dynamics of condensed systems

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In-situ X-ray Scattering of Spruce Wood with Variation of Load and Humidity

A. Müllner*[1], D. Gitschthaler[1] and H. Peterlik[1]

[1]Universität Wien, Fakultät für Physik, Boltzmanngasse 5, 1090 Wien, Austria

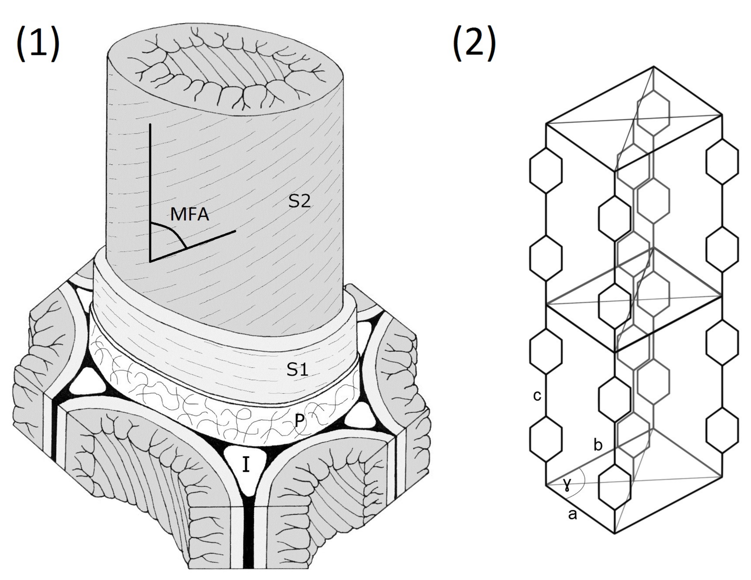
*alexander.muellner@univie.ac.at

Introduction

Due to its availability as natural resource and its good cost-efficiency, wood has become a material of increasing interest for the construction industry in recent years. A particular advantage of wood is its high strength and fracture toughness, whereas humidity could be a limiting factor because it is often accompanied by swelling, softening or fungal infestation. To investigate structural changes on the nanoscale, in-situ X-ray scattering of spruce wood by varying load and humidity was performed. A suited X-ray transparent chamber was developed, which enables measurements at variable strains in a humidity range between 0 and 100% with 0.1% precision. For the wide-angle X-ray scattering (WAXS) signal, an image plate at a sample-to-detector distance of 5.1 cm was used, whereas the small-angle X-ray scattering (SAXS) intensity passed through a hole in the WAXS image-plate and was detected by a second image plate at a distance of 65.6 cm. The microfibril-angle and cellulose lattice spacing were evaluated from both SAXS and WAXS intensities.

Background - Wood

Wood is a highly complex, polymer-like biomaterial, which combines ideal elastic properties: high mechanical strenght at relatively low weight. Differences in fracture toughness, rigidity or elasticity appear depending on the wood species.
Wood is built from parallel tube-like cells, the so-called tracheids. Wood cell walls consist of cellulose microfibrils of ≈ 2.5 nm diameter and ≈ 30 nm length embedded in a matrix of hemicellulose and lignin. Cellulose microfibrils consist of crystalline cellulose and are arranged in several layers of different thicknesses. In these layers microfibrils wind round the lumen in a Z-helix-shape; the pitch of the helices differs in every layer.



(1) Hierarchical structure of a wood cell [1];
(2) Crystalline cellulose

The microfibril-angle MFA is defined as the tilt-angle of the helically arranged microfibrils in the S2-layer relative to the longitudinal axis of the tracheid.
A dependence of the microfibril-angle on external strain ϵ in the form of

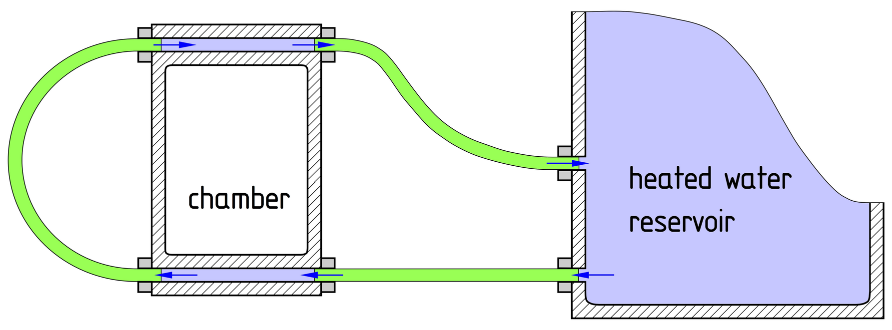
$$MFA(\varepsilon) \approx MFA(0) - \cot[MFA(0)] \cdot \varepsilon$$

was found [2].
Crystalline cellulose is reported [3] to have a monoclinic crystal structure with lattice spacings of $a = 0.7784$ nm, $b = 0.8201$ nm with an angle $\gamma = 96.5°$ and $c = 1.0380$ nm.

Humidity and Tensile Test Chamber

Leak-proof Heated Chamber

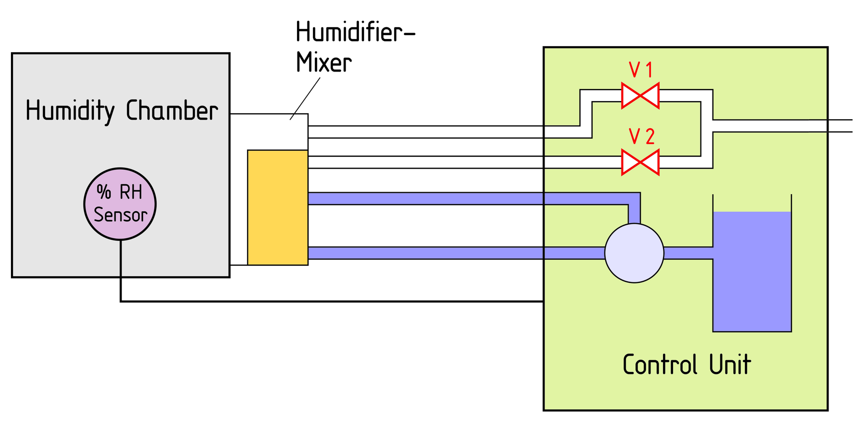
A box with inner dimensions of $12 \times 4.8 \times 5.3$ cm was milled out of a block of aluminium.
Because it should contain air humidity it was designed as leak-proof as possible and a heating system for the chamber walls was installed to prevent condensation on the aluminium surface.



Heating is realized by pumping hot water from a heated reservoir of known temperature through the chamber walls.
X-ray transparent windows on front and backside are needed to perform X-ray scattering on a sample inside the chamber. For this purpose aluminium foil was chosen, because it can be heated alongside the chamber.

Humidity Source

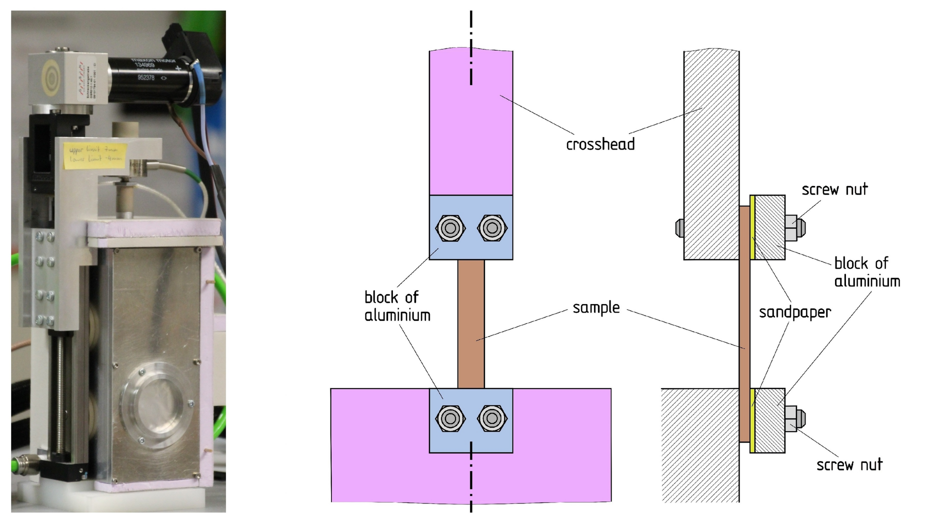
A Modular Humidity Generator (MHG-32 by pro|umid, Ulm, Germany) was used as the source of air humidity.



Air from a compressed air tank gets split up in the control unit. One part enters the chamber unmodified, the other part gets humidified by being blown through a damp sponge in the humidifier mixer.
By controlling vents (V1, V2), the control unit can set a specific wet-to-dry flow ratio and thus adjust humidity inside the chamber. A humidity sensor (%RH) inside the chamber is connected to the control unit, which calculates the wet-to-dry flow ratio needed to reach a target humidity in a closed-loop process.

Load Frame

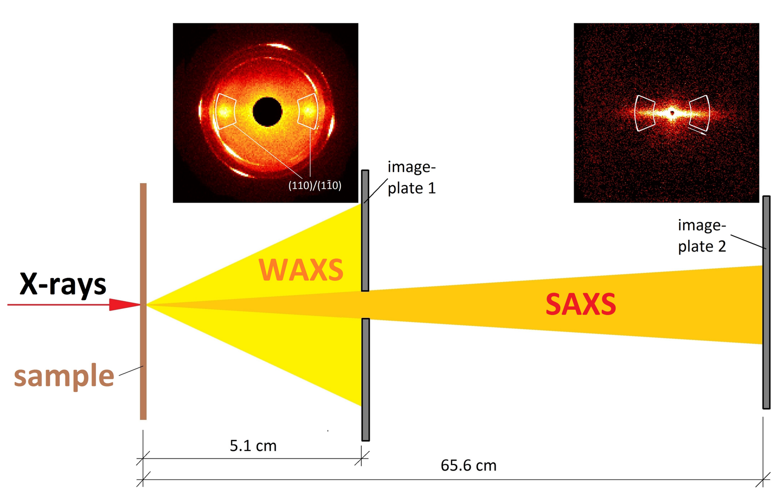
A motor on top of the apparatus drives a threaded rod, which moves the crosshead. Exact displacement of the crosshead is given by the number of rotations of the motor and the pitch of the threaded rod. A load cell is attached to the crosshead and connected to a computer that also controls the motor. An extension of the crosshead is connected to the other end of the load cell and plugged through the top of the chamber.



Picture of complete chamber (left),
schematic view of load frame inside the chamber (right)

X-ray Scattering

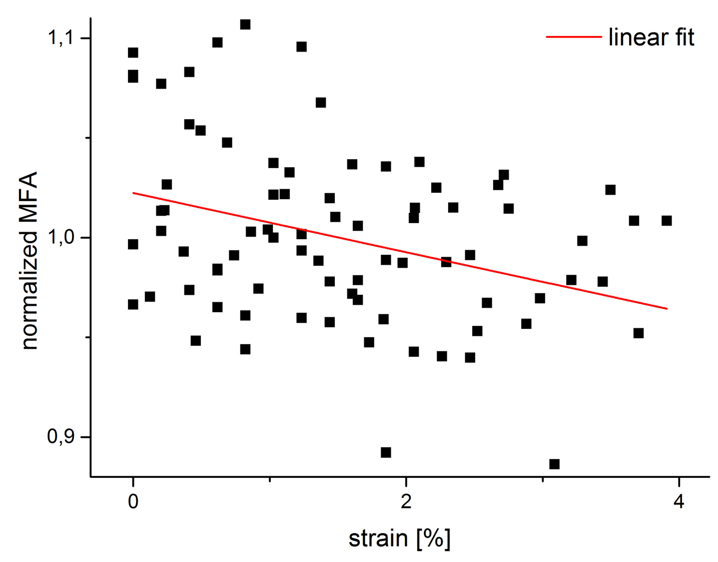
Cu-K$_\alpha$ X-rays with a wavelength of $\lambda = 0.1542$ nm were used in the X-ray scattering experiments.
SAXS covers scattering angles $2\theta \lesssim 4°$, while WAXS covers scattering angles of up to $2\theta \approx 30°$. Bigger scattering angles (in reciprocal space) correspond to smaller objects (in real space), i.e. WAXS can resolve structures with sizes $d \approx \lambda$ whereas SAXS measures objects with dimensions $d \gg \lambda$.
In WAXS measurements two peaks at $2\theta \approx 15.8°$ were observed, which are $(110)/(1\bar{1}0)$ diffraction peaks [3]; four peaks further from the center are caused by the aluminium windows.
SAXS patterns show a horizontal spread of intensity, which corresponds to vertical objects (in this case microfibrils) in real space.



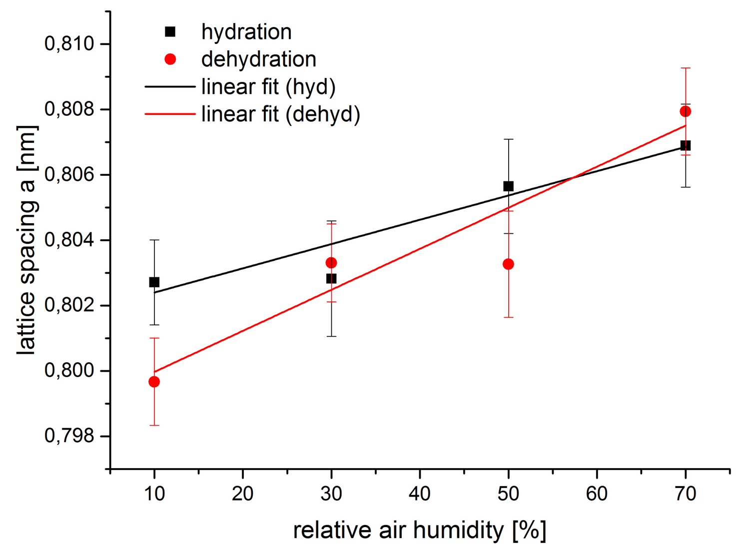
Simultaneous WAXS and SAXS measurement

The MFA was calculated by radial integration of the SAXS pictures and evaluation of FWHM of the resulting peaks.
The lattice spacing a was evaluated from the diffraction peaks in the WAXS intensity using the approximations $a \approx b$ and $\gamma = 90°$.

Results and Conclusion



Dependence of normalized microfibril-angle on strain



Dependence of lattice spacing a on relative air humidity

It turned out that laboratory X-ray intensities are not sufficient to measure a dependence on strain as successfully reported from synchrotron experiments [2].
However, the influence of humidity could be clearly detected: An increasing lattice spacing a of crystalline cellulose was observed and can be explained with swelling of fibrils by absorption of water.

[1] https://commons.wikimedia.org/w/index.php?title=File:Zellwandmodell_Druckholz.jpg&oldid=136408014&uselang=de
[2] Keckes J., Burgert I., Müller M., Kölln K., Hamilton M., Burghammer M., Roth S.V., Stanzl-Tschegg S.E. and Fratzl P. - In-situ WAXS Studies of Structural Changes in Wood Foils and in Individual Wood Cells During Microtensile Tests. Fibre Diffraction Review 13, 48 - 51, 2005.
[3] Zabler S., Paris O., Burgert I., Fratzl P. - Moisture changes in the plant cell wall force cellulose crystallites to deform. J. Struct. Biol. 171 (2010) 133-141.