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NONDESTRUCTIVE EVALUATION OF VISCOUS-ELASTIC CHANGES IN AMMONIA-MODIFIED WOOD USING ULTRASONIC AND VIBRANT TECHNIQUES

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ABSTRACT

The paper presents data on ultrasound responds to elastic properties and constants of modified and unmodified oak wood to changing specimen anisotropy, internal structure, size factor and testing method. Comparative analysis of dynamic MOE values obtained using developed at KTU ultrasonic Lamb wave tester and standard Bruikhuis MTG Timber Grader was focused on validation testing method reliability and measuring uncertainties. Gradual changing of specimens' sizes revealed specific and distinct variation of ultrasound Lamb wave time-of-flow when induced on specimen sides and edges. Size factor, grain and annual ring configuration and ammonia treatment of oak specimens reliably influence also coefficient of damping; the latter was on average 1.6 lower for modified oak wood.

KEY WORDS: ultrasound time-of-flight, modulus of elasticity, coefficient of damping, wood modification

INTRODUCTION

Recent investigation of wood properties distinguishes by gradual movement from destructive towards non-destructive testing (NDT) methods; before mentioned in turn often are used for validation of sometimes indeterminate and relative NDT. Classic static testing is especially time and resource wasteful on large size specimens resulting in rapid replacing them by NDT for production follow-up. Wood properties and internal defects are tested by x-ray or neutron ray examination; near-surface features scanned using video or thermo-graphic image processing and correlation; various ultrasonic, vibration, other dynamic testing methods are well known and widely applied (Bucur 2006, Vobolis and Albrektas 2007). Non-destructive methods allow enhancing species and grade populations for structural timber though requiring permanent and adequate control measures. Critical condition for scientific and industrial NDT applications is measuring uncertainties and overall accuracy.

Being anisotropic biological material wood marks wide spectrum and variation of physical and mechanical properties. Internal structural formations as knots, fissures, spiral grain, early/late wood ratio, etc. make characterization of certain properties even more difficult (Bucur 2006, Baltrusaitis and Pranckeviciene 2003). Detecting and evaluation of such formations could be equivalently sophisticated as tested substance and structure itself. At the same time, those methods have to be accurate, reliable, and productive and cost efficient. Ultrasonic wood examination is universally accepted as corresponding above mentioned requirements especially for evaluation mean values of wood elastic constants such as Young`s modulus (MOE) and indirectly strength (Sandoz et al., Green 2002, Beall 2002).

Fairly large data bank has been collected on ultrasound propagation phenomenon in wood depending on species, density, anisotropy, moisture or temperature. Isotropic grain angle is dominant and critical when assigning characteristic stiffness and strength values. Decrease of ultrasound velocity receding from longitudinal grain direction is coincident to the relevant timber MOE (Najafi et al. 2004, Kabir at al. 1998). Similar inverse effect is observed for growing specimen and even room temperature during testing (Vun et al. 2008, Bachle and Walker 2006). Latter is potentially fast-tracking for monitoring of the performance of timber structures in changing moisture and temperature conditions. Still not clear or scientifically proved moisture migration mechanism in wood together with resulting moisture induced stresses reveal another area of ultrasound application. Widely approved for sawn timber ultrasound testing is equally perspective for estimation elastic properties of sawlogs and even stems or growing trees (Grabianowski et al. 2006, Arriaga et al. 2006). Ultrasonic examination was well approved for testing specific properties of cloned and ageing wood e.g. maintaining hypothesis of changing mechano-sorptive behavior of the latter (Lindstrom et al. 2004, COST Action E55).

Changes of wood properties under heat treatment and modification were open for careful observation for more than 50 years. However, even well known wood ammonia modification remain many questions on plasticization proportions, colorings, extent of structural changes influencing physical-mechanical properties. Emerging new testing methods focused on wood characterization on micro and nano scales seems to be revolutionary in understanding breaking by ammonia treatment intra-molecular hydrogen links. For practical applications of such a modified timber, e.g. wood flooring key interest is monitoring, targeted correction of resonant behavior and grading and batching during production factory control. Scientific scope of this paper cover technical aspects of wave initiation during timber acoustic scanning related to naturally varying and modified wood properties as well as comparative analysis and accuracy validation of two different methods of wood acoustic examination.

Wood is an anisotropic material; consequently, wood material response is depended on the direction of the applied stress. Properties parallel to the grain differ significantly from those in the transverse direction (Barnett and Jeronimidis 2003). In fact, wood is considered an orthotropic material and is analyzed in three perpendicular planes of symmetry: longitudinal direction (L) along fibers (grain), radial direction (R) towards to the growth rings and tangential direction (T) to the annual rings.

The generalized Hook's law (Bucur 2006) can define the elastic properties of solids:

$$\lfloor \sigma_{ij} \rfloor = \lfloor k_{ijkl} \rfloor \cdot [\varepsilon_{kl}] \tag{1}$$

where $\lfloor k_{ijkl} \rfloor$ - are termed elastic stiffness; $\lfloor \sigma_{ij} \rfloor$ - stress tensor; $\lfloor \varepsilon_{ij} \rfloor$ - strain tensor. Experimentally, $\lfloor k_{ij} \rfloor$ the terms of the matrix could be determined from ultrasonic measurements.

The equation of wave can be written:

$$\rho \frac{\partial^2 u_i}{\partial t^2} - k_{ijkl} \frac{\partial^2 u_k}{\partial x_l \cdot \partial x_j} = 0 \tag{2}$$

For a plane harmonic wave with the displacement u:

$$u_i = A_i \cdot \exp\{i(k_j \cdot x_j - \omega t)\} \tag{3}$$

where k_j - the unit wave vector, $k = \frac{2\pi}{\lambda} n = \frac{\omega}{v_{phase}} n$; A_i - amplitude, $A_i = AP_m$; P_m- polarization vector.

The equation of motion is:

$$(\Gamma_{ik} - \delta_{ik} \rho v^2{}_{phase}) P_m = 0 \tag{4}$$

where Γ_{ik} - Kelvin-Christoffel tensor, $\Gamma_{ij} = k_{ij} n_i n_j$; n_i and n_j are the direction cosines; $v_{phase} = C$, C – sound velocity.

The Christoffel's equation relates the elastic constants to the velocities of ultrasonic waves in an anisotropic solid as (Bucur et al. 2002):

$$\begin{bmatrix} \Gamma_{11} - \rho \cdot C^2 & \Gamma_{12} & \Gamma_{13} \\ \Gamma_{21} & \Gamma_{22} - \rho \cdot C^2 & \Gamma_{23} \\ \Gamma_{31} & \Gamma_{32} & \Gamma_{33} - \rho \cdot C^2 \end{bmatrix} \begin{bmatrix} p_1 \\ p_2 \\ p_3 \end{bmatrix} = 0 \tag{5}$$

where ρ is the density and C the velocity.

For a unique solution we have to fulfill further condition:

$$\begin{bmatrix} \Gamma_{11} - \rho \cdot C^2 & \Gamma_{12} & \Gamma_{13} \\ \Gamma_{21} & \Gamma_{22} - \rho \cdot C^2 & \Gamma_{23} \\ \Gamma_{31} & \Gamma_{32} & \Gamma_{33} - \rho \cdot C^2 \end{bmatrix} = 0 \tag{6}$$

For wave propagation along symmetry axes for an orthotropic solid (wood) we obtain three solutions (Bucur 2006):

$$\begin{bmatrix} \Gamma_{11} - \rho \cdot C^2 & 0 & 0 \\ 0 & \Gamma_{22} - \rho \cdot C^2 & 0 \\ 0 & 0 & \Gamma_{33} - \rho \cdot C^2 \end{bmatrix} = 0 \tag{7}$$

Strictly speaking, such a matrix describes somehow idealized substance than wood in respect of both real anisotropy and especially "average" density. Therefore, ultrasonic wood scanning in general and methods discussed in this paper in particular require careful validation of measurement uncertainties especially as regards specimen sizes and grain configurations.

Consequently, wood density and grain direction influences considerably ultrasound velocity correspondingly to the acoustic and elastic wood properties. In principle, wood density and grain direction are variant for each specific spatial point of timber piece. Thus for orthotropic solid (wood) stiffness constants reflect generalization of real eigenvalues in every point of coordinate system.

Most of today's acoustic testing methods and calculated elastic constants describe simplified model based on orthotropic wood elastic constants collected when testing specimens with grain configurations accordingly to the each of orthogonal axis. Such fundamental approaches remain rather theoretical due to experimental complicity and weak adequacy to real wood properties and behavior. Moreover, for applied wood research acoustic scanning along the grain provide sufficient information for estimation timber element structural performance. For that after measuring sound velocity (time-of-flight), generalized modulus of elasticity (MOE) is calculated from simplified equation:

$$MOE = C^2 . \rho \tag{8}$$

where C – sound velocity, m.s^{-1}; ρ – wood density, kg.m^{-3}

For evaluation of wood plastic properties, another amplitude-frequency characteristic – coefficient of damping - is used (Ukvalbergiene and Vobolis 2007):

$$tg\delta \approx \eta = \frac{f_2 - f_1}{f_{rez}} \tag{9}$$

where $tg\ \delta$– tangent of loss angle; η – coefficient of damping.

These cost efficient and universal methods well correlate with the profound knowledge of static wood testing and data. Comparative format of our research focused on distinctions of treated-untreated wood characteristics and measurement method accuracy and uncertainties allowed to narrow testing procedure with the specimens having near-longitudinal grain direction.

MATERIAL AND METHODS

For the study oak (*Quercus robur*) timber was cut into 30 samples with 751 mm length, 75 mm width and 24 mm thickness respectively. A part of the samples were modified with ammonia in industrial autoclave applying vacuum-pressure-vacuum impregnation technology. The average specimen wood density was 658 kg.m^{-3} and initial average moisture content for unmodified oak wood was 9.1 % while that for the ammonia-modified wood was 11.8 %.

For the estimation of sample resonant frequency, MOE and coefficient of damping stress-strength device „Timber Grader MTG“ was used. The measurement device picture is given in Fig. 1, a. The entire measurement system consists of the following devices: measurement instrument „Timber Grader MTG“, wooden specimen, weighing device, wireless bluetooth communication device, PC with „Timber Grader“ software. The measuring principle of the MTG is the measurement of the natural frequency of wooden specimen. The stress waves are introduced with an integrated electric hammer and measured with an integrated sensor, which is adapted to sense a reflection of an impulse transmitted to the object which in turn is adapted for providing a sensor signal (Elbers and Rozema 2006). Vibrations that are brought into the wood are converted into a digital value and sent via the wireless bluetooth connection to the computer. Analogically the data from weighing device are sent to the computer and measuring device. Eigen frequency is determined by the Fourier spectra of vibration. The stress wave velocity can be calculated:

$$C = 2Lf \tag{10}$$

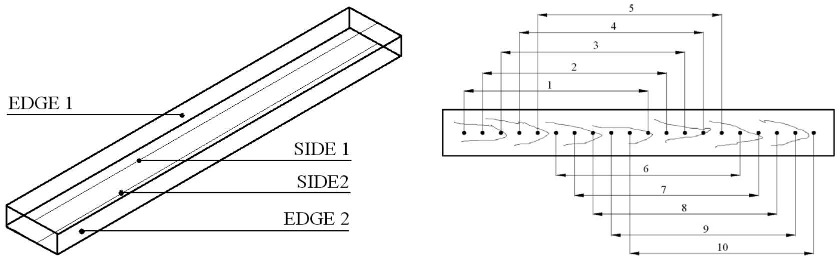
where L – length of the specimen; f – eigen frequency.

a **b**

Fig. 1: Measurement devices: a – Timber Grader MTG (picture from http://www.brookhuis.com): b – hardboard strength meter, developed at KTU

Parallel to the above mentioned were accomplished measurements using acoustic stiffness-strength meter developed at KTU (Fig. 1 b). A technique is based on excitation of acoustic Lamb waves in wood and measurement time-of-flight between fixed at 380 mm transmitter and adapter (Augutis at al. 2007). Sound velocity is calculated from time-of-flight and length ($C = L/t$). Identical testing was performed on the initial specimen sizes using both techniques and procedures for comparative analysis of testing accuracy and measurement uncertainties. Later on series of MOE experiments using KTU stiffness meter were carried out for evaluation size factor and wood anisotropy in changing specimen widths and thicknesses.

Measurements were performed on four specimen planes (Fig. 2 a) denominated Side 1 (S1), Side 2 (S2), Edge 1 (E1) and Edge 2 (E2). Subsequent specimens tangential wood layers were removed by planing sides unfolding that way randomly changing early or late wood configuration recording simultaneously notable variation of given specimen density. Thus, anisotropy and density variation was possible to relate to the measured specimen MOE. In case of planing edges minimal density changes occurred securing that way fixation purely size factor effects on stiffness values. Specimen width was changed by planing repeatedly every 6 mm on edges E1 and that for changing thickness was successive 3 mm on sides S1. Longitudinal ultrasound scanning was performed gradually on every particular specimen plane at 10 steps (Fig. 2b). Scanning data on planed and sawn specimen surfaces was also compared.



a **b**

Fig. 2: Specimen scanning points: a – scanning planes; b – scanning zones from 1 to 10

For evaluation effects of changing specimen moisture content on ultrasound velocity and respective MOE values modified and non-modified oak specimens were for 24 hours wetted in distilled water in standard temperature (+ 21 °C). Then during 17 hours drying period changing ultrasound time-of-flight and moisture content (measured by weighing method) was recorded every successive hour.

RESULTS AND DISCUSSION

Resonant frequencies of unmodified oak wood (Figs.3, 4) and resulting MOE (Tab. 1) measured with Timber Grader MTG were on average by 1.25 % lower than those for modified specimen. (Figs. 3,4) show regularities of vibration change in time and amplitude-resonance characteristics of unmodified (Fig. 3) and modified (Fig. 4) oak wood. Vibration fading time for modified oak on average by 33 % was lower than that of unmodified (illustrated as 18 ms, Fig. 4a and 24 ms, Fig. 3a respectively). Plasticization degree is convincingly exemplified also on FFT graphs (Figs. 3b and 4b) illustrating additionally procedure and data for coefficient of damping calculation (Eq. 9). For cited graphs numerical values of damping coefficients were 0.02999 for unmodified oak while that of modified was 0.05733 or by 1.9 higher. For all tested wood samples last figure balanced between 1.6-1.9.

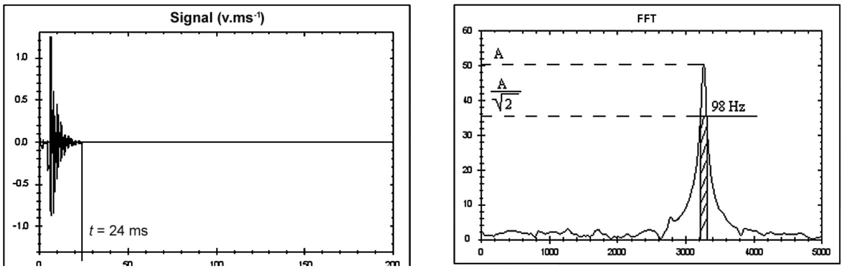


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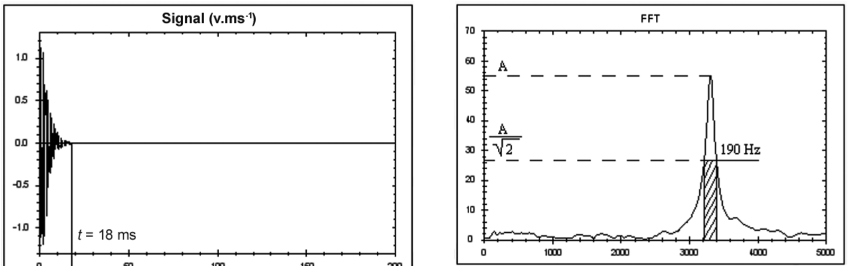
Fig. 3: Amplitude-resonant vibration graphs of non-modified oak: a – vibration time to flat graph; b – amplitude-frequency characteristics (Fast Fourier Transform time data)



a b

Fig. 4: Amplitude-resonant vibration graphs of modified oak: a – vibration time to flat graph; b – amplitude-frequency characteristics (Fast Fourier Transform time data)

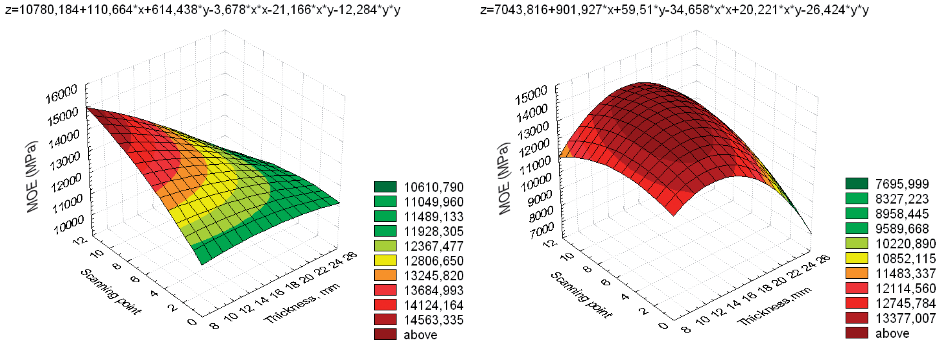
We recommend this simple signal processing procedure using MTG derived amplitude-frequency graphs for standard estimation of wood viscous properties through coefficient of damping broadening this way MTG application area.

After completion testing with Timber Grader MTG, the same specimens were scanned with acoustic KTU stiffness-strength meter. These two devices differs in measurement principles: Timber Grader MTG measures end-to-end stiffness, i.e. the hammer hits the wood sample from the end introducing stress waves and acoustic KTU stiffness meter measures the local stiffness between transmitter and adapter successively going in the ten scanning zones of the specimen and afterwards the mean values are calculated. Comparison results are presented in Tab. 1. Compared to the MTG obtained results last-mentioned method gives up to 28.9 % lower MOE values. Both testers showed high accuracy and near-identical measurement values repeatability (see below). MTG Timber Grader calculates MOE using special software modeling specimen sizes, density, and moisture content and presenting results comparable to the EN 338 requirements. Direct frequency or ultrasound velocity readings and MOE calculations (Eq. 8) for KTU stiffness-strength meter and similar tester types seem to be incorrect for calculation or prediction of standard characteristic values. Such methods could be recommended for comparative and proportional stiffness properties analysis when no official design values are discussed. On the other hand, MTG tester also generalizes "end-to-end" stiffness and thus predicts strength values for the total timber piece. Local stiffness-strength reducing characteristics are not enough readable. Here in turn longitudinal ultrasonic scanning could be more effective or complementing "end-to-end" testing for localization internal weaknesses of wood structure

Tab. 1: Results of oak ultrasonic measurements (mean values)

Specimen	MOEMTG / fMTG (MPa/Hz)	MOEUS / fUS (MPa/Hz)	Difference (%)
Unmodified oak wood			
N1	15109 / 3270	13534 / 3068	10.4
N2	16150 / 3392	15812 / 3230	2.1
N3	15931 / 3367	15114 / 3165	5.1
Modified oak wood			
M1	16685 / 3436	11860 / 3034	28.9
M2	15818 / 3314	15733 /3224	0.5
M3	16588 / 3406	13382 / 3055	19.3

Further testing was focused on estimation size factor on the accuracy of ultrasound measurement. Gradual changing specimen dimensions slightly transform internal structure configuration and that in turn have to be sensed by tester provided measuring accuracy was sufficient. Fig. 5 confirms that hypothesis while repeatable measurements series proved acceptable levels of measurement uncertainties, stability and variability.

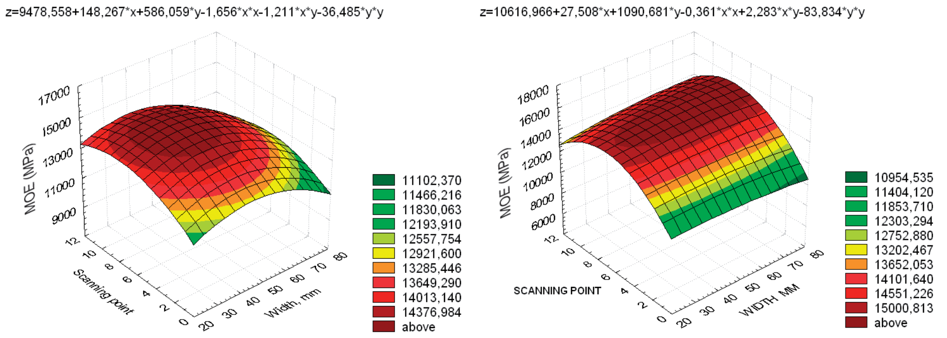


a **b**

Fig. 5: MOE dependence on specimen thickness: a – unmodified oak; b – modified oak

Diminishing of specimen thickness lead to relative increase of MOE. For tested specimens and thicknesses Lamb waves are not sensitive to their changes therefore MOE changes are related to the variable wood internal structure configuration. This phenomenon is particularly seen for smaller thicknesses where low number of remaining annual rings and their superposition while planing resulted in notable density changes (Fig. 5a). Some influence can be credited to the Poisson`s ratio variation. Character and variability of graphs was clearly individual for specific specimen and as example close-to-symmetric MOE values of modified oak is illustrated on Fig. 5b. Thickness reduction from 26 to 8 mm resulted in changes of MOE values up to 25.7 % for unmodified and up to 44.8 % for modified oak wood. As commented above, main reasons of such variations most probably are density changes due to indeterminate early/late wood superposition, especially at the lowest thicknesses. Although well defined and confirmed by our experiments in general, such a hypothesis have to be more accurately and numerically confidently specified by further research based on the special specimen sampling procedure. Modified wood demonstrated obviously higher homogeneity compared with unmodified.

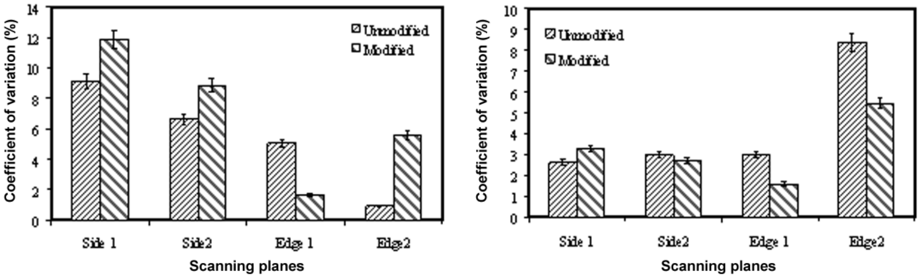
Gradual changing of specimen widths planing edges by 3 mm resulted in less variable MOE changes compared with the thickness reduction (Fig. 6). It can be seen that both for modified and non-modified oak character of the 3D surfaces is similar but modified wood is clearly more homogenous and for illustrated case not related to the changed specimen width. This is another confirmation of our hypothesis on different effects of specimens' dimensions and size factor in general. For tangentially sawn timber pieces with close to parallel to the side plane annual ring layers even slight further planing could cause notable stiffness changes; same done on the edges results in only marginal MOE changes. Still measurements seemed to be sensitive to the localization of the ultrasonic tester closer to the specimen ends: MOE values decreases in some cases by 25-35 % when scanning at both ends. This is also the matter of consideration when developing and usage of industrial articles of similar testers. Next query adverts to the unknown specifics of the Lamb wave propagation in the laminar nature of wood structure.



a **b**

Fig. 6: MOE dependence on specimen width: a – unmodified oak; b – modified oak

Analyzing data of measuring accuracy and uncertainties the general finding was high precision of repeatable measures. Changes of measuring coefficients of variation revealed other appearances to be considered. Those are trustworthy differences between readings on planed and untouched opposite specimen planes (Fig. 7). Coefficient of variation measuring MOE on planed side S1 is by 27.3 % higher than that on reference side S1 for unmodified wood and by 25.2 % for modified (Fig.7a). These figures reflect and specify numerically structural - hypothetically mainly density - changes through resulting MOE variation.



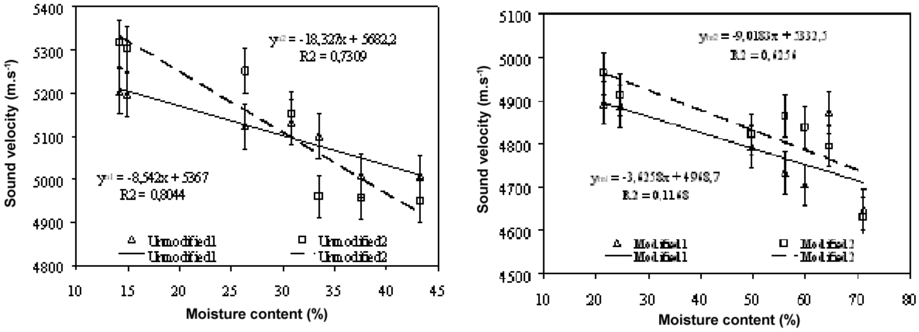
a **b**

Fig. 7: COV graphs: a – for measures when changing specimens thickness (planing on side S1); b – for measures when changing specimens widths (planing on edge E2);

Coefficient of MOE variation when changing width planing edge E2 on that edge was by 64.1 % higher than that on reference edge E1 for unmodified and by 70.9 % for modified oak wood (Fig. 7b). MOE variation on remaining planes remained amazingly stable and essentially lower than 3 %. It reflects marginal longitudinal structure variety on untouched defect-free surfaces. Close to twofold higher coefficient of variation on planed edge proves the hypothesis that Lamb wave and particularly acoustic measuring accuracy in general is specimen size-related. Anyhow, when determining global or "average" stiffness of anisotropic material critical issue is separation of naturally varying structure responds within acceptable overall measuring method accuracy and uncertainties.

Several works show big influence of macro-scale wood anisotropy to sound velocity (Bucur and Declercq, 2006, Vun et al. 2008). Still, there is lack of scientific information to compare our results of mezzo-scale early-late wood proportions impacts on viscous-elastic behaviors of thin wood lamellas.

Wood moisture content (MC) is one of the most important physical characteristics of wood. Fig. 8 presents dependence of ultrasound velocity in wood from MC changes. After 24 hours wetting in distilled water ammonia-modified wood saturation was observably - by 27.7 % - higher than that of unmodified.



a **b**

Fig. 8: Ultrasound velocity in changing specimen MC: a –unmodified oak wood; b – modified oak wood

As an outcome, ultrasound velocity is inversely proportional to the growing MC. For unmodified wood growth of MC from 14.2 % to 43.3 % resulted in decrease of ultrasound velocity by 6.9 % (Fig. 8 a); change of MC in modified oak from 21.5 % to 71.0 % reduced velocity by 6.7 % (Fig. 8 b). Linear models and high correlation of both graphs allow safely prediction of moisture-induced MOE changes by ultrasonic inspection of timber structures. Likewise evident is increased on average by 6% viscosity and thus as many reduced stiffness of modified wood compared with unmodified, illustrated through proportionally reduced ultrasound velocity.

Other studies also showed the decrease of sound velocity as moisture content increases for spruce (Unterwieser and Schickhofer 2010), red oak and hard maple (Simpson 1998), red pine (Vun et al. 2008). Some researchers showed steep decreases in acoustic velocity below fibre saturation point for radiata pine (Chan et al. 2010, Sandoz 1992). There were also estimated that below the fibre saturation point the dependance between sound velocity and moisture content is linear and above this point - non-linear. The same dependence was obtained in this study.

Experimental results unfolded and explained new attitudes towards better understanding of dynamic examination the naturally varying internal structure of modified and non-modified wood.

CONLUSIONS

1. Accuracy and uncertainties of tested measuring methods based on acoustic and natural frequency responds proved valid for the inspection of the internal wood structure. An effect of specimen dimensions (size factor), density variations due to near-surface annual ring thickness or specific grain configuration overall is fairly detectable by vibrant or ultrasonic Lamb waves scanning.
2. Dynamic end-to-end vibrant scanning together with the simultaneously recorded acoustic responds to the local timber properties variations allows combining general dynamic stiffness constants of the whole timber piece with the lengthwise defect tracking.
3. Ammonia modification results in oak plasticization and decreased by 1.33 times vibration

fading period; respectively, coefficients of damping for modified oak are 1.6-1.9 times higher compared with unmodified.

4. Ammonia-modified oak wood saturated water to 27.7 % more than unmodified oak wood. Ultrasound velocity in modified oak is on average by 6 % lower.
5. Linear models of moisture-induced dynamic MOE changes support feasibility of ultrasonic inspection for effective prediction of the load-bearing performance of modified and non-modified timber structures.

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