ESRF	**Experiment title:** **Structural changes in wood upon stretching: in-situ tensile tests on wood foils with SAXS**	**Experiment number**: SC850
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Report:

Wood represents a natural composite with the ability to adapt its structural properties to external mechanical requirements on all hierarchical levels [1]. On the microscopic and nanoscopic scale, the structural optimization of the mechanical behavior of wood is closely related to the cell wall microstructure. It is well-known that the stiffness of wood is mostly given by the semi-crystalline cellulose microfibrils. They are wound in the form of a Z-Helix around the lumen of the tube-shaped wood cells. The tilt angle of the cellulose fibrils versus the longitudinal cell axis, usually called microfibril angle (MFA), plays an important role in determining the actual stiffness of the material. Though there have been a significant efforts to characterize MFA values in different types of wood, on different scales and using various experimental techniques, systematic investigations of wood microstructure under external forces have nor been performed yet. Thus the link between optimized wood parameters and the driving force of the optimization - mechanical stress - is still not understood.

The goal of this experiment was to characterize microstructural changes in wood slices under external loading using x-ray scattering. For this purpose, three different compression wood types were chosen, namely *Ginkgo biloba* L., *Juniperus virginiana* L. and *Picea abies* [L.] Karst. The wood foils of the dimensions 5 x 50 x 0.2 mm were strained in a tensile stage provided by users under various strain rates in wet conditions monitoring strain/stress response and collecting x-ray diffraction pattern (XRD) using a 2D CCD detector. Since the scattering experiments in SAXS geometry did not provide satisfying intensity, WAXS geometry was chosen with a sample-detector distance of about 80 mm. Depending on the strain rate (from the range of 0.005-0.3 mm/s), the WAXS frames were acquired for 1-4 seconds. In order to maximize the scattered intensity and obtain a volume-averaged information from a large part of tissue, the beam size of 0.8 mm in diameter was applied.

The WAXS frames collected during stress-strain experiments were used to determine the magnitude of MFA as a function of strain. The MFAs values were evaluated from the positions of cellulose 200

reflections as demonstrated in our previous work [2]. In Figure 1, two frames collected at the beginning and at the end of a stress/strain experiment are presented.

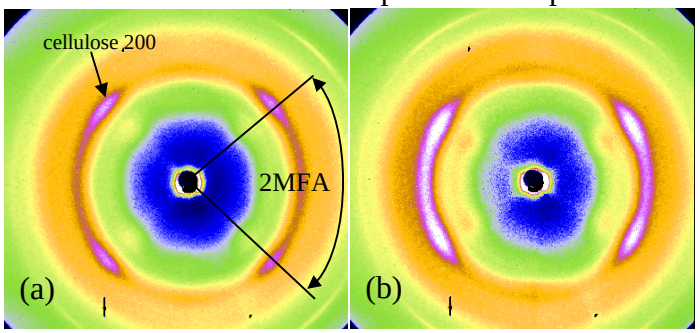

Figure 1: WAXS patterns collected during a stress-strain experiment on a slice of *Picea abies*. The frames left (a) and right (b) were taken at the beginning and at the end of the tensile experiment, respectively. The mutual position of the equivalent cellulouse 200 reflectionse is proportional to the magnitude of 2xMFA. During the experiment, MFA decreased from 45 to 30 degrees.

The frames document a change of MFA from 45 to 30 degrees. After determining MFA values from all frames collected during a specific tensile experiment and relating them with a corresponding stress-strain curve, it was possible to evaluate changes of MFA, MFA distribution and microscopic strain in cellulose fibrils as a function of external strain and strain rate in the slices. Figure 2 presents a typical stress-strain curve together with a MFA(ε) dependence.

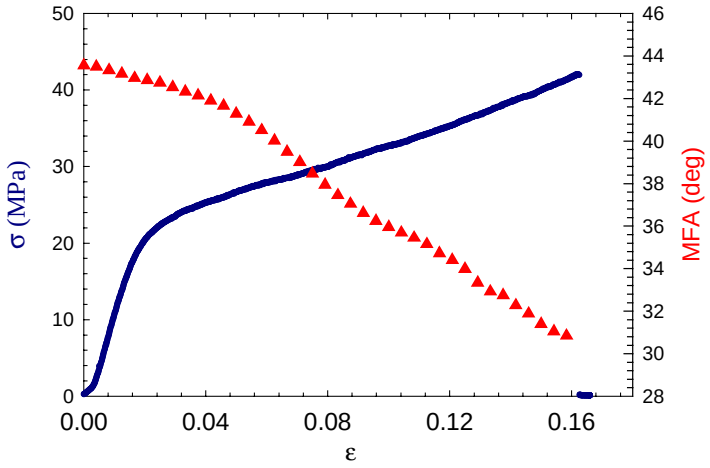

Figure 2: In-situ synchrotron experiment on a wet foil of *Picea abies* - a typical stress-strain curve (blue) and a strain dependence of MFA (red triangels). The sample was strained with a strain rate of 0.025 mm/s and the frames were collected for 4 seconds. The neraly linear experimental dependence of MFA on strain is in a very good agreement with the relationship $MFA(\varepsilon)=MFA(0)-cotg[MFA(0)]\ \varepsilon$.

One of the key features observed for all specimens from the three wood types was that the MFA decreases with the applied strain. Moreover, for the tissues with MFA values from the range of 25-48 degrees, experimental dependencies of MFA on strain were found to depend decisively on the magnitude of MFA in the unstrained state (Fig. 2). A second observation from the in-situ experiments is that the relation between MFA and elongation does not differ significantly before and after the yield point. Interestingly, the tissues with smaller MFA (*Ginkgo biloba* L.) exhibited stress-strain curves with dominant elastic region and maximal strain of about 3%. On the other hand, the stress-strain curves from samples of *Picea abies* with MFA>38 possess always an elastic and a plastic region with a maximal strain up to 20%.

The comparison of the mechanical and the microstructural results from three different compression wood types allows to draw relevant general conclusions regarding the role of different microstructural features of wood on the mechanical behaviour as well as to deduce wood architecture units progressively optimised during evolution.

The data from this experiment and from the complementary experiment SC-910 (in-situ tensile testing of individual wood cells - ID13 beamline) were used to understand a tensile behaviour of wood and deduce a mechanical principle which should be highly conserved throughout species.

[1] Gordon, J. E. & Jeronimidis, G. Composites with high work of fracture. *Phil. Trans. R. Soc. Lond.* **294**, 545-550 (1980).

[2] Lichtenegger, H., Reiterer, A., Stanzl -Tschegg, S.E. & Fratzl, P. Determination of spiral angles of elementary fibrils in the wood cell wall: comparison of small-angle x-ray scattering and wide-angle x-ray diffraction. In: *Microfibril Angle in Wood*, Butterfield, B.G. Editor IAWA-press, pp. 140-156 (1998).