Variation of the tree ring micro-hardness demonstrated on spruce wood

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ABSTRACT: Micro-hardness was used for the study of wood structure (Norway spruce) in the line perpendicular to tree rings (radial surface). The steel indentor 0.25 mm in diameter with flat head was used for this purpose. The individual penetration tests were performed at constant velocity 0.0167 mm/s into a depth of 0.3 mm. Local wood strength was defined as the mean pressure on the indentor head at 0.02 mm penetration. The set of tests (~ 320) gave information about stress variation in dependence on the location of the test place in the tested surface. The stress was understood as a parameter describing the growth properties of wood similarly like the density usually used in dendrochronology. The measured strength variation is in agreement with visually observed tree rings. The acquired data made it possible to determine the mean characteristic points of the tree ring as well as the development of the parameters in dependence on the weather variations.

Keywords: wood; tree ring; micro-hardness; penetration; strength

Regular alternation of growth conditions during a year determines regular alternation of growth cycles for most plants. These changes can be easily indicated also for trees, e.g. in the growth and falling of leaves. Among such indications an important role is played by the tree ring structure of wood. This structure can be observed as a relatively regular alternation of lighter and darker rings in the cross-section cut of grown wood, or vertical parallel bands in radial section. It is known (KOLLMAN 1951; POŽGAJ et al. 1997; FRATZL 2002; ŠLEZINGEROVÁ, GANDELOVÁ 1994) that the ring structure is connected with the alternation of wood density. This structure is traditionally described as the changing of thicker earlywood rings of lighter colour with narrow latewood ones of darker colour. The earlywood with typical water-conducting function is understood as the wood with lower density than the latewood with largely mechanical function. The real tree ring structure is more complicated than this crude description. At least, its structure depends sensitively on climate and weather changes (HORÁČEK 1995). The tree ring structure also reflects special soil, position and weather conditions during tree growth. The special scientific discipline – dendrochronology is based on measurements of the tree ring width as an indication of growth intensity in the separate years. The further details of tree ring structure were studied by new independent methods that are based on light (or X-Ray) absorption in the wood sample (e.g. KOCH 2002).

The mechanical parameters of wood also depend on wood density (KOLLMANN 1951; POŽGAJ et al. 1997; HOLMBERG 2000). It was shown (HOLMBERG 2000) that there was a good relationship between density and hardness and that this correlation was better for higher load angles (angles between load and grain axes). This relationship implies that the tree ring microstructure can be determined also by mechanical means, e.g. by micro-hardness methods (EYERER, BÖHRINGER 1976; WIMMER et al. 1997; RINN et al. 1996; ECKSTEIN, SAß 1994). Micro-hardness was used for tree ring analysis for example by EYRER and BÖHRINGER (1976), who studied the quality of material surfaces after industrial processing by penetration of diamond cone into the wood surface. WIMMER et al. (1997) studied secondary walls of spruce tracheids in longitudinal direction by a diamond indentor impressed into the cell wall. Further methods of this type follow. The resistograph (RINN et al. 1996; ECKSTEIN, SAß 1994) is an instrument which drives a rotating needle into the wood and measures its drill resistance. The pilodyn (e.g. BUCUR 1985) measures the depth of penetration of spring-loaded striker pin injected into the wood with a constant energy of 6 J.

Classical hardness or micro-hardness methods (Brinell, Janka, Knoop, Shore etc.) are based on measurements of diameter (or diameters) of the indentation due to an indentor impressed into the wood surface or also measurement of load necessary for penetration of the indentor into the given depth (HOLMBERG 2000). This paper is an attempt

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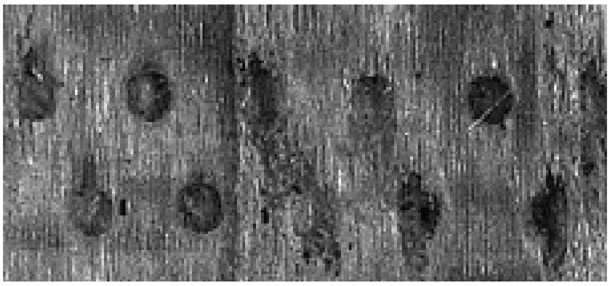

Fig. 1. Photo of penetration traces on the tested sample surface. Fine structure in the figure represents individual wood cells (tracheids). There is an order difference between diameters of the indentor and the individual tracheids

to study the detailed tree ring structure of spruce wood by a modified micro-hardness method, consisting in the penetration of a cylindrical indentor with flat head at a constant penetration rate into the test specimen.

MATERIAL AND METHODS

Norway spruce (*Picea abies* [L.] Karst.) wood from the lower part of a tree was obtained from the University forest in Kostelec nad Černými lesy. The planar part of 100 × 80 × 20 mm in size was sampled for our experiments. The face 100 × 80, part of an ideal radial plan used for experiments was sanded with fine emery paper (up to grain size 320) to obtain flat surface.

All the tests were performed on Universal Testing Machine (UTS) Instron, type 4464. The sample was fixed in a special jig so that the tested face would be perpendicular to the penetration axis. The jig enabled to move the sample precisely in both the main directions parallel to the tested face: parallel (y) and perpendicular (x) to the annual ring traces. The tests began at the side of the sample that is closer to the grain of wood, and ended at the side of the sample closer to the phloem.

The tested surface was penetrated repeatedly by a steel cylindrical indentor (diameter 0.25 mm) with flat head into a 0.3mm depth followed by rapid reloading. The penetration rate 0.0167 mm/s was used in all tests, the number of tests was 320. The centres of the tested areas were arranged into two lines perpendicular to the tree ring traces (Fig. 1) so that the distances between them would be 0.5 mm at least. This concept allowed to reach higher density and accuracy of penetration tests, the used distance of 0.5 mm ensured neglecting the difference in wood properties of these two parallel test lines.

Every penetration of the sample surface provided one deformation curve (Fig. 2) that was the basis for determination of the mean "strength" of the tested surface. This strength was determined from the "flow stress", the mean pressure on the indentation head at 0.02 mm inelastic penetration into the wood. There was an intersection of the corresponding loading curve with a line parallel to the quasi-static part of the loading curve at 0.02 mm higher deformations (Fig. 2).

The x-coordinate (with axis perpendicular to the tree rings – see Fig. 1) was used for determination of the individual test position.

RESULTS AND DISCUSSION

The typical penetration curves obtained in a tree ring are given in Fig. 3a. The x-plot of corresponding strengths on x-coordinate is given in Fig. 3b. The results given in Fig. 3b were approximated either by lines (two sides of the latewood) or by a polynomial of the second degree (earlywood) – the obtained equations are also given in the Fig. 3b caption. The cross-lines of the individual approximations gave characteristic points: EB (initial point of the earlywood and also of the whole tree ring; defined as the cross-line of the polynomial and the decreasing line of the previous tree ring), EM (earlywood minimum corresponding to minimal value of the earlywood strength polynomial approximation), EE (end point of the earlywood defined as the boundary between early and latewood inside the individual tree ring; the cross-line of polynomial and increasing line used approximation of the strength in

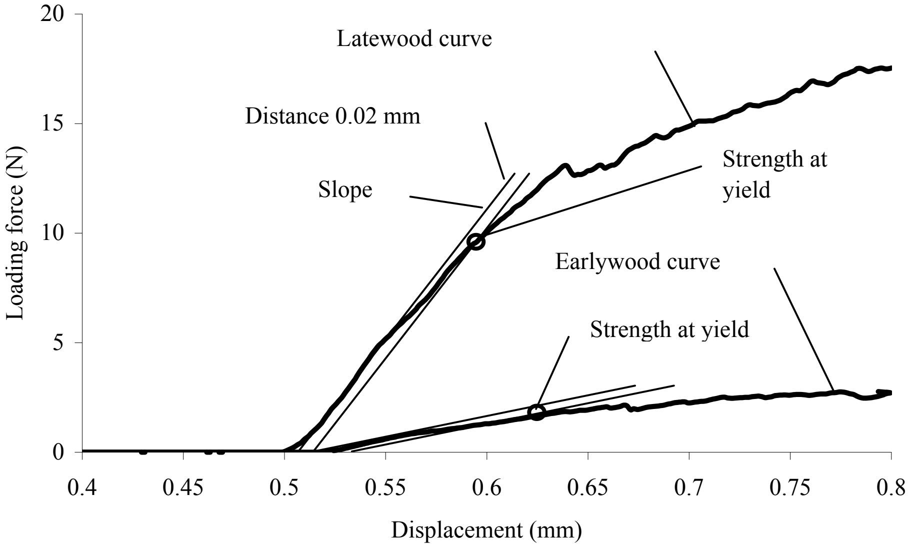

Fig. 2. Typical penetration curve with determination of the strength of the tested wood. Small circles denote the strength found by the analysis

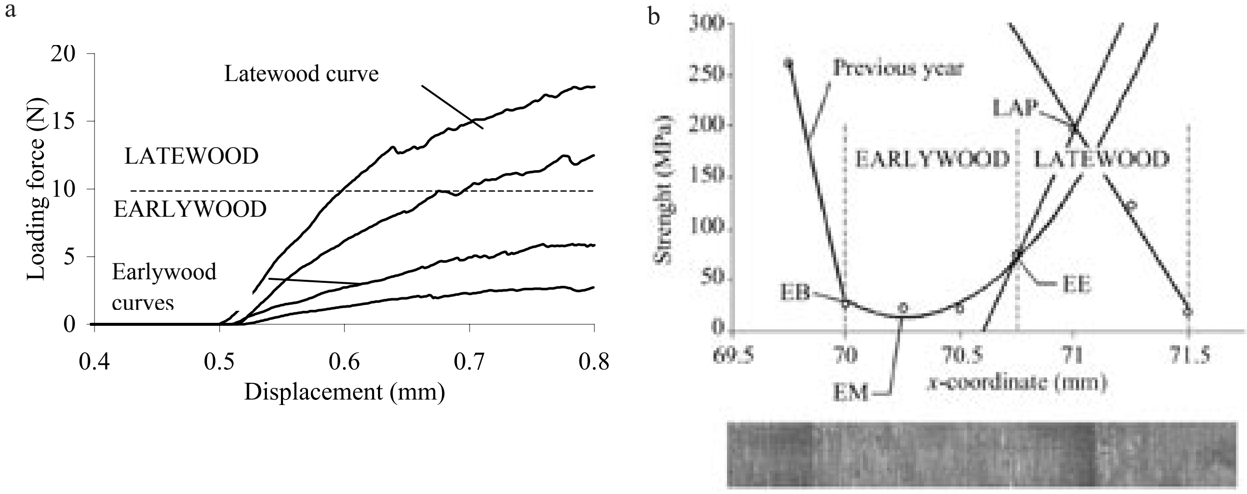

Fig. 3. Typical results obtained for one typical tree ring trace

a. Typical penetration curves for one ring trace

b. Wood strength (STR) plotted against *x*-coordinate. Approximation of the obtained results: spring wood – approximated by a polynomial of second degree – in our case STR = 231.91 x^2 – 32,588 x + 1,144,811 (R^2 = 0.9338), EB – beginning of the spring wood, EM – minimum of the spring wood, EE – end of the spring wood, late wood – approximated by two lines – in our case STR = 491.69 x – 34,713.99 (R^2 = 1) and STR = – 355.68 x + 25.455 (R^2 = 0.9897), LAP – peak of the late wood. For the observed fine structure see Fig. 1. Definitions of all the important points (EB, EM, EE, LAP) are in the text

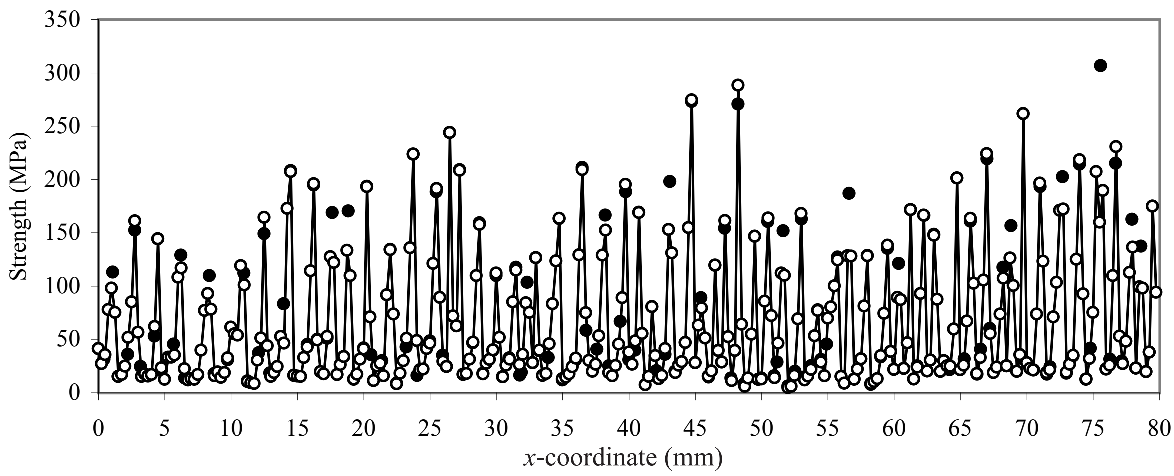

Fig. 4. Overview of the obtained strength values plotted against *x*-coordinate, as measured from the first test (connected open circled). Full circles denote the values obtained by approximation of the experimental data (under Fig. 3b)

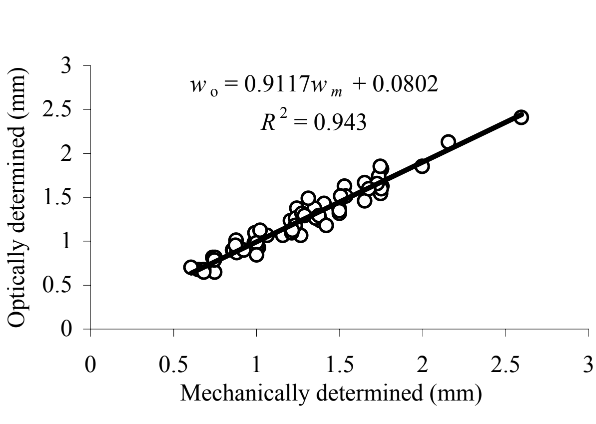

Fig. 5. Ring widths obtained by optical microscopy of the tested wood plotted against ring widths (RW) determined from strength-distance plot (Fig. 4)

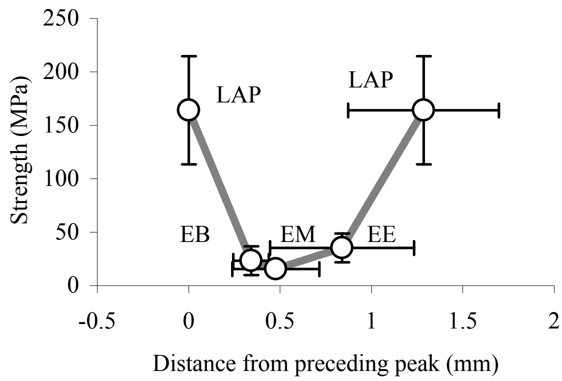

Fig. 6. Mean strength profile of the tree ring. The distances between the characteristic points were constructed according to the data given in Table 2; mean strength values were taken from Table 2. The bars denote SD of the data

Table 1. Basic statistical characteristics of the defined widths (MV – mean value, SD – standard deviation, CV – coefficient of variation)

Width	MV (mm)	SD (mm)	CV (%)
Ring width (RW)	1.285	0.413	32.1
Earlywood width (EWW)	0.498	0.297	59.6
EWW1	0.136	0.141	103.7
EWW2	0.363	0.274	75.5
Latewood width (LWW)	0.793	0.212	26.8
LWW1	0.448	0.196	43.7
LWW2	0.341	0.097	28.5

Table 2. Basic characteristics of the defined strengths (MV – mean value, SD – standard deviation, CV – coefficient of variation)

Strength	MV (MPa)	SD (MPa)	CV (%)
Late wood peak (LAP)	164.0	50.6	30.9
Earlywood beginning (EB)	23.4	13.5	57.8
Earlywood minimum (EM)	15.7	6.40	40.8
Earlywood end (EE)	35.4	13.5	38.0

latewood) and LAP (corresponding to latewood strength peak – defined as cross-line of two approximation lines in latewood).

Fig. 3b represents year period of the nearly periodical dependence of the strength. The same process of approximation as in Fig. 3b was also applied to the other results and the characteristic points were obtained for all the measured tree ring traces – an overview of the obtained results is given in Fig. 4. The figure contains the obtained EEs, EBs, LAPs. Large differences between spring wood and late wood strengths as well as the inter-season differences are clearly demonstrated in this figure.

The distance (x-coordinates) between two neighbouring LAPs is the ring width (RW), the distance corresponding to the yearly local tree thickening at the place of determination. The distance between neighbouring EE and EB points can be termed as the earlywood width (EWW). The difference between the x-coordinate of EB and the x-coordinate of preceding EE was called the latewood width (LWW). The EWW was divided into two parts separated by corresponding minimum EM (given by corresponding x-coordination – x_{EM}). The EWW was then composed of two sub-widths – EWW1 and EWW2 for x lower than x_{EM} and higher than x_{EM}, respectively. Similar division was also done for latewood, the point of separation was LAP, and the resulting widths were denoted as LWW1 (for lower x-values) and LWW2 (for higher x-values). The basic statistical characteristics for the defined widths are given in Table 1. The RW determined from the strength-distance plot were in good agreement with the RW data directly determined microscopically as distances between dark latewood traces (Fig. 5).

The basic statistics of the ring widths are given in Table 1 and similar data for the characteristic strengths were inserted in Table 2. The values from Tables 1 and 2 served as the basis for construction of the 'mean' ring profile of the tested wood that is displayed in Fig. 6. Table 2 shows that the peak strength values were about ten times higher than the minimum strength values in the earlywood. Mean strength values of the earlywood were about 4–6 times lower than the wood strength at LAPs. The wood strength increases with increasing reduced density approximately as a power function (POŽGAJ et al. 1997) so that the plot of reduced density against x-value should have a similar character like the strength plots against the distance (Figs. 4 and 6).

Fig. 7 shows that the ring width decreased with increasing age of the tree as is usually mentioned in textbooks (SCHWEINGRUBER 1988), but moreover some variation in the ring widths from year to year was also observed.

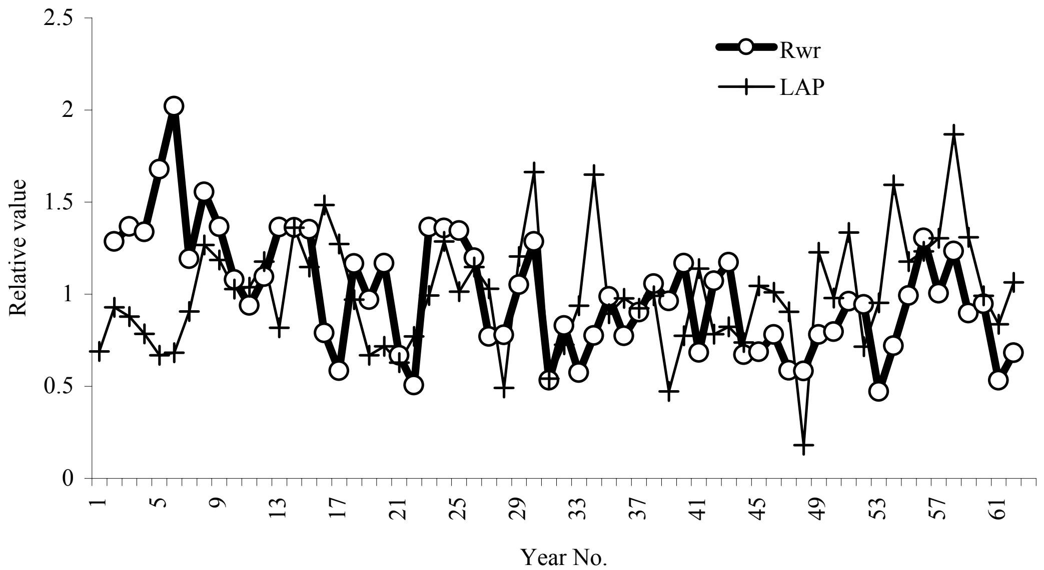

Fig. 7. Relative values of ring widths (Rwr) and LAP for the tested tree rings. Relative value was obtained as a ratio of the actual value to the mean value (Tables 1 and 2) of the corresponding quantity

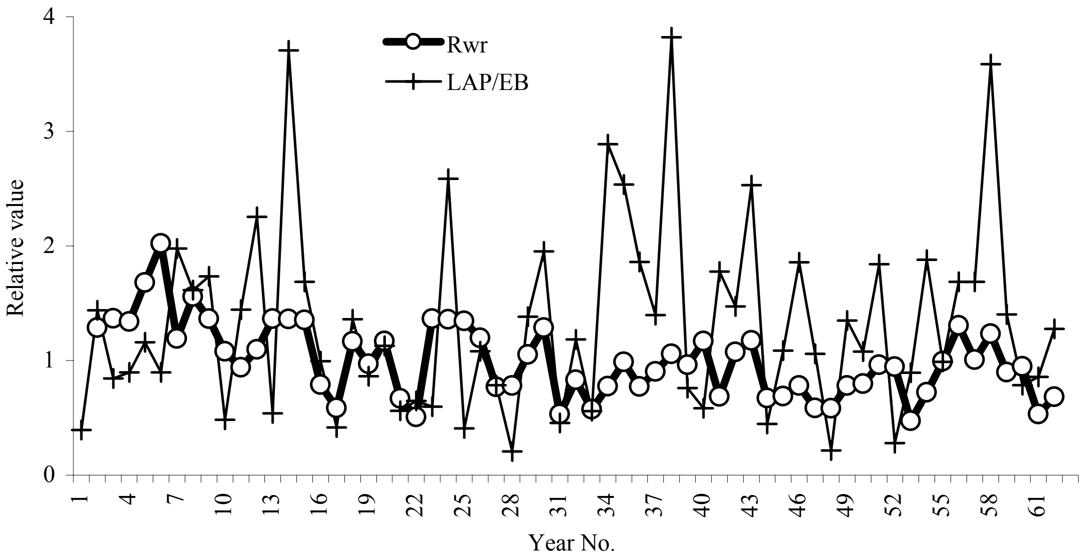

Fig. 8. Relative value of the ring width (Rwr) and ratio of the relative values for LAP and EB for the tested individual tree rings; see caption to Fig. 7

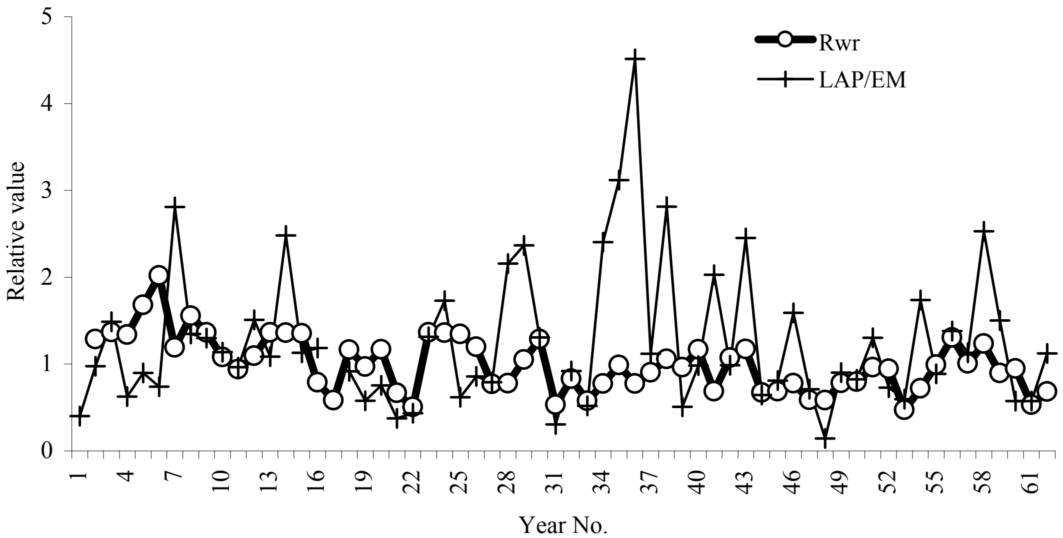

Fig. 9. Relative value of the ring width (Rwr) and ratio of the relative values for LAP and EM for the tested individual tree rings; see caption to Fig. 7

The most important indications of this plot are the points in which Rwr (relative ring width; ratio of the actual ring width value to the mean value of the whole plot) reached values close to 0.5. These minima corresponded to such periods of tree growth in which lower values of LAP (maximal value of the strength, see definition above) were observed (Fig. 7) and the strength of earlywood was low (Figs. 8 and 9). The wide tree rings were observed in the cases in which the earlywood was of lower strength and the well-matured latewood of higher strength was observed.

CONCLUSIONS

The study of wood micro-hardness makes it possible to describe variation of wood strength, including the annual changes. The annual strength variation had a similar character like the wood density and its detailed study led to approximately the same tree ring periodicity as the optical methods.

The differences between the early and late wood strength are really marked; they are represented by the values of an order difference that seem to correlate with the tree ring width.

Lower strength values of latewood seem to be characteristic of narrow tree rings. On the other hand, lower strength and higher strength seem to be respectively characteristic of earlywood and latewood of wide tree rings.

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Změna mikrotvrdosti letokruhu smrkového dřeva

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ABSTRAKT: V práci byla zkoumána struktura dřeva smrku ztepilého pomocí měření jeho mikrotvrdosti na radiální ploše vzorku v linii kolmé na letokruhy. Pro jednotlivá měření byl využit ocelový trn o průměru 0,25 mm s plochým čelem, který pronikal rychlostí 0,0167 mm/s do hloubky 0,3 mm. Pevnost dřeva v místě průniku byla definována jako střední tlak na čelo trnu při deformaci 0,02 mm. Soubor všech 320 měření poskytl informaci o proměnlivosti napětí v rámci letokruhu. Napětí bylo chápáno jako parametr popisující růstové podmínky dřeva podobně jako jeho hustota. Změna napětí přitom odpovídá proměnlivosti letokruhů pozorované opticky. Získaná data umožňují určit charakteristické body letokruhu stejně jako parametry, které jsou závislé na klimatických podmínkách.

Klíčová slova: dřevo; letokruhy; mikrotvrdost; průnik; pevnost

Práce je pokusem o analýzu struktury letokruhů smrku pomocí měření mikrotvrdosti dřeva. Zkušební vzorek byl připraven ze dřeva smrku ztepilého (*Picea abies* [L.] Karst.). Hranol měl rozměry 100 × 80 × 20 mm s radiální plochou o rozměrech 100 × 80 mm, která byla následně broušena brusným papírem do zrnitosti 320 tak, aby byla vytvořena hladká plocha pro měření mikrotvrdosti. Vzorky byly při všech měřeních uchyceny ve speciálním polohovatelném svěráku umožňujícím jemný posun ve dvou směrech: kolmo na letokruhy (osa *x*) a rovnoběžně s letokruhy (osa *y*).

Vlastní mikrotvrdost byla měřena na univerzálním laboratorním měřicím přístroji INSTRON 4464 pomocí ocelového trnu s plochým čelem o průměru 0,25 mm. Tento trn byl vtlačován do povrchu dřeva do hloubky 0,3 mm při konstantní rychlosti 1 mm/min. Vzorky byly mezi jednotlivými měřeními posunovány ve dvou směrech – osách *x* a *y*. Posun vzorků ve směru osy *x* byl 0,25 mm, ve směru osy *y* byly vzorky posunovány střídavě v kladném a záporném smyslu osy, vzdálenost přitom byla 0,5 mm. Tímto postupem bylo dosaženo větší hustoty a tedy i přesnosti měření při zanedbatelných změnách vlastností dřeva ve směru vláken.

Výstupem z každého jednotlivého měření byla deformační křivka (obr. 2), která byla základem pro stanovení pevnosti vzorku v místě měření. Tato pevnost byla získána z průsečíku deformační křivky s přímkou rovnoběžnou s kvazi-statickou částí deformační křivky při deformaci 0,02 mm (obr. 2). Souřadnice osy *x* byly zjištěny z polohy jednotlivých měření.

Obr. 3a zobrazuje typické deformační křivky pro jeden letokruh. Pevnost jednotlivých měření, vyjádřená graficky proti souřadnicím osy *x*, charakterizuje průběh pevnosti v letokruzích (obr. 3b). Hodnoty pevnosti z obr. 3b byly následně aproximovány pomocí dvou přímek (v případě letního dřeva) nebo pomocí polynomu druhého řádu (v případě jarního dřeva) – odpovídající rovnice jsou uvedeny v textu obr. 3b. Průsečíky těchto aproximací byly chápány jako body charakterizující letokruh: EB (počáteční bod jarního dřeva a tedy i celého letokruhu definovaný jako průsečík polynomu a klesající přímky předchozího letokruhu), EM (minimální hodnota pevnosti jarního dřeva, resp. minimum aproximačního polynomu), EE (definuje rozhraní mezi jarním a letním dřevem uvnitř letokruhu; průsečík polynomu a stoupající aproximační přímky letního dřeva) a LAP (nejvyšší hodnota pevnosti; průsečík dvou přímek aproximujících hodnoty letního dřeva). Stejný postup byl použit pro všechny letokruhy, výsledný graf je zobrazen na obr. 4. Graf je tvořen hodnotami EE, EB a LAP. Velmi zřetelné jsou rozdíly pevnosti nejen mezi jarním a letním dřevem, ale také mezi různými letokruhy.

Vzdálenost souřadnic *x* mezi dvěmi sousedními body LAP je šířka letokruhu (RW). Vzdálenost mezi sousedními body EE a EB může být označena jako šířka jarního dřeva (EWW), vzdálenost mezi body EB a souřadnicí bodu EE předcházejícího letokruhu zase jako šířka letního dřeva (LWW). Tyto šířky jsou ještě členěny na dvě části (EWW1 a EWW2, resp. LWW1 a LWW2), rozdělené bodem EM, resp. LAP. Tyto základní statistické charakteristiky jsou uvedeny v tab. 1. Obr. 5 ukazuje korelaci hodnot šířek letokruhů RW zjištěných pomocí měření pevnosti a zjištěných opticky.

Tab. 2 obsahuje průměrné hodnoty pevnosti v rámci letokruhu. Hodnoty z tab. 1 a 2 spolu vytvářejí průběh pevnosti „průměrného“ letokruhu testovaného vzorku, průběh zobrazený na obr. 6. Tab. 2 také ukazuje, že pevnost letního dřeva je asi 10× větší než minimální pevnost jarního dřeva a asi 4–6× větší než jeho průměrná pevnost.

Z obr. 7 je patrné, že šířka letokruhu klesá s rostoucím věkem stromu, rozdíly jsou pozorovatelné ale také mezi jednotlivými letokruhy. Důležitým ukazatelem je také Rwr (relativní šířka letokruhu; poměr šířky jednotlivých letokruhů a průměrné šířky), který dosahuje hodnot téměř 0,5. Toto minimum souhlasilo s nejmenšími hodnotami LAP (obr. 7) a s nízkou pevností jarního dřeva (obr. 8 a 9).

Měřením mikrotvrdosti lze zkoumat průběh pevnosti ve dřevě s podobnými výsledky, jakých lze dosáhnout u optických metod. Zřetelné jsou rozdíly mezi pevností jarního a letního dřeva; obě části letokruhu lze určit podle její velikosti.

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