

# Influence of Drying on the Micromechanical Properties of Wood

Z. Prošek<sup>1,\*</sup>, J. Topič<sup>1</sup>, P. Tesárek<sup>1</sup>, V. Nežerka<sup>1</sup>, V. Králík<sup>1</sup>

<sup>1</sup> CTU in Prague, Faculty of Civil Engineering, Thákurova 7, 166 29 Praha 6, Czech Republic

\* zdenek.prosek@fsv.cvut.cz

**Abstract:** This paper discusses the influence of drying on the micromechanical properties of spruce wood cells. Cell wall is composed of several layers, which are for their small size difficult to characterize, and therefore nanoindentation had to be employed for that purpose. It was found that the indentation stiffness of the cells walls ranges between 16.17 GPa for naturally dried wood samples and 13.84 GPa when artificially dried. The difference between the elastic stiffness of natural and artificial drying samples is attributed to the formation of nanoscale cracking.

**Keywords:** Spruce Wood; Nanoindentation; Artificial Drying; Latewood.

## 1 Introduction

Wood is one of the oldest building materials, used in the form of structural timber. Nowadays its popularity is still increasing even in developed countries, because it is renewable material having a relatively high strength to weight ratio. Before using this material for construction, wood should be dried to reach 12 % moisture content. The drying results in higher strength and resistance to wood-decaying fungi, insects and molds. In the past, wood was naturally dried for several months or even years, while today it is dried artificially using a hot air drying, microwave drying, chemical drying or vacuum drying, mainly for economic reasons. It is well known that artificial drying of wood has a negative impact on the macrostructure because of formation of drying cracks and therefore it cannot be used e.g. in aerospace industry [1].

The presented work examines the effect of artificial drying on micromechanical properties of individual wood cells, which was investigated by means of nanoindentation. This technique was first used by Wimmer et al. [2, 3] who managed to determine the value of Young's modulus and hardness, equal to 13.5 GPa and 0.25 GPa for the spruce earlywood samples, respectively. They found that latewood Young's modulus and hardness were higher, 21.0 GPa and 0.34 GPa, respectively. In the following study by Gindl and Gupta [4] nanoindentation was successfully used to compare plain and melamine-modified spruce wood. Quite recently the nanoindentation dynamic method (nanoDMA) was successfully employed to analyze mechanical properties of spruce wood [5] and the comparison with statically measured data is provided in the overview by Prošek et al. [6].

## 2 Material and Samples

We focused on the properties of spruce wood, when one sample was artificially dried using and other one dried in natural conditions. First sample had been artificially dried at 100 °C using hot air drying kilns with controlled air humidity. A second sample was naturally dried by air a temperature of 23 °C. Both 10 × 10 mm samples were of a cross section perpendicular to the grain of size were prepared to study the microstructure at micro- and nano-scale. Consequently, the samples were submerged to epoxy resin (Struers Epofix Kit). After hardening of the epoxy, the samples were cut into individual slices and then grinded in several steps to achieve the best possible quality of the sample surface. For the first step, the silica paper with grit 800 grain/cm<sup>2</sup> was used to remove the greatest inequality after cutting. In the other steps finer silica papers were used: 1200 grain/cm<sup>2</sup> for 3 minutes, 2400 grain/cm<sup>2</sup> for 7 minutes and 4000 grain/cm<sup>2</sup> for 7 minutes. The whole process of grinding was done under water. In the last step of grinding the emulsion containing nanodiamonds with size of 0.25 micron was used for 15 minutes. After each step, the sample was cleaned in an ultrasonic bath submerged in distilled water. When the sample, thus prepared, reached sufficient quality of surface for the next measurement.

### 3 Experimental Methods

Optical microscopy (Carl Zeiss Axio Imager M2m) was used for the mapping of the wood microstructure and description of the cell phases. Nanoindentation was accomplished using Hysitron Tribolab device, located at the Faculty of Mechanical Engineering at the Czech Technical University in Prague. Elastic properties of the investigated wood cells were evaluated using an indentation dynamic method continuous stiffness measurements (CSM). The CSM is accomplished by imposing a harmonic force, which is added to the nominally increasing load onto the indenter tip. The displacement response of the indenter at the excitation frequency and the phase angle between the two are measured continuously as a function of depth. Solving for the in-phase and out-of-phase portions of the response results in an explicit determination of the contact stiffness, as a continuous function of depth. From the recorded displacement amplitude and phase lag are determined the storage and loss modulus [7].

### 4 Results and Discussion

The study was focused on the cells of latewood tracheids, in particular secondary cell wall and composite middle lamella was tested. Latewood tissue has the greatest influence on the final strength of wood, because as clearly visible, the latewood cells are thick-walled, having the wall thickness between 7 and 12  $\mu\text{m}$  and containing a small lumen.

To obtain representative results, each type of wood was indented at 11 distinct locations. In order to avoid any interaction between individual indents, their minimum spacing was set to 3  $\mu\text{m}$ . Fig. 1 illustrates the tested location within the microstructure, while positions of individual indents appear in Fig. 2. Representative cells were selected for the testing from the two materials, which had approximately the same dimensions and the same shape.

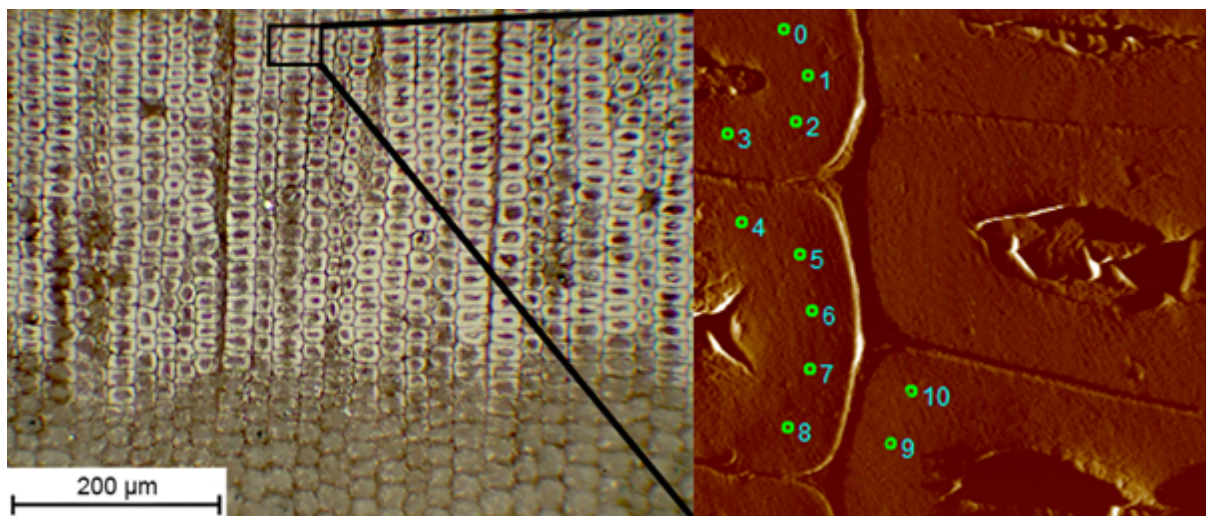


Fig. 1: Microstructure of natural dried wood with testing position by indentation.

Indentation (CSM) was accomplished by applying a small oscillation to the force signal at frequency of 80 Hz. The average amplitude of the force oscillation was 60  $\mu\text{N}$  and the nominal contact force was 160  $\mu\text{N}$ . The average penetration depth was equal to 100 nm which sufficient with respect to the surface roughness, but not too large to avoid interaction between phases of wood cells. The mean deviation of the surface roughness of the tested samples was lower than 30 nm. With respect to the negligible viscosity, indicated by the relatively small magnitude of the measured loss modulus, the values of storage moduli can be considered as the reduced elastic stiffness modulus. The mean values of indentation modulus and hardness with indicators of standard deviation are presented in Fig. 3.

The values seem reasonable with respect to the effective properties of spruce wood, published in available literature [2, 6]. The difference between the elastic stiffness of naturally dried wood and artificially dried samples, in particular in the stiffness of their cell walls, is attributed to the rapid loss of water from the cell walls when artificially dried, which results in formation of nanoscale cracks. The nano-cracks were not visible under

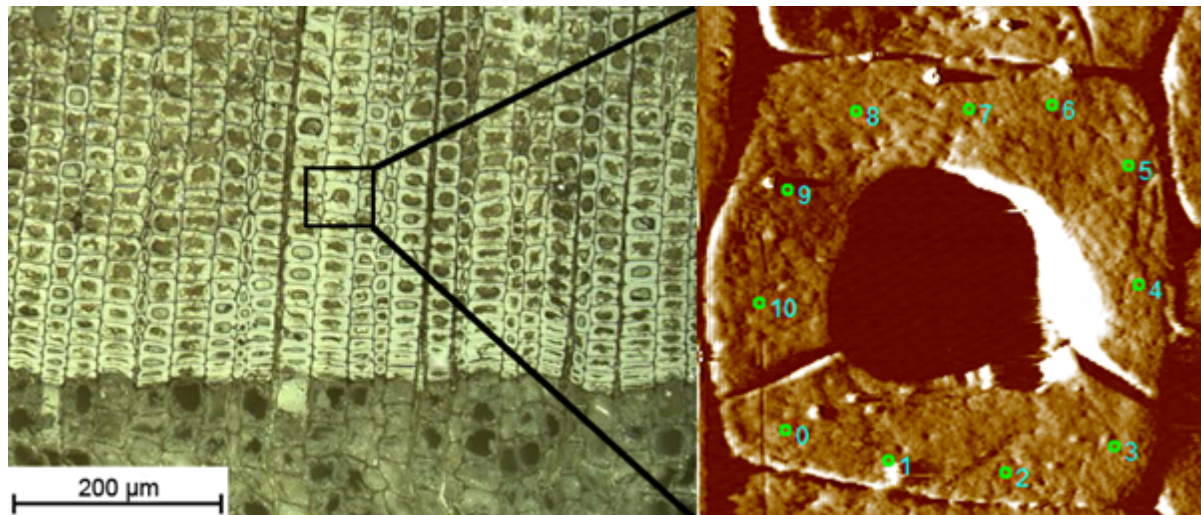


Fig. 2: Microstructure of artificial dried wood with testing position by indentation.

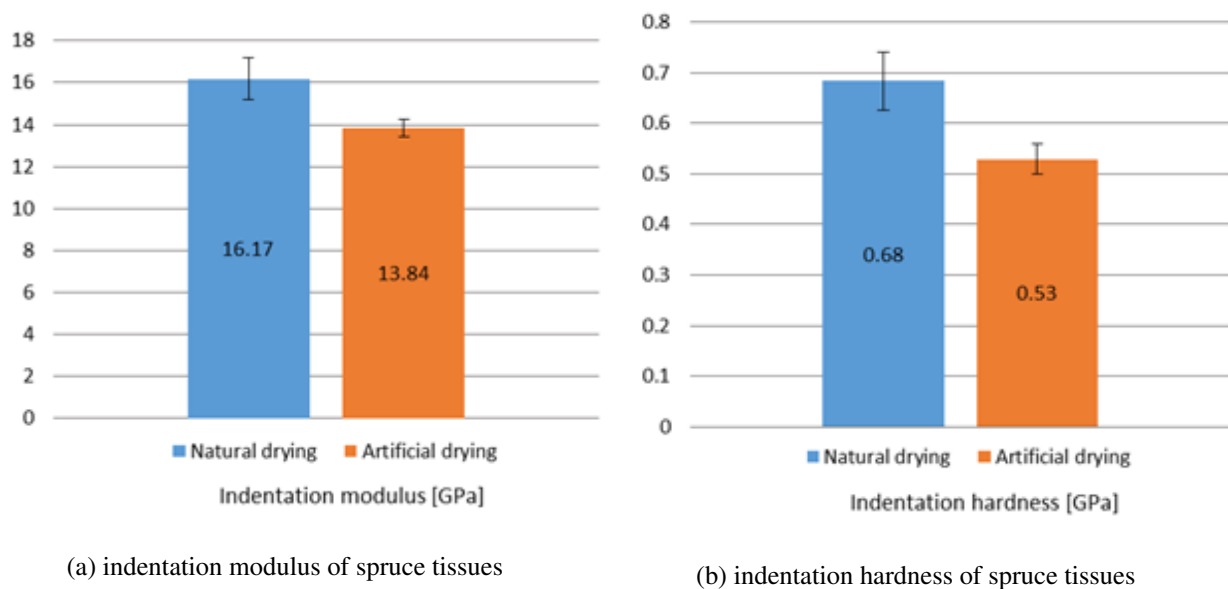


Fig. 3: Micromechanical properties of spruce tissues natural and artificial drying wood.

an optical microscope, but they have quite significant impact on the reduction of elastic modulus. Electron microscopy, allowing higher magnification (more than 1000 $\times$ ), could be used for detection and mapping of such nanoscale cracking.

## 5 Conclusion

Results on naturally dried wood are in agreement with the data published by other authors [2, 6]. The difference between values obtained on naturally and artificially dried wood samples is about 15 % for indentation modulus and 22 % for indentation hardness. This difference is attributed to the rapid loss of water from the cell walls, resulting in formation of nanoscale cracking. Our future research will be focused on the testing of macroscopic properties by means of impulse excitation method, successfully exploited for investigation of timber elements e.g. by Klapálek et al. [8].

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## References

- [1] P. O. Kettunen, *Wood Structure and Properties*, Tampere, Trans Tech Publications, 2006.
- [2] R. Wimmer et al., Longitudinal Hardness and Elastic Stiffness of Spruce Tracheid Secondary Walls Using Nanoindentation Technique, *Wood Science and Technology* 31 (1997) 31–141, doi: [10.1007/BF00705928](https://doi.org/10.1007/BF00705928).
- [3] R. Wimmer et al., Comparing Mechanical Properties of Secondary Wall and Cell Corner Middle Lamella in Spruce Wood, *IAWA* 18 (1997) 77–88.
- [4] W. Gindla, H. S. Gupta, Cell-wall Hardness and Young's Modulus of Melamine-Modified Spruce Wood by Nano-indentation, *Composites: Part A* 33 (2002) 1141–1145, doi: [10.1016/S1359-835X\(02\)00080-5](https://doi.org/10.1016/S1359-835X(02)00080-5).
- [5] Z. Prošek et al., Microstructure Description and Micromechanical Properties of Spruce Wood, *Acta Polytechnica* 55 (2015) 39–49, doi: [10.14311/AP.2015.55.0039](https://doi.org/10.14311/AP.2015.55.0039).
- [6] Z. Prošek et al., Micromechanical Properties of Spruce Tissues Using Static Nanoindentation and Modulus Mapping, *Applied Mechanics and Materials* 732 (2015) 115–118, doi: [10.4028/www.scientific.net/AMM.732.115](https://doi.org/10.4028/www.scientific.net/AMM.732.115).
- [7] L. Xiaodong, B. Bharat, A review of nanoindentation continuous stiffness measurement technique and its applications, *Materials Characterization* 48 (2002) 11–36, doi: [10.1016/S1044-5803\(02\)00192-4](https://doi.org/10.1016/S1044-5803(02)00192-4).
- [8] P. Klapálek, L. Melzerová, Methods of non-destructive assessment of timber, *Applied Mechanics and Materials* 732 (2015) 369–372, doi: [10.4028/www.scientific.net/AMM.732.369](https://doi.org/10.4028/www.scientific.net/AMM.732.369).