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NANOINDENTATION TO STUDY WITHIN-TREE VARIABILITY OF WOOD CELL WALL STIFFNESS

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Motivation

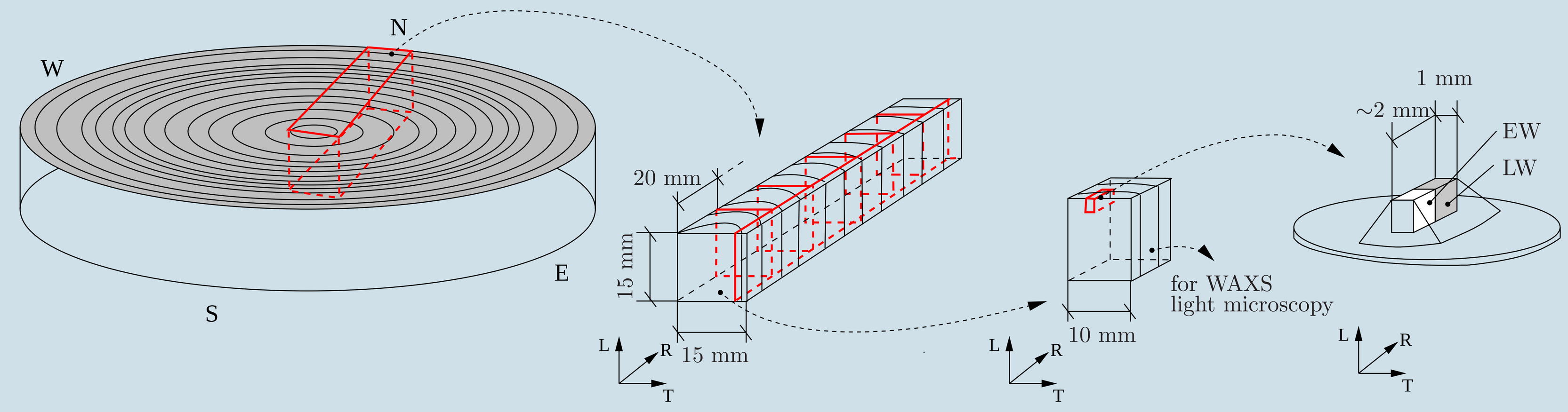
Microstructural variability

Wood is not a man-made material, it cannot be designed for purpose and does not exhibit uniform properties. Aside knots and other growth irregularities, distinct variability patterns of wood microstructure can be observed within a single tree. The mechanical properties of a solid piece of wood depend on its microstructural characteristics and can be described using structure –function relationships. The mass density and the microfibril angle (MFA) of the S2 cell wall layer dominantly influence the macroscopic material behavior of clear wood. Variability patterns of these microstructural characteristics within a tree occur at different length scales and in different anatomical directions, e.g. from the base to the apex (longitudinal direction), from pith to bark and from earlywood (EW) to latewood (LW) (radial direction).

Herein, we focus on the variability of the cell wall stiffness and investigate samples from pith to bark, as well as from EW to LW within selected annual rings [1]. Besides a minor influence of the local chemical composition, MFA remains the only micro-structural parameter of interest. Thus, it is determined for specific annual rings by wide angle X-ray scattering (WAXS) and across selected annual rings by light microscopy [3]. To study the mechanical properties of wood cell walls, exhibiting typical thicknesses of 2-5 μm, micromechanical experiments are conducted. Nanoindentation (NI) can provide such local measurements of mechanical properties.

Material and sampling

Four Scots pine (*Pinus sylvestris* [L.]) trees, originating from four different plantation sites across Scotland, were harvested. Sampling discs were cut from each tree at breast height. The north-facing radial sections were then cut into consecutive cubical samples. From selected annual rings, small pieces of wood (~1.5x2x1 mm³, LxRxT) containing both earlywood and latewood of that specific annual ring were prepared for nanoindentation.

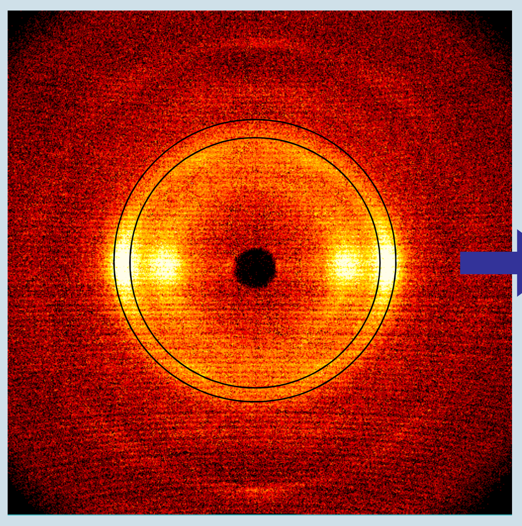


Sampling methodology: sampling disc > radial section > cubical sample > NI-specimen

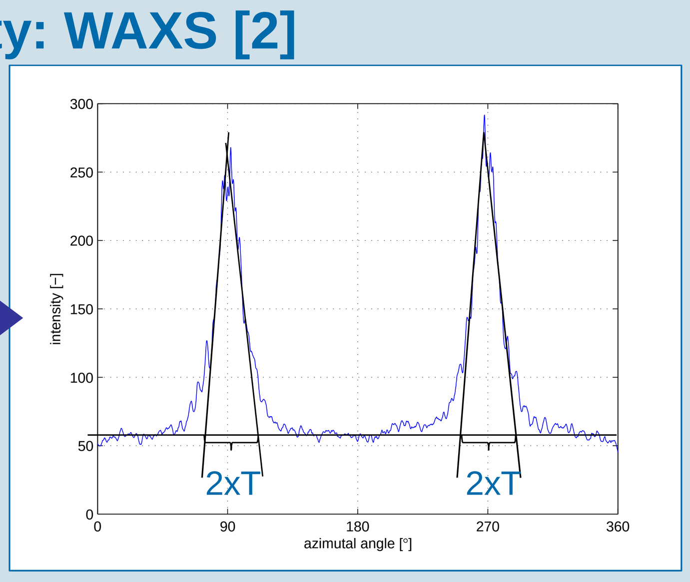
Experimental Methods: MFA Determination and Nanoindentation

MFA Determination

Pith-to-bark variability: WAXS [2]



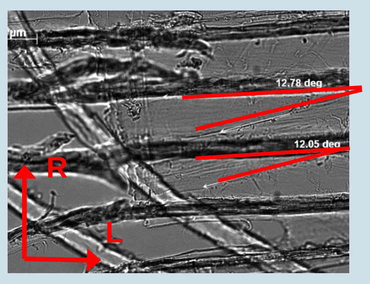
WAXS pattern



integrated intensity of the 002 reflection

MFA=0.6xT → *EW/LW-specific MFA for each investigated annual ring*

EW to LW variability: Light microscopy [3]



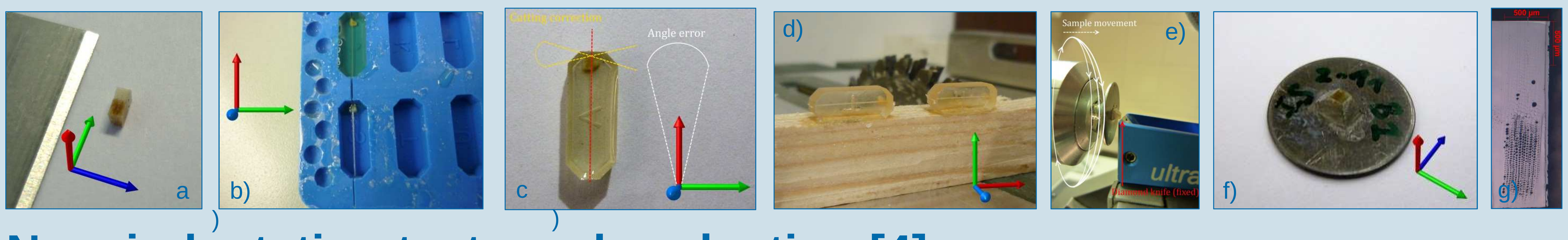
MFA on a thin section

MFA is measured directly on thin sections, where aligned cracks in the S2 cell wall layer induced by harsh drying are aligned in parallel to cellulose fibrils.

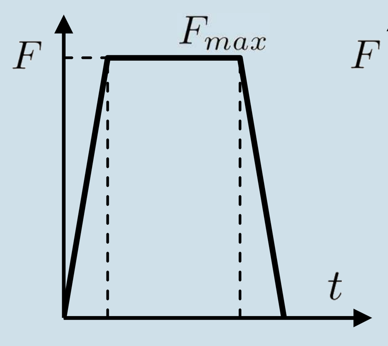
→ *MFA profile across one annual ring*

Nanoindentation – Sample preparation

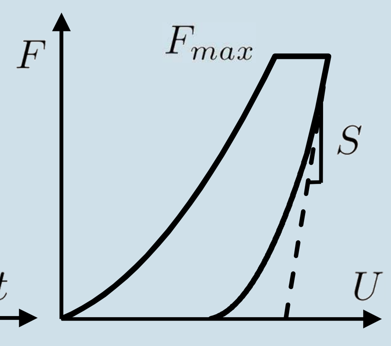
Small wood pieces (a) were embedded in an epoxy resin (b), checked for their alignment with respect to the fiber axis (c) and then cut from the resin block using a double blade circular saw (d). Then the embedded specimens were glued onto steel discs in order to cut a smooth cross sectional surface using a rotatory microtome, equipped with a diamond knife (e). The sample was then clamped to the magnetic stage of the Hysitron TriboIndenter® for testing (f). The final smooth cross section exhibited EW and LW cells of a whole annual ring (g).



Nanoindentation tests and evaluation [4]



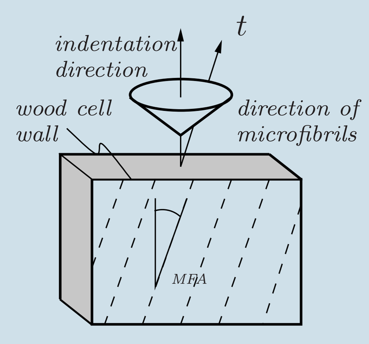
load history



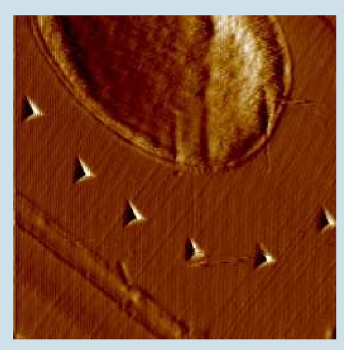
load-penetration curve

$$S = \partial F / \partial U \,|_{U=U_{max}} \qquad M = \frac{\sqrt{\pi}\, S}{2\sqrt{A_C}}$$

A_C… contact area
M… indentation modulus
S… initial unloading stiffness
F… load U… penetration depth



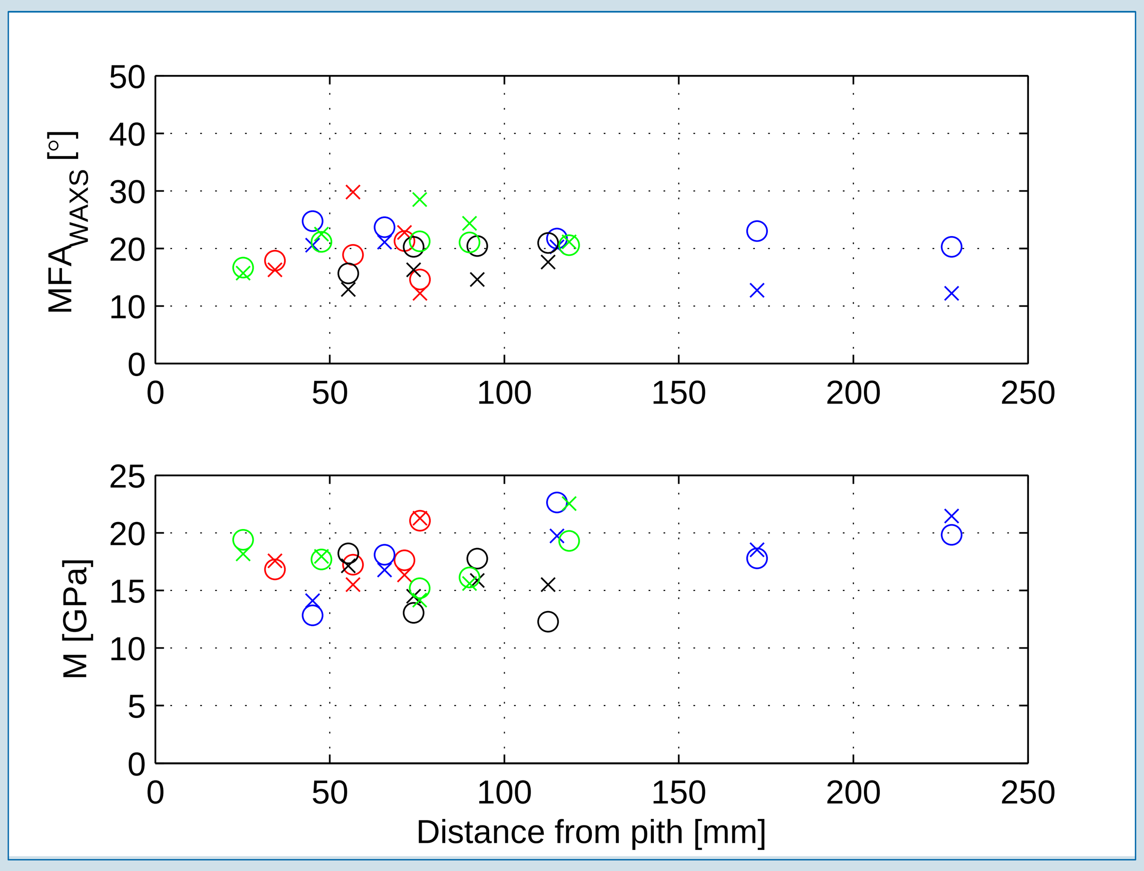
indentation in the anisotropic wood cell wall



remaining imprints on the wood cell wall

$M = f(\text{elastic constants of cell wall material}, \boldsymbol{MFA}) \rightarrow$ **no pure material property**

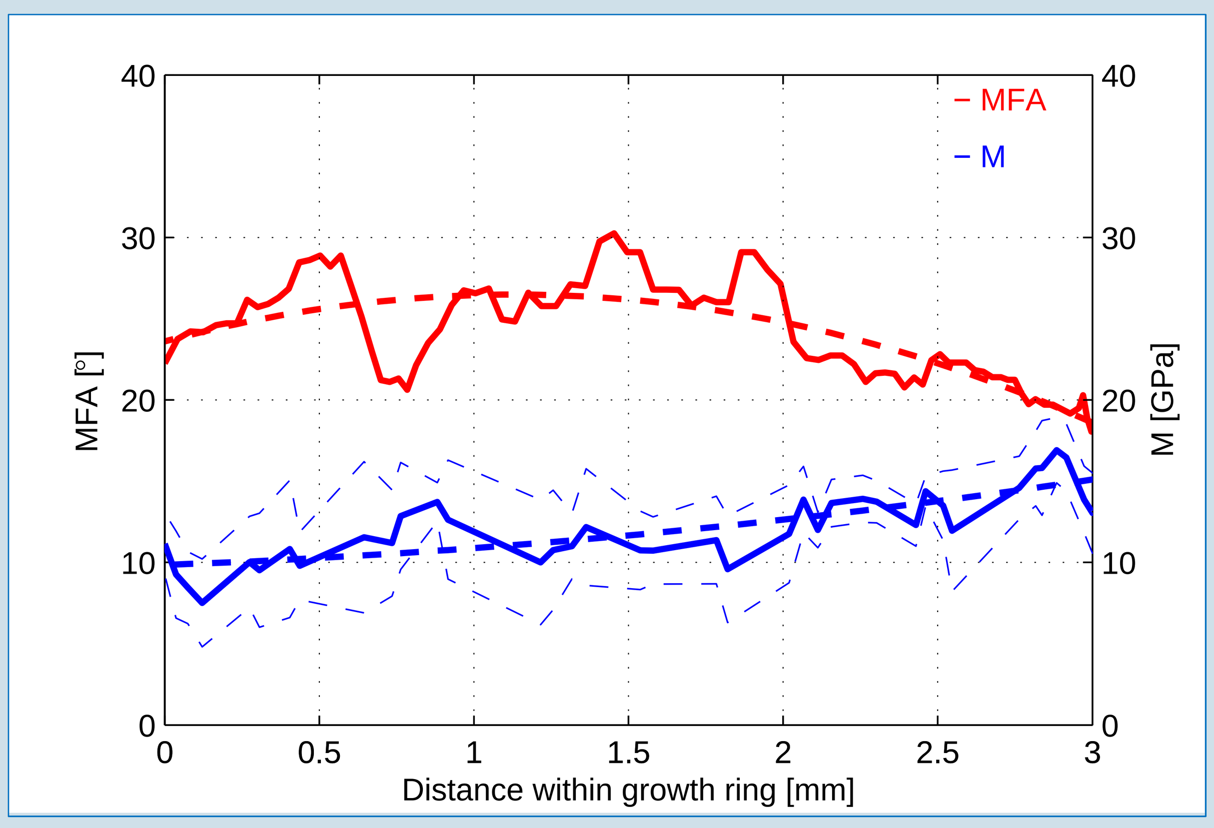
Results & Discussion



MFA (WAXS) and M plotted from pith to bark, o…EW/x…LW

EW and LW-specific indentation moduli M and the corresponding MFA, determined by means of WAXS, are shown. Known trends of decreasing MFA and, in consequence increasing indentation moduli, from pith to bark can not be observed in the four investigated trees. This may result from the nature of the WAXS measurement, giving an average MFA of 30 to 50 cells only for EW and LW, respectively.

Also M yields a measure of the cell wall stiffness of the indented cells only. Such local measures of MFA and M are susceptible to fluctuations within one annual ring or within single cells, which may override trends on a larger length scale, e.g. the known MFA decrease from pith to bark. Despite the local fluctuations, the dependence of M on the MFA is visible in terms of a decreasing M with increasing MFA.



MFA (light microscopy) and M plotted from EW to LW

Stepping down one length scale, profiles of M and MFA across selected growth rings are presented. Here, trends from EW to LW become obvious, namely a decrease of MFA from EW to LW and a corresponding increase of M. The detected trends over annual rings are again overlaid by pronounced local variations of both MFA and M. Local maxima and minima of M can be associated with local minima and maxima of MFA.

Due to the anisotropy of the cell wall, M is not a pure material property, but crucially depends on the MFA in addition to the elastic constants of the cell wall material, which can be accounted for by anisotropic indentation theory [5].

References:
[1] Wagner et al. (2012), accepted by *Trees-Struct Funct*
[2] Cave (1966), *For Prod J* 16(10):37-42
[3] Senft&Bendtsen (1985), *Wood Fiber Sci* 17(4):564-567
[4] Oliver&Pharr (1992), *J Mat Research* 7(6):1564-1583
[5] Jäger et al. (2011), *Comp A* 42:677-685

29th DANUBIA-ADRIA SYMPOSIUM on Advances in Experimental Mechanics, September 26-29, Belgrade, Serbia