X-Ray microdiffraction on cellulose fibres and wood under tensile stress

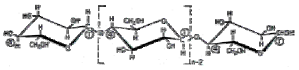
Klaas Kölln[1], Claas Behrend[1],
Ingo Grotkopp[1], Martin Müller[1]*,
Martin Dommach[2], Sergio S. Funari[2],
Manfred Burghammer[3],
Stefan V. Roth[3]

[1] *Institut für Experimentelle und Angewandte Physik,*
Christian-Albrechts-Universität zu Kiel, D-24098 Kiel
[2] *Max-Planck-Institut für Kolloid- und Grenzflächenforschung,*
D-14424 Potsdam c/o HASYLAB Hamburg
[3] *ID13, ESRF, F-38043 Grenoble*

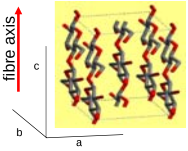
* mmueller@physik.uni-kiel.de; http://www.ieap.uni-kiel.de/solid/ag-mueller

Cellulose – a fibrous biopolymer

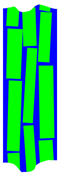
The base unit: β-1,4 linked glucose rings.



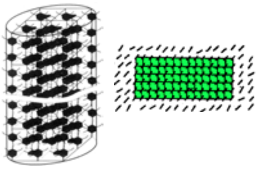
Cellulose fibres consist of crystalline parts surrounded by disordered regions.



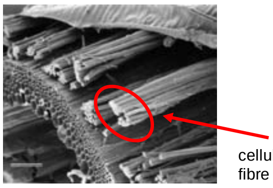
monoclinic unit cell
($0.80 \times 0.82 \times 1.03$ nm^3)



The crystalline unit: microfibrils
- ∅ 2 - 20 nm
- length of several 100nm



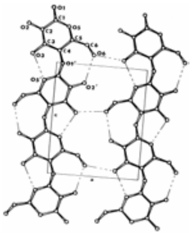
Flax fibres –
an example of highly oriented cellulose



cellulose
fibre bundle

flax stem   fibre diameter: 20 µm

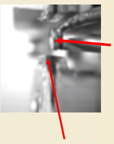
The mechanical properties of cellulose are influenced by
- hydrogen bonds (different for cellulose I - IV) and
- microfibril size and orientation (varies for different species).



hydrogen bonds
of cellulose I

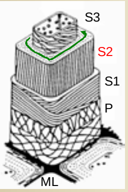
In-situ stretching experiments
X-ray microdiffraction at beamline ID13

piezo stretching cell for
in situ X-ray scattering on single fibres



The stretching apparatus of beamline ID13. It makes the simultaneous measurement of the macroscopic stress-strain relationship of the fibre and the straining of the microfibrils using X-ray diffraction possible.

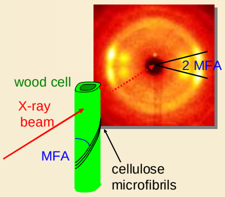
Micro- and nanostructure of softwood cell walls



- ML: middle lamella
- P: primary cell wall
- S1…S3: secondary cell walls

Softwood cells have a diameter of roughly 50 µm and a length of several mm. The cell wall is a composite material of cellulose and matrix materials such as lignin and hemicellulose displaying unique mechanical properties.

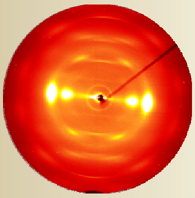
The microfibril angle (MFA)
A key parameter for the mechanical properties of wood cells.



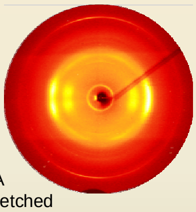
Norway spruce wood

latewood, low MFA, **wet**

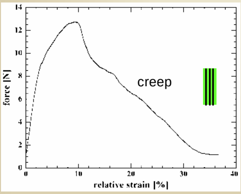
earlywood, high MFA, **dry**

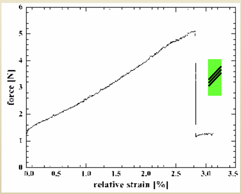


- high extensibility
- decreased Young's modulus
- MFA decreases
- microfibrils are *not* stretched



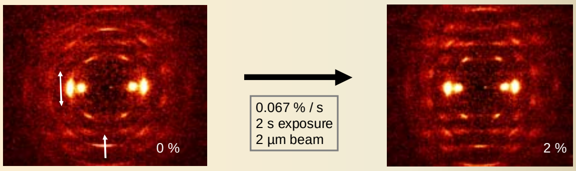
- low extensibility
- high stiffness
- *no change* of MFA
- microfibrils are stretched

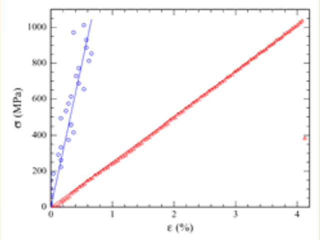




What are the different mechanisms on the *nanoscale* that govern the mechanical properties of dry and wet wood?

Experiments on single flax fibres



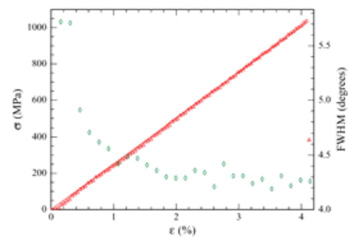


Stress-strain curves
of the microfibrils and the fibre

- The microfibril stress-strain relation is calculated from the shift of the 004 reflection.
- The fibre and the microfibrils show an elastic behavior.
- Their Youngs moduli differ by a factor of three to four.

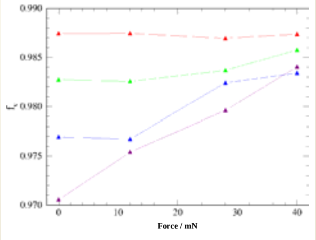
Rotation of microfibrils

- A rotational motion of the microfibrils leads to an increased orientational order (= alignment) with increasing strain.
- This ordering takes place during the initial phase of straining.



Mesh scan over a single fibre

- Hermans' orientation function for the microfibrils f_c is calculated from the width of the 200 reflection.
- The fiber shows well ordered and less ordered regions.
- The well ordered regions show no effect upon straining, while the less ordered regions show a significant improvement of f_c.



How do the rotation of the microfibrils and the straining of the crystal influence the mechanical properties of cellulose fibres?

Funding: DFG Project Pr 325/15-1
Project Mu 1673/2-3
HASYLAB Project II-01-054