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NANOINDENTATION OF WOOD CELL WALLS: EFFECTS OF DIFFERENT SAMPLE PREPARATION METHODS

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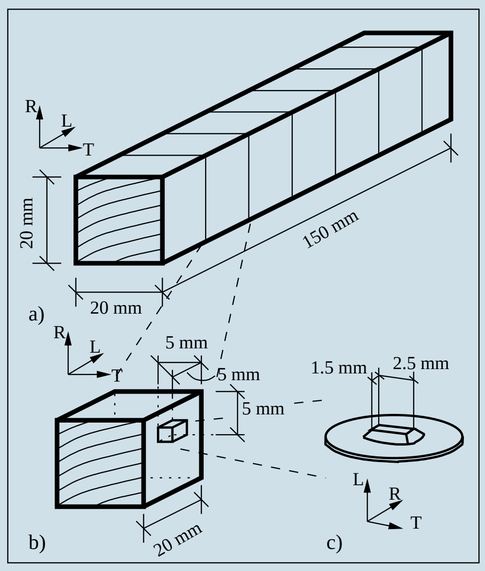
Motivation

Nanoindentation [1] has established itself as the method of choice when investigating mechanical properties of wood cell walls [2,3]. In addition to the S2 layer, also the middle lamella can be indented in the cell corners. In the cell corner middle lamella (CCML), proper indentation parameters have to be determined as compromise between the surface roughness and the size of the CCML, by indenting the CCML at different depths. Wood is commonly embedded into resin in order to stabilize the cell walls during microtome cutting [2,3,4]. It remains uncertain whether the resin penetrates the cell wall and, thus, influences the response of the material during nanoindentation. Herein, indentation properties from samples embedded into two different types of resins, as well as from testing apparently non-embedded wood cell walls, are presented. In addition, the exposure of the samples to heat either during the embedding process itself or prior to the sample preparation, e.g. during oven drying, may affect the resulting material properties. Temperatures during these procedures might exceed the glass transition temperatures of the wood polymers [5]. Also cracks in the cell walls may occur upon drying, influencing the measured quantities. Test results are presented for samples having undergone multiple harsh drying cycles from a fully water saturated state, in order to identify potential effects of sample heating on the measured indentation modulus and the hardness of wood cell walls.

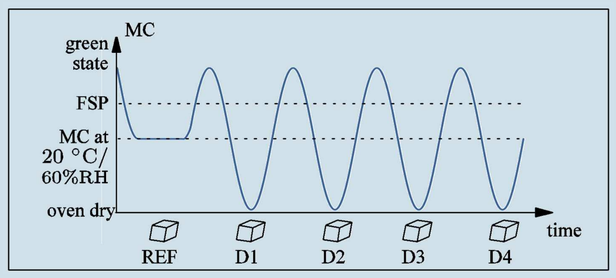
Material and Experimental Methods

Material and Sampling



A piece of Norway spruce (*Picea abies* [L] Karst.) wood (a) was cut in five cubes (b). After four of these cubes faced further processing, specimens for nano-indentation were pre-pared (c) from all five cubes.

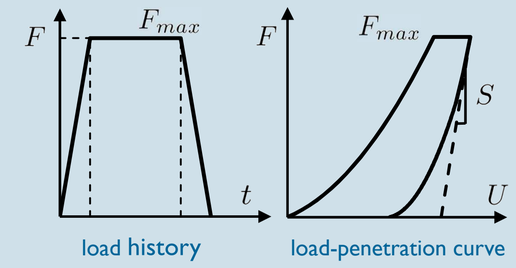
Sample Processing and Preparation



Four cubes were subjected to repeated wetting and drying cycles resulting in a series of 1 to 4 cycles (D1 to D4) next to the untreated reference (REF).

NI-specimens were prepared from each cube using the standard sample preparation procedure [3]. In order to study the influence of the embedding material, modified procedures were used to prepare two additional samples: one was embedded in a different resin curing, at room temperature (E1), while a second one was not subjected to vacuum during embedding (E2) [3]. Indentation parameters for the CCML were identified using the REF NI-specimen.

Nanoindentation Tests



$$S = \partial F / \partial U \,|_{U=U_{max}}$$

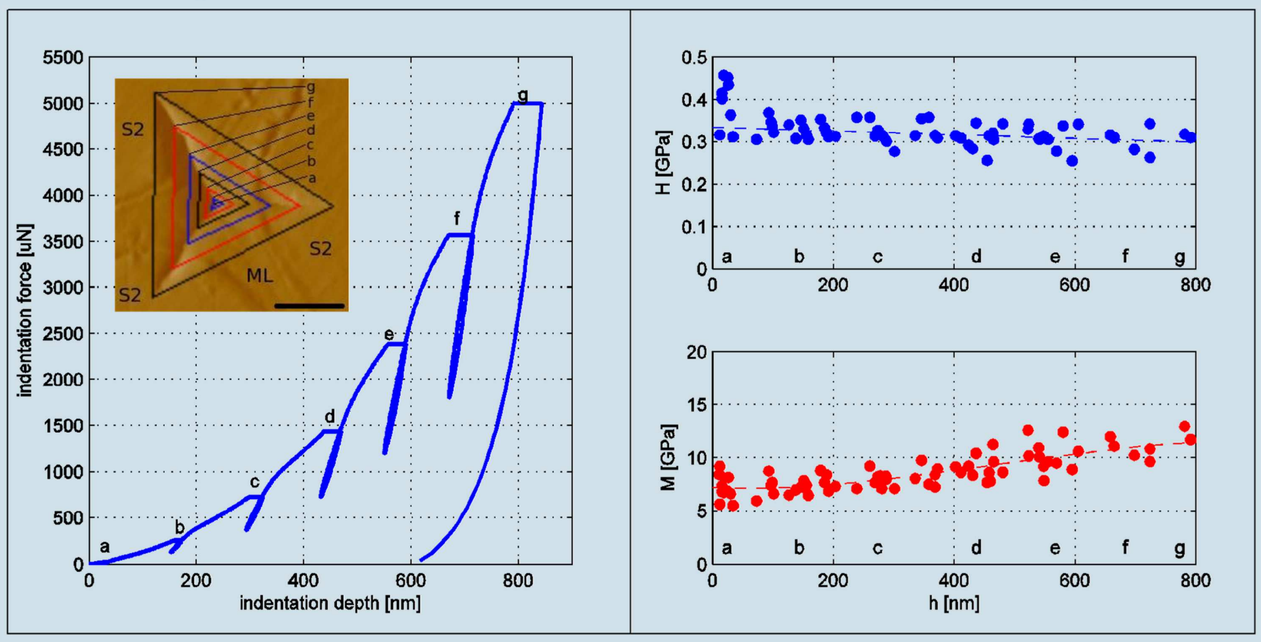
$$M = \frac{\sqrt{\pi}\, S}{2\sqrt{A_C}}$$

$$H = \frac{F_{max}}{A_C}$$

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

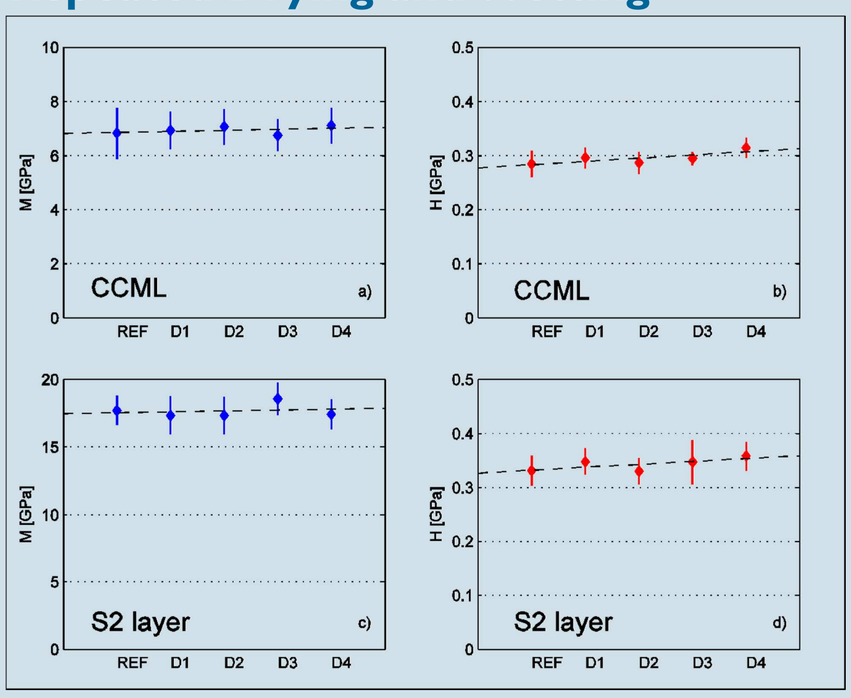
Results & Discussion

Indenting the Cell Corner Middle Lamella (CCML)



H in CCML showed a slight overall decrease, which did not become significant ($p>0.01$). M remained at the same level until indentation depths h of ~250-300 nm and then showed an increase with higher indentation depths. Consequently, suitable indentation depths would lie in the range of 150-180 nm.

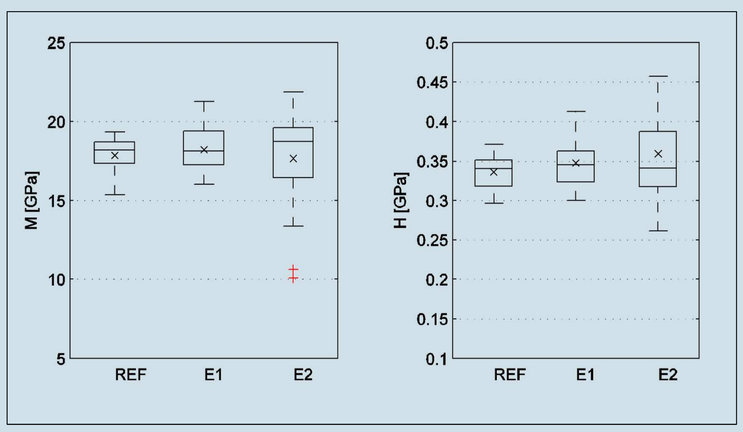
Repeated Drying and Wetting

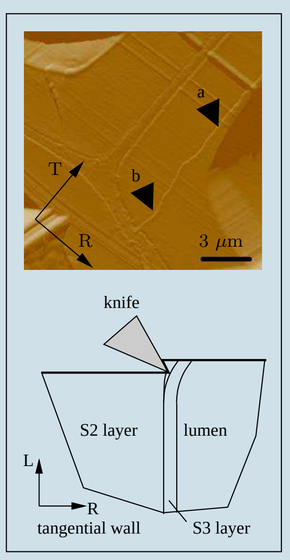


After repeated drying, losses of dry mass occurred, while the EMC did not change. Thus, it can be assumed that no carbohydrates were lost during repeated wetting and drying. No significant changes of M in the S2 layer and in the CCML were observed ($p>0.01$), while H slightly, but also not significantly ($p>0.01$), increased in both the S2 layer and the CCML. An extraction of soluble agents during the repeated wetting might explain this increase, as these agents act as plasticizers in the cell walls.

Different Embedding Procedures

Embedding in a different resin (E1) did not result in significantly different M and H ($p>0.01$). Neither the different mechanical properties nor the different curing temperatures had an effect on M and H. The tests on the non-embedded cell walls (E2) also did not show significantly different ($p>0.01$) M and H. However, the variability (SD) increased, compared to the reference values, by a factor of 3 and 2 for M and H, respectively. This higher experimental scatter might result from the sample preparation process, inducing cracks and delaminations in the cell walls.





References:

[1] Oliver&Pharr (1992), *J Mat Res* 7(6):1564-1583
[2] Wimmer et al. (1997), *IAWA J*, 18(1):77-88
[3] Wagner et al. (2013a), *Trees-Struct Funct* 27:321-336
[4] Wagner et al. (2013b), *J Mat Sci* doi:10.1007/s10853-013-7680-3
[5] Irvine (1984) *TAPPI J*, 67:118-121

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