ESRF	**Experiment title:** Cellulose microfibril organization in cellulose fibres and cell walls	**Experiment number:** SC-419
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Names and affiliations of applicants (* indicates experimentalists):

P. Fratzl, H. Lichtenegger* & O. Paris*
Erich Schmid Institut der Österreichischen Akademie der Wissenschaften, Jahnstrasse 12, A-8700 Leoben

H. Chanzy, C. Czihak*, M. Müller* & H. Schober*
ESRF

Report:

The cell wall of plants is a natural fibre composite, reinforced by crystalline cellulose microfibrils, the arrangement of which is known to be a crucial factor in determining the mechanical properties of the cell wall, in particular for wood cells (1). In wood, the major part of the cell wall, the secondary wall S2, is built up by cellulose fibrils that are strictly parallel to each other and trace a steep spiral around the cell. In the present experiment we focussed (a) on the cell wall texture, i.e. the correlation between the arrangement of the microfibrils and the orientation of the cell wall, and (b) on the helical orientation of the cellulose spiral in the S2.

These questions cannot be addressed using a beam from a conventional X-ray source that hits a great quantity of cell walls of various orientation which results in a superposition of signals that does not allow any conclusions about fibril orientation in a particular cell wall.

The experiment was started with a texture measurement: 10 μm thin slices of spruce wood, cut in tangential direction, were positioned perpendicular to the beam and then rotated by ± 30° around the longitudinal cell axis in steps of 10°. At each rotation angle, linear scans across several cells were performed, the diameter of the X-ray beam being so small, that only a single cell was hit at the time, and wide-angle diffraction patterns recorded at each point. In this measurement geometry, the (020) reflection of cellulose and the (110) + (110) reflections

do not lie in the same plane of observation. So the lack of any significant variation in the intensity ratio of these reflections when rotating the sample led to the conclusion that the cellulose fibrils are randomly rotated around the c-axis.

In the second part of the experiment we investigated the helical orientation of the cellulose microfibrils in *Picea abies*, using 10 μm thin cross sections of latewood with known cellulose tilt angle (20° with respect to the longitudinal cell axis). Two dimensional scans in 2μm steps were performed.

(a) (b)

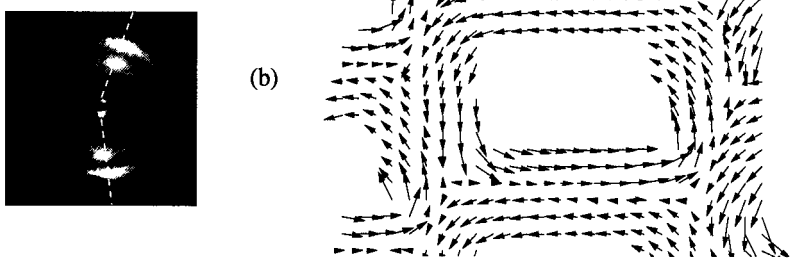

Fig. 1: (a) Wide angle X-ray diffraction spectrum of a single cell wall of latewood with fibril tilt angle 20° in *Picea abies*. Ewald sphere effects lead to spectra that are not symmetric versus rotation by 180° (the lines are guides to the eye). (b) Quantitative evaluation of the asymmetry of a single spectrum yields the orientations of the cellulose fibrils in one measured pixel denoted by an arrow. The whole picture represents a mesh scan over one complete cell and parts of neighbour cells. Note the regions filled with arrows exactly correspond to the cell walls, the empty regions to the lumina.

The resulting spectra were highly assymetric (Fig. 1a) due to an effect caused by the curvature of the Ewald sphere when using the actual experimental wavelength of 0.78 Å. The direction of the asymmetry provides information on the orientation of the fibrils (Fig. 1b) and thus allows one to draw two most interesting conclusions: (i) in a single wood cell the cellulose spiral runs in one direction only, there are no crossed spirals, (ii) in all the cells we investigated, the helical orientation was right–handed (see for example Fig. 1b). The mechanical implications of result (i) are striking: the extensibility of cells built up by a single spiral is expected to be very high in contrast to crossed spirals.

It would be of great importance to perform additional experiments of this kind to find out whether the absence of a cell wall texture and the existence of a right–handed cellulose spiral only are common features in *Picea abies* in particular or even in wood in general.
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(1) C. Matthek and H. Kubler, The internal optimization of trees, Springer Series in Wood Schience, Berlin 1995.