EAN 2014
52nd conference on experimental stress analysis

Micromechanical Properties of Spruce Tissues Using Static Nanoindentation and Modulus Mapping

PROŠEK Zdeněk[1,a], TOPIČ Jaroslav[1,b], TESÁREK Pavel[1,c], INDROVÁ Kateřina[1,2,d], NEŽERKA Václav[1,e], KRÁLIK Vlastimil[1,f] and KLAPÁLEK Pavel[1,g]

[1]CTU in Prague, Faculty of Civil Engineering, Thákurova 7, 166 29 Praha 6, Czech Republic

[2]CTU in Prague, Faculty of Biomedical Engineering, nám. Sítná 3105, 272 01 Kladno 2, Czech Republic

[a]zdenek.prosek@fsv.cvut.cz, [b]jaroslav.topic@fsv.cvut.cz, [c]tesarek@fsv.cvut.cz, [d]infrova.katerina@fbim.cvut.cz, [e]vaclav.nezerka@fsv.cvut.cz, [f]vlastimil.kralik@fsv.cvut.cz, [g]pavel.klapalek@fsv.cvut.cz

Keywords: Atomic force microscopy, static nanoindentation, modulus mapping, Norway spruce.

Abstract. Cell wall of Norway spruce is composed of several layers, which is, due to their small size, difficult to characterize. For this reason, the work uses a combination of methods, atomic force microscopy (AFM) is used to determine the topography of a sample and nanoindentation has been used to locally characterize the surface mechanical properties of earlywood tracheids. Additionally, differences in the mechanical properties of cells wall phase were successfully characterized as a function of surface morphology by using a high resolution modulus mapping technique. Mechanical properties of spruce wood are in fair agreement with literature data.

Introduction

Structure of spruce wood is composed of two types of cells: tracheids and parenchymal cells, which are oriented in the axial direction. Tracheids have the largest effect on the resulting mechanical and physical properties of wood. Tracheids makes up to 95% of the entire volume of the wood. The cell walls of tracheid consist of layers with different spiral angle of cellulose microfibrils. Depending on these angles, the cell in the direction from the periphery to the center of the cell, divides on: middle lamella, primary wall and secondary wall which is composed of three layers [1].

Wimmer et al. [2] were the first ones who used the nanoindentation in the 1997 as a tool for the determination of the micromechanical properties of wood cell. They managed to determine the value of Young's modulus to 13.49 ± 5.75 GPa and the hardness to 0.25 ± 0.07 GPa on the spruce earlywood samples. The Young's modulus of transition wood was equal to 21.27 ± 3.12 and the hardness to 0.29 ± 0.04 GPa. The Young's modulus of latewood was higher and equals to 21.0 ± 3.34 GPa, as well as the hardness that equals to 0.34 ± 0.03 GPa. Later on, Gindl and Gupta [3] successfully carried out the nanoindentation of a single wood cell, located at the interface between early and latewood tissues. They found that the value of Young's modulus was equal to 16.1 GPa and hardness to 0.24 GPa. The values of Young's modulus were measured on the secondary walls of tracheids using static nanoindentation method. The dynamic method (nanoDMA) is nanoindentation technique and

generally was successfully used for mechanical analysis of many bio-materials [4] (such as human trabeculae). Experimental studies of wood cells by using the dynamic method are still lacking.

Material and Samples

The study was focused on the cells of earlywood tracheids. The samples, which were used for examining the properties of spruce wood, were made of glue laminated timber. Spruce plates were dried at 100 °C, to the desired moisture content of 9 %, by using hot air. Samples of wood with a cross section of 10 × 10 mm, were cut on required directions and then sealed with epoxy 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^2 was used to remove the greatest inequality after cutting. In the other steps finer silica papers were used: 1200 grain /cm^2 for 3 minutes, 2400 grain /cm^2 for 7 minutes and 4000 grain /cm^2 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, the surface roughness was determined by AFM.

Experimental Methods

Optical microscopy (the metallurgical microscope NEOPHOT 21) was used at first for mapping of wood microstructure, followed by the atomic force microscope (AFM) for more precise description of the cell phases presented in the spruce wood. Atomic force microscopy uses probe rasterizing of the sample using sharp tip with a radius of curvature of 10 nm. Size of the area that has been scanned was 65 × 65 microns.

Modulus mapping technique combines nanoDMA (dynamic method analysis) and in-situ SPM (scanning probe microscopy) imaging to create unique characterization capabilities. NanoDMA allow applying a small oscillation to the force signal at a relatively high frequency. NanoDMA measurement can be performed on a larger sample area by using so called in-situ imaging capabilities. During the imaging process, the system continuously monitors the displacement amplitude and phase shift as a function of the position. From the recorded displacement amplitude and phase lag, the storage and loss modulus are determined at each image pixel (typically 256×256 px) if the geometry of the indenter is known. At the same time one can also gain information about the sample surface morphology.

The storage modulus depends on the elastic recovery of the sample, which is the amount of energy recovered from the sample subsequent to a loading cycle (i.e. stiffness of the material). The loss modulus relates to the damping behavior of the material. Detailed description of modulus mapping technique can be found in Asif [5].

Static nanoindentation was performed by using a Hysitron Tribolab device, located in the Center for Nanotechnology in Civil Engineering at the Faculty of Civil Engineering at CTU in Prague. Elastic properties of the wood cells were evaluated by using both (the quasi-static nanoindentation and modulus mapping) techniques.

The results of the static loading are in set of nanoindentation curves. This curve describes the response of the material to mechanical loading, in particular the relation between the loading force and the depth of penetration. Matrix of 4 × 5 indents were performed on the cell wall and inside the lumen by using quasi-static nanoindentation. The indents are located in minimal distance of 3 μm from each other to avoid their mutual influence. Loading diagram of standard controlled load test of an individual indent consisted of three segments: loading on

maximum force P_{max}, holding at the peak and unloading. In our case, the loading and unloading of this trapezoidal loading function lasted for 5 seconds and the holding part lasted for 8 seconds. Maximum applied load was 400 μN. During loading, the material under the indenter deforms both elastically and plastically. Upon unloading, however, only the elastic deformation can be assumed in most cases, which allows the calculation of an effective local stiffness of the material known as the indentation or reduced modulus. The reduced modulus can be related to the sample Young's modulus *E* using the derived relations [6].

Results and Discussion

An internal structure of the earlywood cells was monitored by using AFM as shown in Fig. 1. It is clearly visible that the earlywood cell is thin-walled, having the wall thickness between 2 and 3 μm and being equipped by a large lumen with approximate dimensions of 17 × 22 μm.

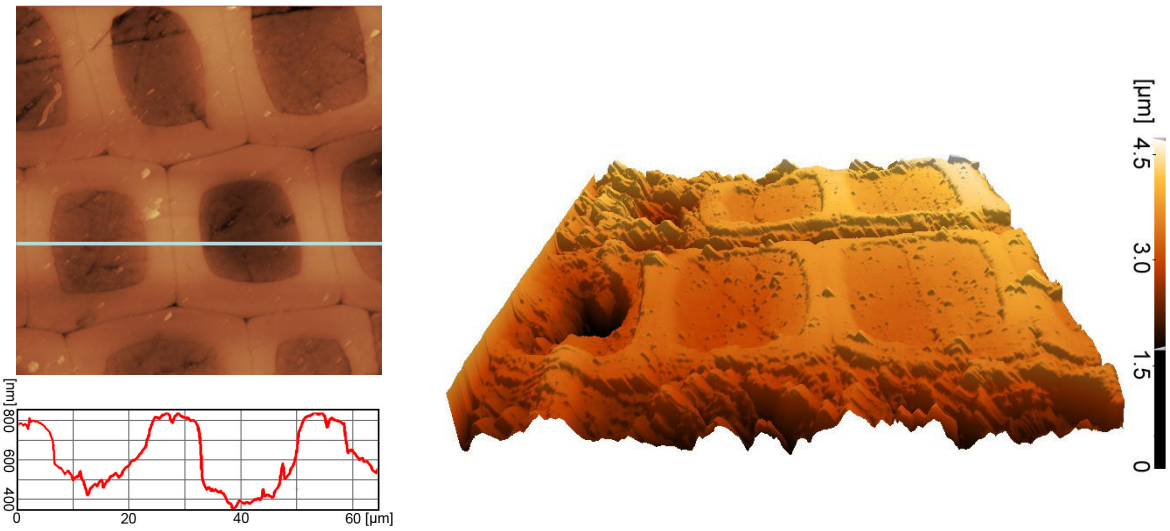

Fig. 1. AFM image of a earlywood cells 65 × 65 μm, cross section of cells – (left) and AFM image of a earlywood cells in axonometry (right).

The modulus mapping measurement was accomplished by applying a small oscillation to the force signal at frequency of 180 Hz. The amplitude of the force oscillation was 5 μN and the nominal contact force was 12 μN. A large area of 16 × 16 μm was chosen after setting an appropriate gain for the lock-in amplifier and an initial dynamic force for the tip oscillation, along with a scan rate of 0.2 Hz. Due to small viscosity, the loss components were relatively small compared to storage components, suggesting that the storage modulus is practically equal to the Young's modulus evaluated in the quasi-static case.

The map of storage modulus in the selected area is depicted in Fig. 2. It is easy to observe that the cell wall have a higher Young's modulus than the lumen. However, notable local variations in both phases could be observed from the map (Fig. 2). The average value of Young's modulus of cell wall was 10.2 ± 1.4 GPa.

The output of our measurements by static nanoindentation is the set of force displacement nanoindentation curves. The average contact depth was established as 270 nm for earlywood cells and 324 nm for lumen. These values are sufficient with respect to the surface roughness, but not too large to avoid interaction between phases. The values of Young's modulus are 10.2 ± 0.9. The values were obtained by our measurement, which are reasonable with respect to the effective wood properties and results reported in literature [2,3].

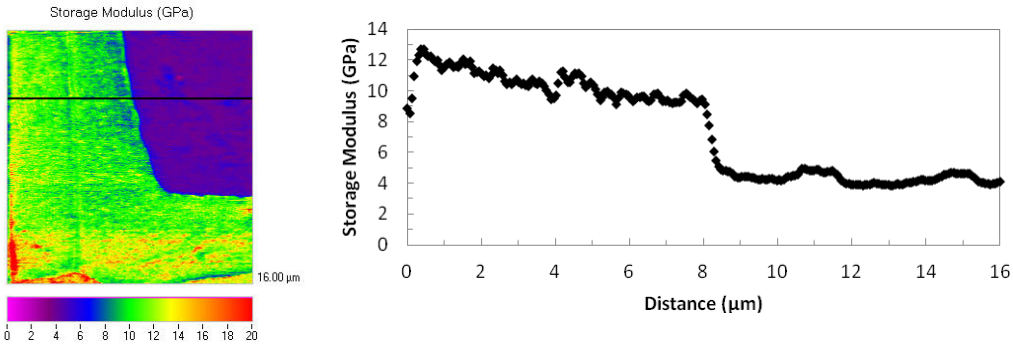

Fig. 2. Imaging of the spruce tissues Storage Modulus on an area 16 × 16 µm – (left) and the Storage Modulus value from a cross-section (right).

Conclusions

The presented results obtained by the dynamic indentation (Fig. 2) and determined by the static indentation are in a great agreement: 10.2 ± 0.9 GPa (static indentation) and 10.2 ± 1.4 GPa (modulus mapping technique). The slight differences between the results are within the measurement error. The values are reasonable with respect to the effective wood properties and results reported in literature [2, 3]. Both methods can be used to characterize mechanical properties of wood tissues of Norway spruce at micro/nano scale.

Acknowledgement

This outcome has been achieved with the financial support of the Czech Technical University in Prague SGS14/122/OHK1/2T/11. Special thanks belong to the Center for Nanotechnology in Civil Engineering at Faculty of Civil Engineering CTU in Prague and Joint Laboratory of Polymer Nanofiber Technologies of Institute of Physics Academy of Science of Czech Republic and Faculty of Civil Engineering CTU in Prague.

References

[1] P. O. Kettunen, Wood structure and properties, Trans tech publication, Tampere, 2006.

[2] R. Wimmer, B.N. Lucas, T.Y. Tsui, W.C. Oliver, Longitudinal hardness and Young's of spruce tracheid secondary walls nanoindentation technique, Wood Science and Technology 31 (1997) 131-141.

[3] 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.

[4] O. Jiroušek, D. Kytýř, P. Zlámal, T. Doktor, J. Šepitka, J. Lukeš, Use of modulus mapping technique to investigate cross-sectional material properties of extracted single human trabeculae, Chem. Listy 106 (2012) 442-445.

[5] S. A. Asif, K. J. Wahl, R. J. Colton, O. L. Warren, Nanoindentation and contact stiffness measurement using force modulation with a capacitive load-displacement transducer, Review of Scientific Instruments 70 (1999) 2408-2413.

[6] W. Oliver, G. Pharr, An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments, Journal of Material Research 7 (1992) 1564-1583.