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

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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. Nevertheless, 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 load and 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 strength 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 with 2.5 nm diameter and a 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 thickness. In these layers microfibrils wind round the lumen in a 2-helix-shape; the pitch of the helices differs in every layer.

Humidity and Tensile Test Chamber

Leak-proof Heated Chamber

A box with inner dimensions of 12 × 4.8 × 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.

Humidity Source

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

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.

X-ray Scattering

Cu-Kα X-rays with a wavelength of λ = 0.1542 nm were used in the X-ray scattering experiments. SAXS covers scattering angles 2θ ≤ 4°, whereas WAXS covers scattering angles of up to 2θ = 30°. Bigger scattering angles (in reciprocal space) correspond to smaller objects (in real space), i.e. WAXS can resolve structures with sizes d ≈ λ whereas SAXS measures objects with dimensions d > λ.

In WAXS measurements two peaks at 2θ = 15.8° were observed, which are (110)/(110) diffraction peaks. The first 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.

Results and Conclusion

It turned out that laboratory X-ray intensities are not sufficient to measure a dependence on strain as successfully reported from synchrotron experiments. 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.