Microdiffraction studies on the structure of single wood cell walls

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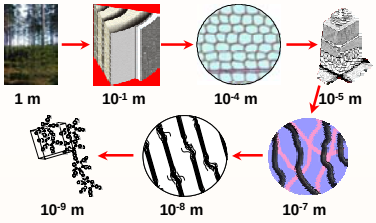
INTRODUCTION

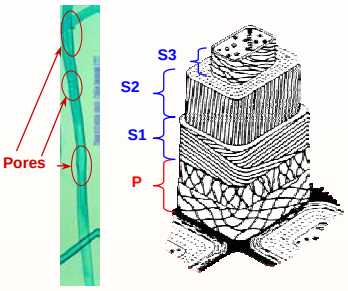


A schematic view of wood on different length scales.

A: The whole stem. B: Several annual rings. C: Several cells. D: One cell (tracheid). E: Cellulose (black) and hemicelluloses (pink) in a lignin matrix (blue). F: Cellulose microfibrils. G: Unit cell of crystalline cellulose.

One of the most important parameters affecting the mechanical properties of wood is the orientation of partly crystalline cellulose microfibrils in the cell wall. The angle that the microfibrils form with the longitudinal cell axis is called the microfibril angle (MFA). Due to experimental difficulties, there is a scarcity of information about the variation of MFA in a single cell wall.

The microfocus beamline ID 13 provides a unique opportunity to observe and characterise this complex structure. In addition, two different methods of MFA determination, x-ray diffraction and polarisation microscopy, were compared by measuring the same area of several cell walls with both methods.



A micrograph of a conifer (Scotch pine) tracheid and a schematic illustration of the wall structure.

Pores on the cell wall are highlighted by arrows and red ellipsoids. P: Primary cell wall. S1-S3: Secondary cell wall layers.

Measurements and data analysis

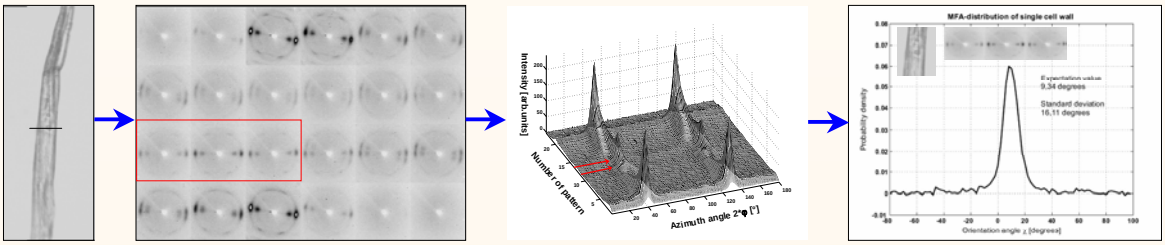
The x-ray beam was guided through pores in the cell walls. The measurements were carried out by positioning the pore on the x-ray beam and by scanning the beam horizontally across the cell, producing a set of diffraction patterns. The microfibril orientation distribution of the single cell wall was obtained from the azimuthal intensity profiles of reflection *200* [2] of cellulose. Patterns just outside the pores were used to align the orientation distribution.

During the first experiment session (ME-270) Norway spruce tracheids were measured using the glass capillary optics with a beam diameter of 2 μm. In addition to x-ray diffraction, polarisation microscopy was used to determine the average orientation of microfibrils [3].

During the second session (ME-550) Scotch pine and silver birch tracheids were studied using pinhole optics with a beam diameter of 5 μm. No polarisation microscopy measurements were performed.

Samples

The samples were single wood cells (tracheids) of Norway spruce *(Picea abies* [L.] Karst.), Scotch pine (*Pinus sylvestris* [L.]) and silver birch (*Betula pendula* [L.]), separated from pieces of wood by Franklin's method of maceration [1]. During this process pores on the cell walls, through which adjacent cells are connected, are left open (pit apertures; see the micrograph above). These act as windows through which the other side of the cell can be observed. The single cells were attached by epoxy glue on top of glass capillaries, which were mounted on a goniometer head to facilitate the correct alignment of each sample in the x-ray beam.



An example of the measurement procedure on Norway spruce.

From left: Microscope image of the tracheid, the scanning position is shown by the vertical line. Set of diffraction patterns across the cell, patterns inside the red rectangle are taken through the pore. The intensity profiles of the reflection 200 of cellulose, the red lines mark the area from a single cell wall. The orientation distribution of cellulose microfibrils in a single cell wall.

Sample	Annual ring	Mean MFA by XRD / PM [°]
A	12th	13 / 13
B	12th	14 / 7
C	15th	19 / 32
D	18th	9 / 20
E	18th	15 / 10

Summary of results on Norway spruce.

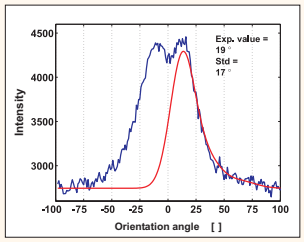
Comparison between by x-ray diffraction (XRD) and polarisation microscopy (PM).

Results and conclusions

The measurements on Norway spruce were successful in revealing the microfibril orientation distribution of a single cell wall. The distributions are narrow, but of finite width, and asymmetric with respect to the primary peak. These features should be taken into account when modelling the structural and mechanical properties of wood.

Comparison between x-ray diffraction and polarisation microscopy methods shows that even though the measurements are conducted on the same position of the cell, these methods do not give consistent results. The reason for this behaviour is not yet clear.

In the case of birch, the small size of the pores prevented their use as windows. However, the microfibril orientation distribution could be obtained from the double cell wall intensity profile by fitting. The measurements on Scotch pine tracheids were not very successful, most likely due to wrinkling of the samples during preparation.



The microfibril orientation distribution of a silver birch tracheid obtained from the entire cell.

Blue line, measurement data; Red line, MFA distribution of single cell wall.

References

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