure time and scan spacing, it could be bypassed. If scanning fast enough and with step-width large enough, the scan seems to be faster than the diffusion of the secondary beam damage. One-dimensional scans could be performed with a resolution of 200nm and 1 second exposure time, 2D meshes (Fig. 4) with a resolution of 200x400nm² exposure times of 0.3 seconds. Figure 3 shows very nicely how the fibre orientation in the cell wall turns, visible as a flipping of the diffraction pattern along the equator.

Figures 5 and 6 show the azimuthal scattering intensity of the cellulose 200 reflection and the resulting calculated MFA distribution. While the analysis is still in an early stage, the higher MFA in the S1 wall can be seen in the central region of the scan.

Due to the problems of the sample alignment mentioned above and time constraints, only one type of wood could be measured extensively. Of the 12 schedules shifts, the first seven shifts had to be used for beamline commissioning as the present experiment was the first user experiment with the new set-up.

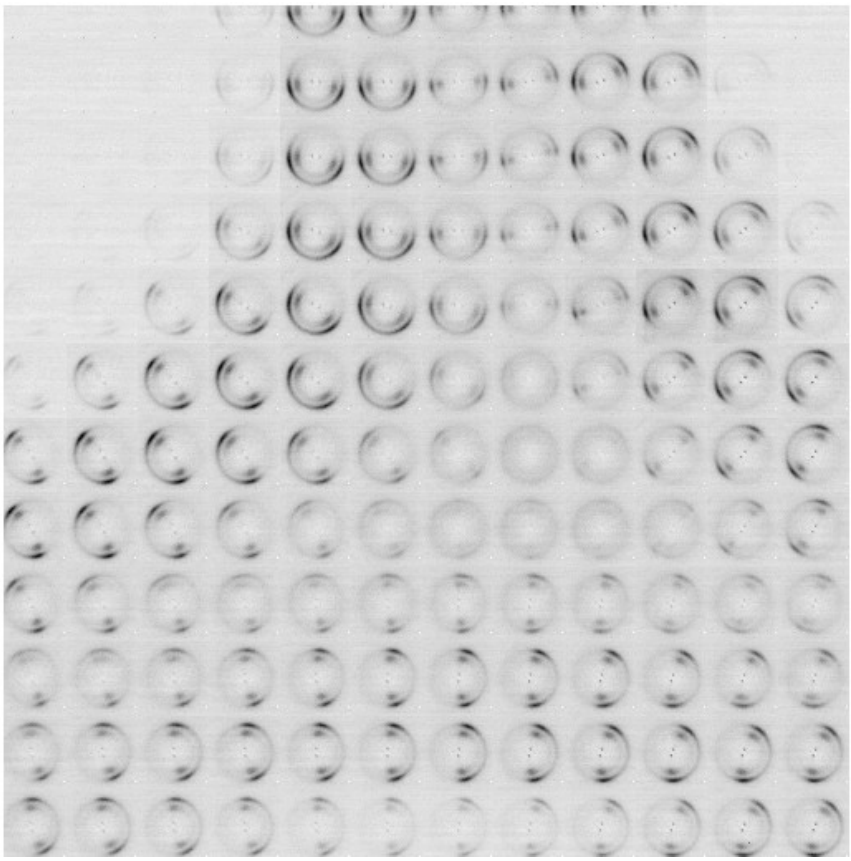

Figure 3: Composite image of diffraction patterns, mesh width is 400 x 400 nm².

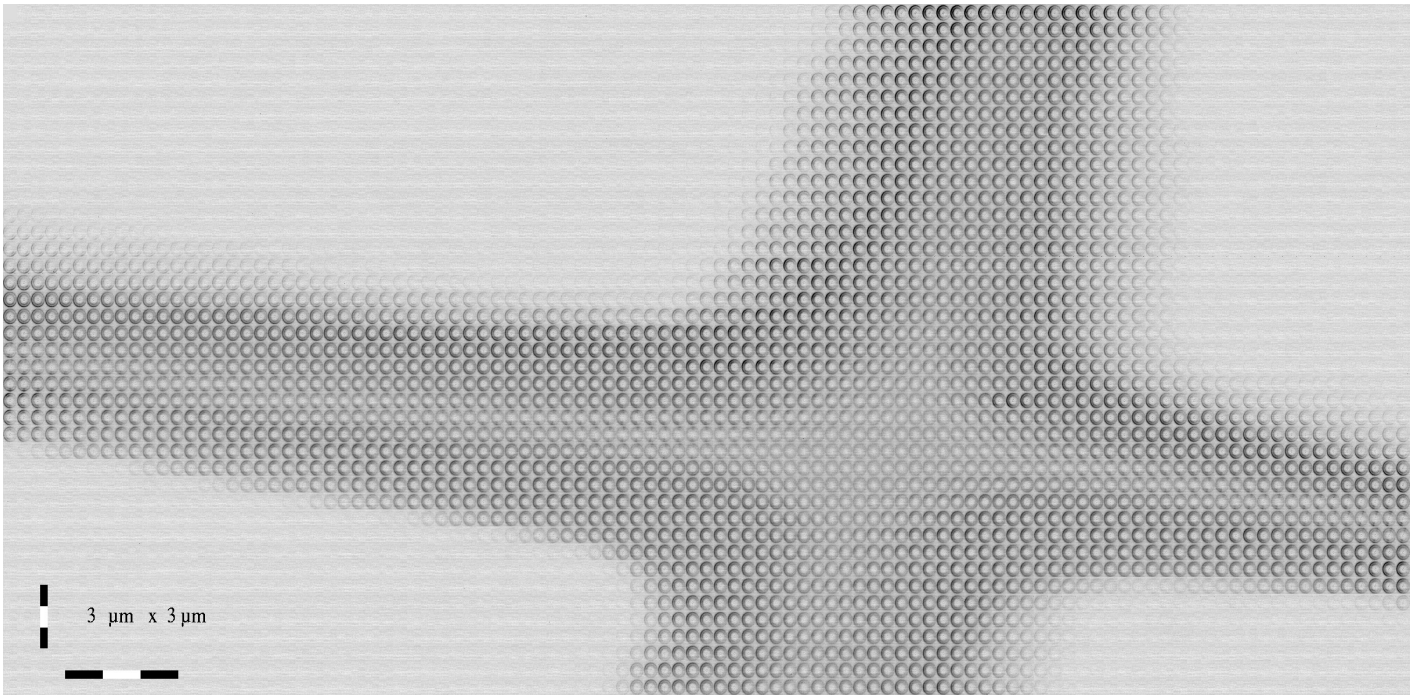

Figure 4: Mesh scan of a corner region, where 4 tracheid cells meet. The wall layer structure is clearly visible.

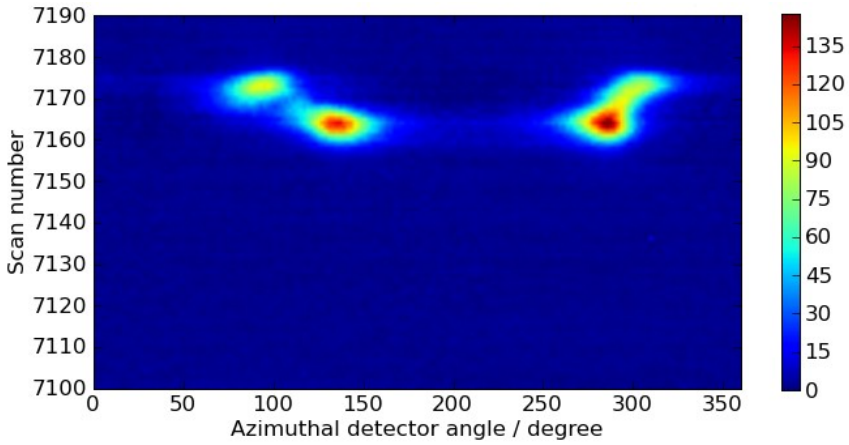

Figure 5: Scan resolved azimuthal scattering intensity.

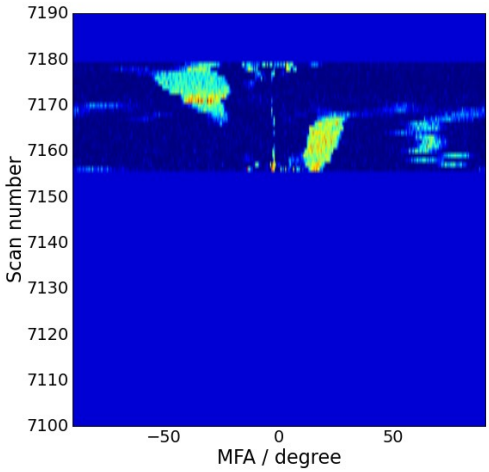

Figure 6: Scan resolved MFA distribution.

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