ESRF	**Experiment title:** Cell wall structure of Norway and Sitka spruce tracheids	**Experiment number**: ME-270
Beamline: ID13	**Date of experiment**: from: 12.11.01 to: 16.11.01	**Date of report**: 1.3.02
Shifts: 9	**Local contact(s)**: Christian Riekel	*Received at ESRF:*

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Report:

The aim of the study was to determine the microfibril angle (MFA) distribution from single, macerated tracheids of Norway spruce by using synchrotron x-ray microdiffraction from reflection 200. The Norway spruce samples were a part of a nutrition optimisation experiment, taking place in Flakaliden, northern Sweden (64°07'N, 19°27'E, altitude 310 m). Longitudinal sections made from the same Norway spruce samples and transverse Sitka spruce and ancient wood sections were also measured. Their analysis is still in progress.

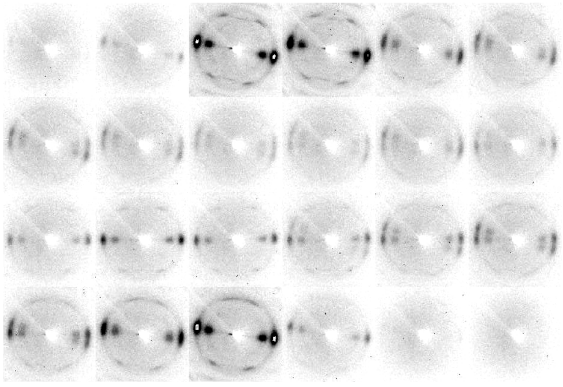
Previously the average MFA distributions from the same wood material were determined using a conventional x-ray source [1,2]. The size of the sample was 1cm*1cm*1mm (referred to as bulk sample) and the x-ray beam was scattered from several thousands of tracheids. MFA measurements of single, macerated fibres were also carried out using polarization microscopy [3]. The results were not the same. The microfocus beamline ID13 at ESRF (European Synchrotron Radiation Facility), Grenoble provided the opportunity for reliable comparison of these methods.

Radiation damage to the sample was found to be considerable, which limited the exposure time to 20 seconds per sample.

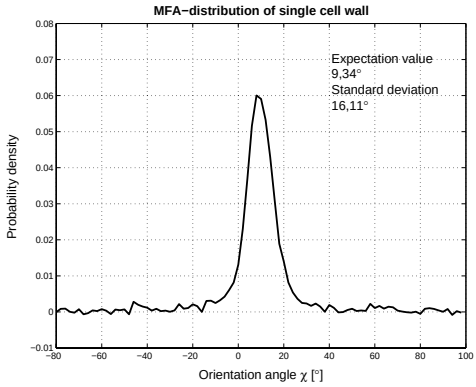
The experiment with macerated tracheids was very successful in revealing the diffraction pattern of a single cell wall through a bordered pit. This is the first time such a pattern has ever been measured from an intact tracheid. It allowed direct comparison between polarization microscopy and X-ray diffraction from exactly the same position of the cell wall. The average MFA values given by microscopy and microdiffraction are notably different, a result, which needs further quantification by a more thorough series of measurements.

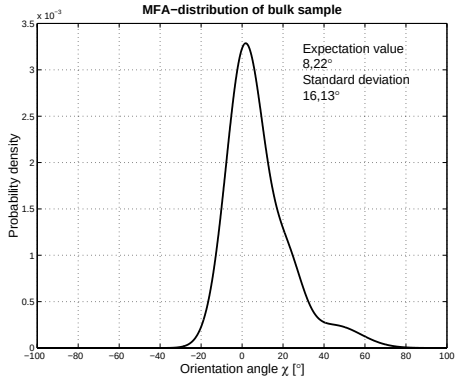
When the MFA distribution obtained by microdiffraction was compared to the distribution measured by a conventional x-ray source, the shape of the distribution was found to be very similar, although the latter is much wider. This is probably due to the difference in sample preparation (bulk sample with hundreds of tracheids versus one macerated cell). The primary peak shows a clear asymmetry towards large angles, which may indicate either an asymmetry in the contribution of S2-layer or the presence of transition layer(s) between S1 and S2 or S2 and S3. Unfortunately the statistical accuracy of the measurements at ESRF did not allow for any contribution of S1- or S3-layers to be identified.

Picture on the right: Diffraction patterns of a macerated tracheid scanned in 2 μm steps. The steps run from left to right, row after row forming a 48 μm long and 2 μm wide path across the cell. Note the disappearance of the other stripe-shaped pattern in the middle frames. These show the diffraction pattern of a single cell wall measured through a bordered pit.



Pictures below: MFA distribution of a single cell wall measured at ESRF (left) and of a larger sample (right). The single tracheid was obtained by maceration from the larger sample. The MFA distribution of the single tracheid wall is based on the diffraction patterns shown above. Note that the expectation values and standard deviations given are descriptive statistics of the distribution, not indications of measurement accuracy.





The mean MFA values obtained from different sample types and measurement methods are listed below:

Sample type	MFA (x-ray diffraction)	MFA (microscopy)
Bulk	8,2 °	13,0 ° (mean value*)
Single cell wall	9,3 °	20 °

*Microscopy result of bulk sample is based on measurements of 50 tracheids from a macerated sample parallel to the one measured by x-ray diffraction (conventional x-ray source at University of Helsinki).

References:

[1] T. Paakkari, R. Serimaa: *Wood Sci. Technol.* ***18*** *(1984) 79.*

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[3] L.A. Donaldson: *Wood Fiber Sci.* ***23*** *(1991) 290.*