MICROMECHANICS OF NON-EMBEDDED SPRUCE WOOD: NOVEL POLISHING AND INDENTATION PROTOCOL

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1. Introduction

The material properties, such as the reduced elastic modulus, of wood can be determined by indentation protocols and resulting unloading displacement curves [1]. There are several indentation campaigns performed in wood [2,3]. Nevertheless, most of them use embedding substances, such as resin, to stabilize the material for surface polishing protocols [4]. Thus, we propose a novel polishing protocol without the need of an embedding medium. This will be complemented by an extensive number of indents and a statistical nanoindentation technique [5,6] to identify the phase with non-mechanical damage. Namely, we are focusing on indents different than air phase and mechanically damaged cell wall, as seen in Fig.2.

2. Materials and Methods

12 cubes of Norway spruce (*Picea abies*) with dimensions of approximately 2x2x2 cm^3 were harvested by means of different circular saws. Subsequently, each of the cubes was attached to a microscope glass slide using a 2 components glue. Later, the samples were stored in a climate chamber at 21 centigrade and 35% relative humidity until further steps.

2.1 Polishing

The surface perpendicular to the grain direction was polished by means of an ultra-miller (Leica Microsystems GmbH, Germany). The resulting finish of the surfaces were examined first by means of a light microscope, as seen in Figs. 1 and 2. Furthermore, the roughness of a region in each sample was examined by means of a Triboindenter (Hysitron Inc., USA), equipped with a three-sided pyramid-shaped tip (Berkovich type).

2.2 Indentation

The nano-indentation protocol was performed in displacement control mode by the beforementioned indenter. Conditions inside the indenter chamber were kept constant at 21 centigrade and 35 % RH. Two early and two late wood areas were selected at each of the 12 samples surfaces. Subsequently, 100 indents were performed on each of the areas, resulting in a total of 4800 indents. The indentation depth was 300 nm, and the separation in between indents was 30 micrometers. The probability distributions of the resulting elastic modulus were interpolated using a linear combination of three log-normal Probability Density Functions. The non-mechanically damaged phase will be represented by the distribution with the highest statistical mean.

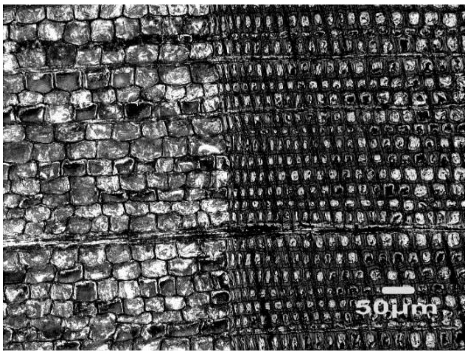

Fig. 1. Light microscopy image of spruce wood with 100x magnification of the transition area between early wood (left) and late wood (right). Sample number 2/12.

3. Results

Three probability distribution functions work adequately for our resulting reduced elastic modulus. Figs. 3, 4, 5, show the beforementioned statistical approach. The mechanical undamaged phase (cell wall) is the mean of the distribution with the highest mean elastic modulus and is represented in our figures as a red-colored PDF.

4. Discussions

The proposed polishing and indentation protocol allow a new methodology to study the microstructure of wood without the influence of external agents. The embedding with resin of wood, consist of an unknown saturation of porosity with resin. This has unknown effects on cell wall stiffness. [7] documented around 17 GPa for embedded samples. Meanwhile, we obtained around 15 GPa for the mean late wood undamaged phase. Our lower value may result from a higher hydration level of the cell wall, meditated through open pores in a climate chamber. Embedding of wood with resin typically does not lead to complete filling of lumen pores.

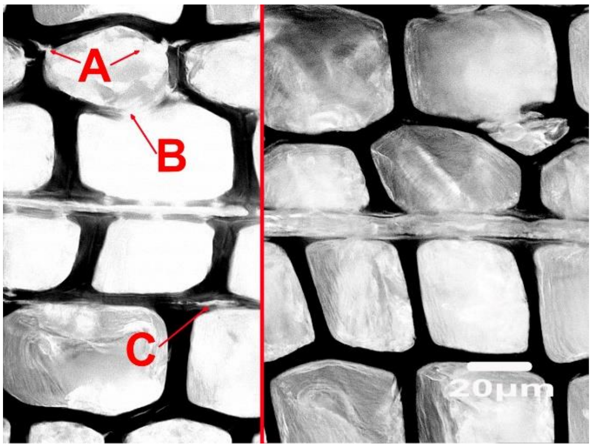

Fig. 2. Light microscopy image of spruce wood with 500x magnification of early wood. Sample number 4/12 (left) shows the mechanical damages: (A) cracks (B) wall failure (C) delamination. Sample number 1/12 (right) shows mostly desired undamaged cell walls.

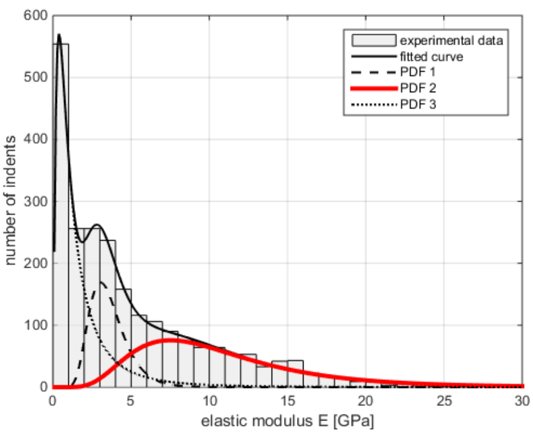

Fig. 3. Probability distribution functions and histogram of elastic modulus from 2400 indents on early spruce wood (fitted curve with Root Mean Squared Error of 0.00165 and R-Squared of 0.999). PDF 2 represents the undamaged cell wall.

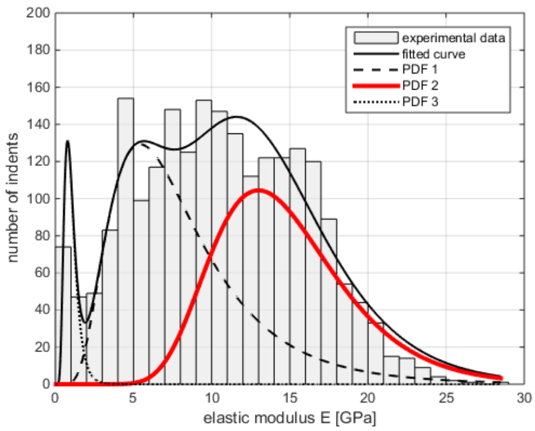

Fig. 4. Probability distribution functions and histogram of elastic modulus from 2400 indents on late spruce wood (fitted curve with Root Mean Squared Error of 0.00828 and R-Squared of 0.925). PDF 2 represents the undamaged cell wall.

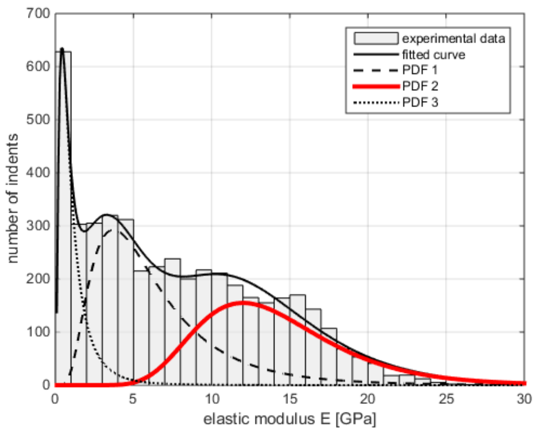

Fig. 5. Probability distribution functions and histogram of elastic modulus from 4800 indents on early and late spruce wood (fitted curve with Root Mean Squared Error of 0.00266 and R-Squared of 0.992). PDF 2 represents the undamaged cell wall.

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