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RADIAL GRADIENT OF MODULUS OF ELASTICITY OF WOOD AND TRACHEID CELL WALLS IN DOMINANT PINE TREES (*PINUS SYLVESTRIS* L.)

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SYNOPSIS. This paper presents results of measurements of radial gradients of density and wood elasticity modulus determined in the tensile test for microtome samples obtained from early- and latewood in annual growth rings of pine trees dominant in the tree stand. On the basis of the results, the elasticity modulus of cell walls has been determined and then analysed as a function of the microfibril angle in tangent tracheid walls.

KEY WORDS: wood density, call wall, microfibril angle

INTRODUCTION

In recent research on wood much attention has been paid to the relationship between the ultrastructure of cell walls and wood properties. The interest in this relationship has been related to the fact that wood density, the feature assumed as most informative about the mechanical performance of wood has appeared not enough reliable. The wood density is not a simple parameter; it depends on wood species, thickness of cell walls, cell diameter, and content of latewood in annual growth rings, chemical composition and wood moisture content. In oven-dry wood, this property is an indicator of the content of wood substance in a unit volume. It has been found that the quality of wood substance does not necessarily remain constant, however, its density remains practically unchanged. Changes in the quality of wood substance depend on variations in the content and distribution of cellulose in cell walls – mainly in the S2 layer. Cellulose formed as microfibrils, in which crystalline regions exhibit great rigidity in the direction parallel to the microfibrils, is responsible for rigidity and strength of cell walls. The arrangement of microfibrils as steep spirals is the optimum for a growing tree (Cave and Walker 1994). In coniferous species, as the wood tissue matures, the mean value of MFA in S2 layer, relative to lengthwise cell axis, tends to decrease (Sahlberg et al. 1997), while the density increases (e.g. Zhang 1998, Alteyrac et al. 2006). Both values show the greatest tendency to change in juvenile tissue. So, mature wood (further from the pith) has better mechanic properties than juvenile wood (near-pith). Besides the genetic factors, the juvenile period of growth depends also on the tree status within the forest stand (e.g. ZOBEL and BUIJTENEN 1989).

Since the wood substance density may be treated as invariant, any change in wood density along the tree radius results mainly from changes in the wall thickness. The S2 layer constitutes 79-86% of thickness of the whole wall (FENGEL and STOLL 1973) and it determines properties of the wood. If the cellulose ordering in the wall was independent of its thickness, the wood strength and elasticity modulus along the grains would change as much as the wood density. However, many studies have shown that wood tensile strength is much higher than the increase in the wood density would suggest. In other words, the wood tensile strength at the same wood density may differ substantially (e.g. BUNN 1981, BAMBER and BURLEY 1983, ZHANG 1997). The same applies to the wood elasticity modulus (COWDREY and PRESTON 1966 after CAVE and WALKER 1994, CAVE 1968, BENDTSEN and SENFT 1986). According to these authors the value of elasticity modulus may increase by 5 or 6 times at the MFA decrease from 40° to 10°. A study of the relation between MFA in tracheid walls and mechanical parameters of wood subjected to strain along the grains clearly shows that the wood and cell walls tensile strength and elasticity modulus are the higher, the smaller the microfibrile angle, yet these relations are not linear (CAVE 1976, DINWOODIE 1981, CAVE and WALKER 1994, REITERER et AL. 1999, GROOM et AL. 2002 a, b). It follows from studies conducted by REITERER et AL. (1999) on spruce wood (*Picea abies* Karst.) that the strength of cell walls of early and late tracheids and their elasticity modulus, determined in the tensile test along grains of microtome wood samples, show similar values on condition that the microfibril angles in these cells are similar. Moreover, an increase in MFA brings about greater deformation at the moment of destruction. The studies carried out by SEDIGHI-GILANI and NAVI (2007) on individual tracheids did not confirm such a direct relationship, but general dependencies of mechanical parameters of wood on MFA were similar.

Since the wood density and MFA affect the value of MOE, in the present study an attempt was made to analyse changes in wood density and elasticity modulus, determined in the tensile test for microtome samples along the grains obtained from the rings of different cambial age. The values of density and MOE were also used to calculate the elasticity modulus of cell walls. Also the MFA values measured in tangential tracheid walls are determined and MOE of cell walls is analysed as a function of MFA. The results are expected to provide information on the relation between the cyclic inhomogeneity of pine (*Pinus sylvestris* L.) wood obtained from trees dominating in even-aged tree stand and its mechanical properties.

MATERIALS AND METHODS

The radial gradient of MOE was determined for pine wood taken from a dominant tree from a group in an even-aged (62 year old) forest stand. The tree selected for studies was characterized by a cylindrical, straight shaft and a regularly spread crown. The tree was 32 cm in diameter at the height of 1.3 m and its height was 24.5 m. At breast height of the tree trunk, a 70-cm-long billet was cut out from the tree, from which then a central balk of 60 mm in width was cut. When the balk was sawn along the pith, from its north section a slab 13 mm in width was cut out in such a way that the annual rings were tangent to its thickness. Then this slab was chipped to make it 10 mm wide in the tangent direction. Having marked the zones of linear course of the annual rings along the length of the slab, two sites were chosen from which two samples of 12 cm in length were cut. The sample slabs were then subjected to plasticization treatment by boiling in distilled water at temperature of 100°C for 35 hours. Afterwards, using a sliding microtome, samples were chipped from the previously marked annual rings in the tangential direction to obtain samples of 200 µm in thickness. Thus shaped samples were arranged in the same sequence they were obtained, placed on filter paper and labelled so as to identify their position in the annual ring. From each ring, depending on its width, from a few to over ten samples were obtained. The diagram illustrating the way the samples were obtained is shown in Figure 1.

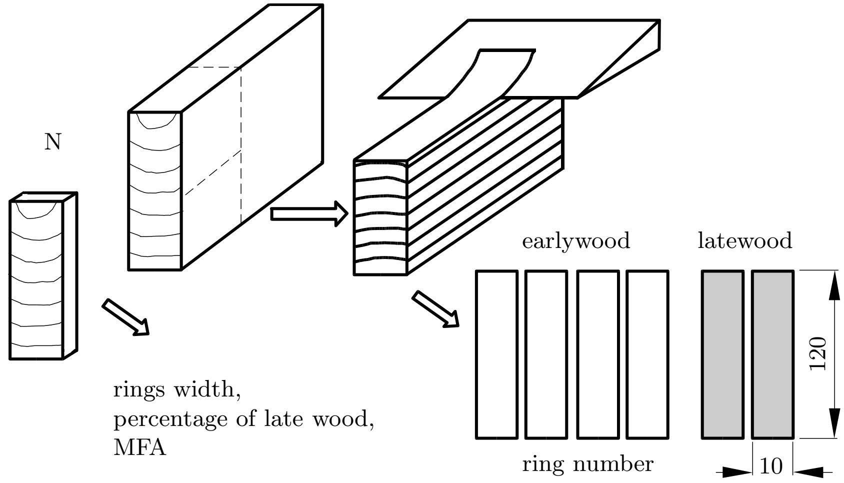

Fig. 1. Schematic presentation of sample preparation

Thus prepared sets of samples were air-conditioned in the laboratory conditions (t = 21°C, RH = 33-41%) to achieve equilibrium moisture content. The width of the samples was measured using a increment meter BIOTRONIK to the accuracy of 0.01 mm, and their thickness by using a micrometer screw to the accuracy of 0.001 mm in the middle of their length and 2 cm from the centre. The length of the samples was measured using a linear rule. Next, each sample was weighed on a laboratory balance to the accuracy of 0.001 g, and then density of wood in each sample was determined. Before the tensile strength measurements, the terminal sections of the samples over a length of about 2 cm were glued to fibreboard pieces of the size 3 mm in thickness, 2 cm in width and 2 cm in length. The attachment of the fibreboard to the mount pieces of the sample prevented the sample crushing in the jaws of the testing machine.

The tensile strength tests were performed on a ZWICK ZO50TH testing machine with the use of an extensometer BTC-EXMARCO.001. After the introduction of the crosswise dimensions of the sample and the base of the extensometer to the computer of the testing machine, the tensile stress was applied at the rate of 0.5 mm/min. The measurements for the samples breaking near the middle of the length were assumed correct. The diagram showing application of the tensile stress and the way of measurement of the deformations caused by tensile strain is presented in Figure 2.

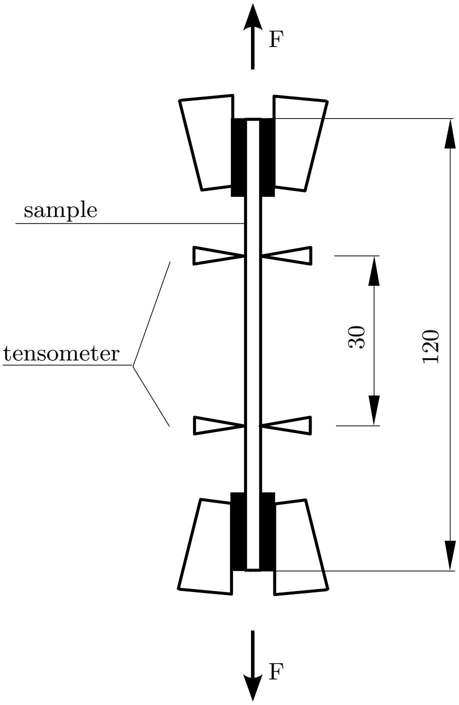

Fig. 2. Schematic presentation of tensile stress application and the way of deformation measurements

From the slab from which samples for the tensile test were obtained, a 1-cm thick strip was also cut out. On this strip first the width of annual growth rings and latewood content in the rings were marked (Fig. 1). Then the strip was divided into three parts, and each of them was held in a 20% solution of $Cu(NO_3)_2$ salt at temperature of 80°C for 24 hours, using a water-bath. After the heating, the material was rinsed with distilled water to stop the action of the salt, and then boiled in distilled water at the temperature of 100°C for 2 hours. Afterwards, from the annual growth rings, from which samples for tensile test were cut out, tangential preparations of about 20 µm thick, were made for microscopic analysis. In these preparations the microfibril angles in tangent tracheids walls were measured with the use of a computer image analyzer. In each growth ring, preparations were cut out at more or less every 0.5 mm. Always, after each preparation was made, its position in an annual growth ring was determined using a Brinell magnifier. In each preparation 20 MFA were measured. On the basis of the obtained results it was possible to correlate the values of the elasticity modulus of tracheid walls with MFA.

RESULTS AND DISCUSSION

At first, the results of radial variations of wood density are discussed since wood density is considered as one of the most dominant factors determining wood mechanical properties. The results are shown in Figure 3 separately for earlywood and latewood zones of selected annual growth rings. The results for the samples obtained from the intermediate zones between earlywood and latewood of the annual rings studied have not been shown. As follows from the figure, the density of earlywood decreases as a function of the ring cambial age in juvenile wood zone. Decrease in wood density with increasing cambial age of annual growth rings has been also reported for other species, e.g. *Tsuga heterophylla* (Panshin et al. 1964), *Picea mariana* (Zhang 1998) and *Larix kaempferi* (Koizumi et al. 2005). This observation may be related to the increase in cross-sectional dimensions of the lumen of early tracheids at this stage of tree growth.

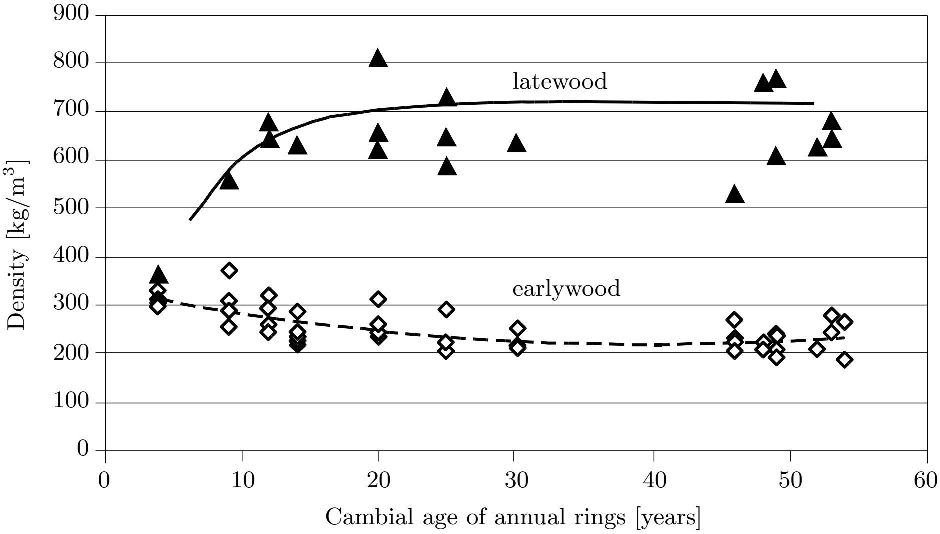

Fig. 3. Changes in the early and latewood density versus the cambial age of annual rings

The density of latewood increases with the cambial age of annual rings till up to ring 20, counted from the pith. In ring 4 the density of latewood is only slightly, ca. 20%, higher than the density of earlywood. In ring 20, the difference in the density between early and latewood is over 2.5 times. In further annual rings the density of latewood compared to that of the early wood increases only slightly. This means that as the tree tissue matures, its cyclic inhomogeneity increases. So mature wood is more inhomogenous than juvenile wood.

The wood density changes along the radius of the tree are accompanied by the changes in the linear elasticity modulus, which is illustrated by the data compiled in Figure 4. However, a thorough analysis of the MOE values indicates that in earlywood MOE is more or less stable with the growing cambial age of annual

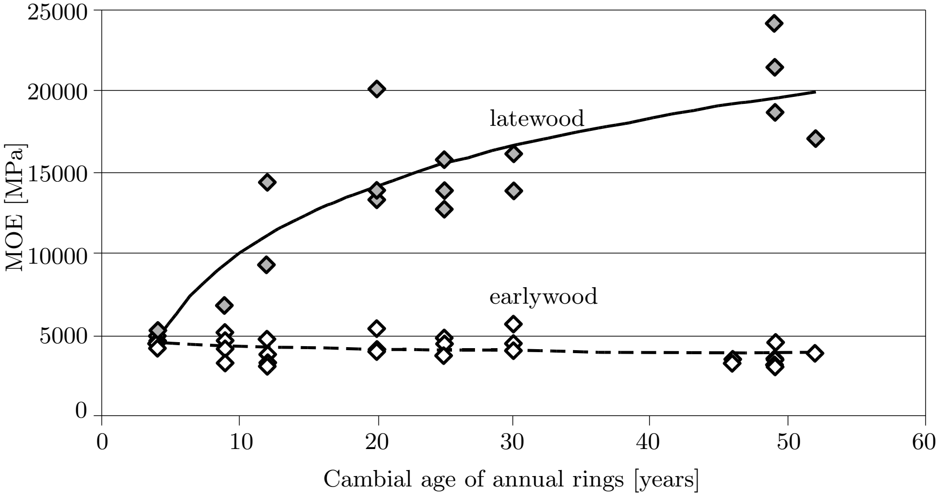

Fig. 4. The longitudinal elasticity modulus of the early and latewood versus the cambial age of annual rings

rings, in spite of a decrease of density in earlywood with increasing cambial age. For latewood the changes in MOE values along the wood radius are greater than those in density. The increase in density of latewood from 365 kg/m^3 in ring 4 to 688 kg/m^3 in ring 49, which is by 1.88 times, was accompanied by the MOE increase on average from 5530 MPa to 21 440 MPa, respectively, i.e. by 3.88 times. So the increase in MOE value in latewood was more than twice greater than the increase in the wood density. This observation confirms earlier reports (e.g. Cave and Walker 1994) that wood density cannot be treated as the only one, universal and reliable descriptor of its mechanical performance. The above observations also indicate a nonlinear character of the MOE relation with wood density; this relation plotted with the use of all results obtained in this paper, is shown in Figure 5.

The measured wood density (ρ) and elasticity modulus of particular samples (MOE) enabled us to calculate the modulus of elasticity of cell walls (MOEc.w.), using the following expression:

$$\text{MCEc.w} = \text{MOE}\,\frac{1500}{\rho}$$

where: 1500 – density of wood substance [kg/m^3],
MOE – elasticity modulus of wood [GPa],
ρ – density of wood in particular samples [kg/m^3].

The above way of calculating the elasticity modulus of cell walls was used by many researchers working in the field (e.g. Mark 1967, Reiterer et al. 1999, Bergander and Salmen 2002, Groom et al. 2002 a, b, Sedighi-Gilani and Navi 2007). Analogously, the actual stress in cell walls during the susceptibility test of single wood fibres (tracheids) to creep in stable and varied moisture content was calculated (Kojima and Yamamoto 2004, 2005). Thus calculated values of the elasticity modulus of tracheid walls of early and latewood as a function of the

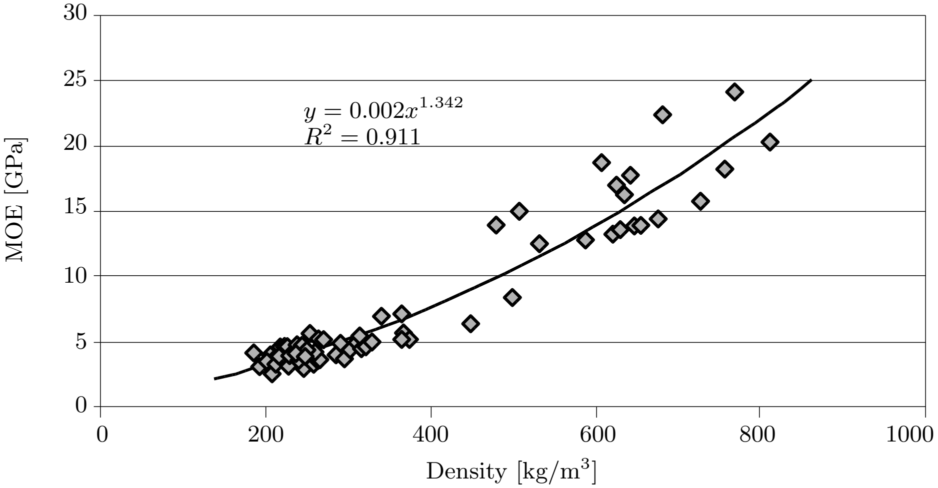

Fig. 5. The elasticity modulus of wood versus its density

cambial age of annual rings are shown in Figures 6 and 7. These figures show only the results for the samples to which it was possible to assign the average value of microfibril angle (MFA) calculated on microscopic preparations. According to the results presented, the elasticity modulus of cell walls is not a constant value. In the early tracheids the elasticity modulus changes in the range 21.2 GPa (growth ring 9) to 31 GPa (growth ring 30). For late tracheids this value ranges from 22 GPa in ring 4 to 53.6 GPa in ring 53.

As follows from the figures, the reason for such a significant change in the elasticity modulus for early and late tracheid walls is the change in MFA. The trends illustrated by the results shown in these figures mirror each other images. The

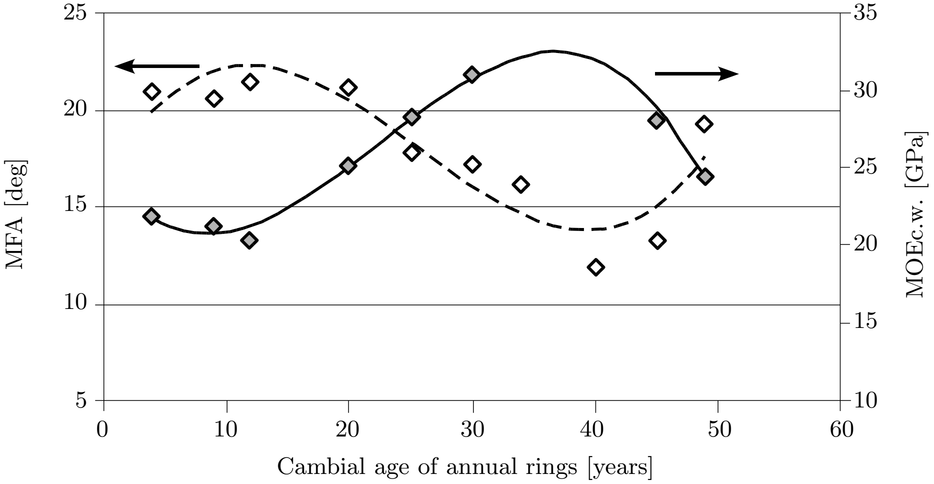

Fig. 6. Radial variation in the elasticity modulus of tracheid walls and MFA of earlywood

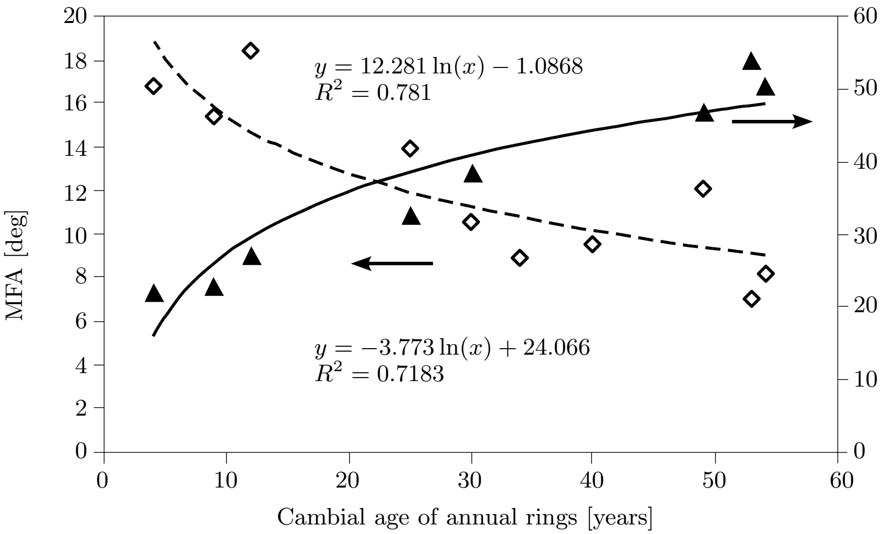

Fig. 7. Radial variation in the elasticity modulus of tracheid walls and MFA of latewood

Young modulus of cell walls also increases with decreasing MFA. The relation between these two parameters, shown in Figure 8, can be approximated with a linear function, for the range of measured MFA values. According to the determination coefficient of this relation, the Young modulus of cell walls in the axial direction is in 72% dependent on MFA. Taking into account the fact that MFA values assigned to particular preparations are the averages determined only for the tangent tra-

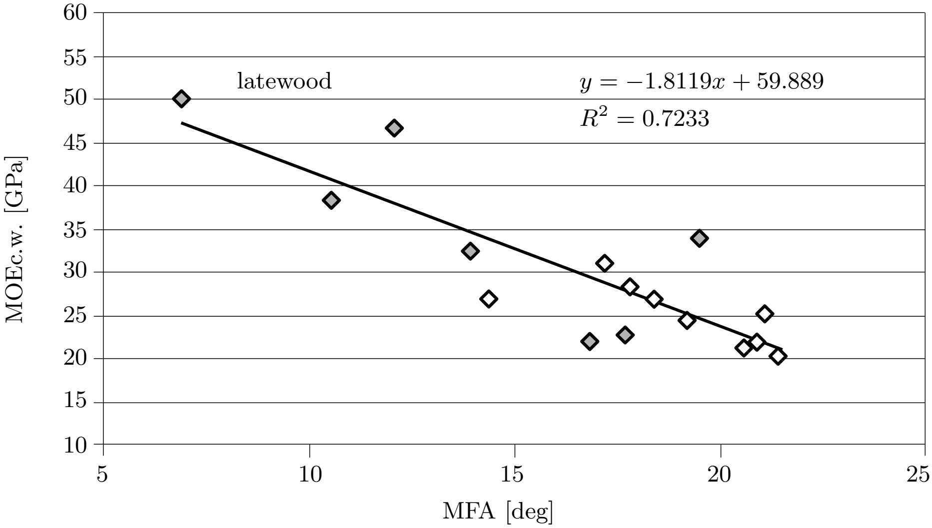

Fig. 8. Empirical relation between the elasticity modulus of tracheid cell walls in early and late wood and the microfibril angle

cheid walls, the correlation between the values is very high. In reality, MFA in thin microtomic slices changed in quite a wide range. The variability coefficient of MFA each time determined from 20 measurements, ranged from 7 to 22%, depending on the site from which a given preparation was obtained. Since the variation in the values of MFA in single tracheids (Anagnost et al. 2002) and in different tracheids in the tangential samples is known (e.g. Fabisiak and Moliński 2007 a, b), there is no need to present the data here.

The MFA variation in different tracheids, equidistant from the border of the previous annual growth ring (which means they were formed practically at the same time of the vegetation period) indicate the profound inhomogeneity of wood tissue, which is responsible for the poor correlation of the parameters compared. Sedighi-Gilaniand and Navi (2007) have shown that application of tensile strength to single tracheids leads to local destruction of the matrix incrusting the cellulose skeleton, which leads to decrease in the value of the elasticity modulus. Hence the value of the elasticity modulus must depend on such local defects in the cell wall. In view of the above findings, it seems fair to conclude that formation and localization of deformations in cell walls and the related decrease in MFA must depend on their distribution in strained samples, mainly in the tangent tracheid walls which are most strained (Reiterer et al. 1999).

It should be noted that the values of the elasticity modulus of cell walls, both in early and late tracheids, determined in the present study fully correspond to the values determined, within the same MFA range, for the wood tracheids of Radiata pine (Cave 1996).

CONCLUSIONS

1. In the juvenile tissues, the earlywood density decreases, while the density of latewood increases. In mature wood (above ring 20) the densities in the early and latewood stabilise. Thus, with increasing maturity of the wood tissue its cyclic inhomogeneity increases.
2. The elasticity modulus of earlywood is practically independent of the cambial age of annual rings, while that of latewood increases with increasing cambial age of annual rings.
3. The longitudinal elasticity modulus of cell walls of early tracheids varies in the range 21-31 GPa, while in cell walls of late tracheids, the value of this modulus increases with increasing cambial age of annual rings from 22 GPa to 53,6 GPa. The main reason for such a significant change in the elasticity modulus of early and late tracheid walls is the change in MFA. The correlation between MOEc.w. and MFA can be approximated by a linear equation with a high determination coefficient ($R^2 = 0.723$).

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