Tensile Properties Along the Grains of Earlywood and Latewood of Scots Pine (*Pinus sylvestris* L.) in Dry and Wet State

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Mechanical parameters of Scots pine wood (*Pinus sylvestris* L.*)* of low (about 8%) and high (higher than the fiber saturation point) moisture content (MC) subjected to tensile stress along the grains were studied. The measurements were performed for microtome samples sliced from either earlywood or latewood and for samples containing both earlywood and latewood. The effect of MC on the mechanical properties of earlywood and latewood of Scots pine was different. The MC was found to have greater influence on the tensile strength and modulus of elasticity in latewood than in earlywood, but its effect on strain at failure was greater in earlywood. As determined individually for earlywood and latewood, the tensile strength, modulus of elasticity, and the strain at failure that were calculated from the rule of mixtures (the weighted mean for earlywood and latewood) did not differ significantly from the values found in the samples containing both zones. This similarity was observed at low and high MC.

Keywords: Earlywood; Latewood; Moisture content; Pinewood; Rule of mixtures; Tensile stress

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INTRODUCTION

Many wood properties vary within the extremely diverse and hierarchic structure of wood (Zobel and Buijtenen 1989). Wood properties are directly related to the wide variety of their applications. Due to its excellent mechanical properties, wood is a widely used construction material. The main factors influencing the mechanical properties of wood are the structure and technological quality of its cell walls; these features determine the unique mechanical properties of wood in the axial direction, even when the wood density is low (Gibson and Ashby 1997). In practice, the effect of the microstructure on the mechanical properties of wood is evaluated *via* wood density (ρ_w). This parameter is universal, and in the absence of water, it illustrates the overall packing of wood substance (ρ_{ws}) in a unit volume (ρ_w/ρ_{ws}). Given the mechanical parameters of the cell walls and density, it is possible to predict wood mechanical strength along the grains (Gibson and Ashby 1997).

The quality of wood substance is not constant, although its density practically remains constant. The variation in wood substance quality is related to changes in the amount of basic chemical components—cellulose, hemicelluloses, and lignin—and their distribution within particular wood cell wall layers. Cellulose microfibrils consist of crystalline regions with high rigidity in the direction parallel to the microfibrils, and they are responsible for the strength and rigidity of cell walls in the longitudinal direction. Therefore, the tensile strength and elasticity constants of earlywood in this direction, are usually lower than those measured for latewood (Wimmer *et al*. 1997; Cramer *et al*. 2005; Moliński and Krauss 2008; Krauss 2010; Krauss *et al*. 2011; Roszyk 2014). In earlywood generated in the beginning of the vegetation season, the microfibril angles (MFA) in the S2 layer are generally higher than in latewood generated at the end of the vegetation season (Preston 1934; Abe *et al*. 1992; Sarén *et al*. 2001, 2004; Anagnost *et al*. 2002; Fabisiak and Moliński 2007a, 2007b; Krauss 2007, 2010). If the MFA values are small, then cellulose determines the behavior of the wood under tensile stress. With increasing MFA, the matrix encrusting the cellulose skeleton (hemicelluloses and lignin) has a greater influence on the mechanical properties of cell walls (Bergander and Salmén 2002; Barnett and Bonham 2004; Gindl and Schöberl 2004; Salmén 2004).

The main differences between earlywood and latewood upon tensile stress application can be attributed to their different mechanical parameters and the dependence of these parameters on the moisture content (MC). In general, an increase in bound water content causes a smaller decrease in the tensile strength or modulus of elasticity in earlywood than in latewood. As reported already by Biblis (1969), the tensile strength along the grains of earlywood of *Pinus taeda* in the wet state was only by 13% lower than in the air-dry state, while that of wet latewood was by 30% lower than in the air-dry state. In similar studies (Helińska-Raczkowska and Raczkowski 1979) on the earlywood and latewood of *Pseudotsuga menziesii* Franco, it was found that the tensile strength along the grains in earlywood was practically the same in the wet ad air-dry states (10% MC), while that of latewood was by about 40% lower in the wet state than in the air-dry state. The modulus of elasticity of the wood in the wet state was by 20% smaller in earlywood zone and by 40% in latewood zone than in the air-dry state. Recently, the strain at failure in earlywood was shown to be greater with higher MC (Roszyk *et al*. 2013; Roszyk 2014). For latewood, there was no clear influence of MC on this parameter. The different responses of earlywood and latewood to the MC in cell walls are interpreted in terms of ultrastructure (Kojima and Yamamoto 2004; Roszyk 2014) because the above-mentioned authors reported that the decrease in the modulus of elasticity with its increasing MC became greater as the MFA in the tracheid walls became smaller.

Although much research work has been focused on tensile stress in either earlywood or latewood, it has been unclear whether there is a relationship between their individual mechanical parameters and the parameters of entire annual rings (earlywood and latewood together). In this comparative study, this potential relationship was examined.

EXPERIMENTAL

Microtome cuboidal samples were cut to a length (*L*) of 90 mm and a cross-section of 10 mm (tangentially, *T*) × 0.25 mm (radially, *R*) for tangent samples and 0.25 mm (*T*) × 10 mm (*R*) for radial samples. These dimensions were used in previous studies (Helińska-Raczkowska and Raczkowski 1979; Robson 1989; Reiterer *et al*. 1999; Moliński and Krauss 2008; Krauss 2010; Roszyk *et al*. 2010, 2012, 2013; Roszyk 2014).

Pine wood (*Pinus sylvestris* L.) samples were obtained from a center plank with a linear arrangement of fibers along the radial plane. The plank was divided into smaller elements of 90 mm in length according to the scheme shown in Fig. 1. On the front surface of these elements the widths of annual rings and the widths of latewood zones were measured to the accuracy of 0.1 mm with the use of a Brinell Eschenbach glass (Karlsruhe, Germany). The measurements permitted determination of the contribution of latewood in particular annual rings. The wood was plasticized by boiling in distilled water for 30 h, and a series of tangent samples were sliced by a sledge microtome from the earlier selected annual rings. Radial samples were from the same annual rings.

All samples were cut from annual rings chosen on the basis of their widths and their latewood content. They were sliced from annual rings with high latewood content, such that a few tangent samples from the earlywood and latewood zones were obtained.

Both tangent and radial samples were divided into two groups. One group was used for measurements after air-drying, with a MC of about 8%, while the other group was used for wet measurements, where the MC was greater than the fiber saturation point (FSP). The samples to be measured in the air-dry state, of MC of about 8%, were stored in a laboratory in which the air temperature was 20 ±1°C, while the relative air humidity was 40 ±2%. The samples to be measured in wet state were stored in distilled water.

After securing the desired MC in the samples, the thickness was measured to an accuracy of 0.001 mm with a micrometric screw (Mitutoyo, Kawasaki, Japan), and the width was measured to an accuracy of 0.1 mm using a Brinell glass. To calculate the wood density, the length of the samples with 8% MC was measured to an accuracy of 1 mm with a linear ruler, and their mass was measured to the nearest 0.0001 g with an analytical balance (Radwag, Radom, Poland).

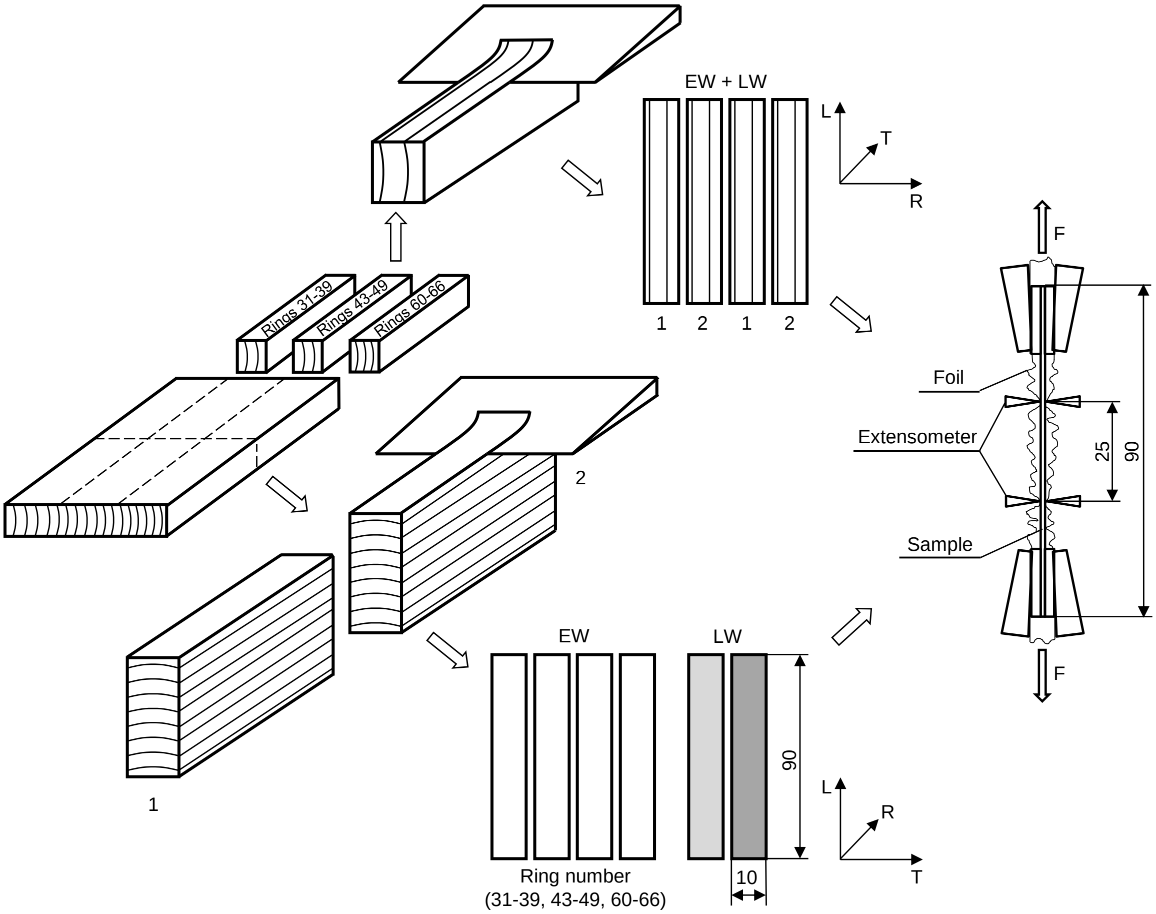

Fig. 1. Sample preparation and tensile stress application (1: 8% MC; 2: MC > FSP; EW: Earlywood; LW: Latewood)

Prior to the tensile stress application, the ends of the samples over the length of 2 cm were covered with pieces of hardboard of 3-mm thickness and 2-cm width, which were glued with polyacetatevinyl glue. For the samples of elevated MC, single-component waterproof polyurethane glue was used. These pieces of hardboard protected the samples against damage from the testing machine mounts (Moliński and Krauss 2008; Krauss 2010; Roszyk *et al*. 2010, 2012, 2013; Roszyk 2014).

Tensile tests were performed on a Zwick ZO50TH testing machine (Ulm, Germany) with a Zwick BTC-EXMACRO.001 extensometer at the rate of 0.5 mm/min. Only the results obtained for samples that were broken more or less at the middle of their length were assumed as correct. To prevent the samples from drying out during the tests, they were placed in special foil envelopes. The envelopes were made in the laboratory using a Clatronic FS 3261 bag sealer (Kempen, Germany). After placing each sample in an envelope, the excess air was removed, and the envelope was hot sealed and closed. Because the envelopes were larger than the samples, they had no impact on the strain and deformation being measured (Roszyk *et al*. 2013; Roszyk 2014).

The samples studied were subjected to measurements of tensile strength, modulus of elasticity and strain at failure (*i.e*. at the tensile strength). The actual MC of each sample was measured immediately after breaking by the gravimetric method.

RESULTS AND DISCUSSION

The mean latewood content in the annual rings and the wood densities are given in Table 1. The separate densities of earlywood and latewood were calculated as the mean values from all of the tangent samples sliced from particular zones of the annual rings. The density values were determined for each variant of the study for 20 radial samples, and the density values were calculated as weighted means of those determined for earlywood and latewood according to Eq. 1, otherwise known as the rule of mixtures (Gibson and Ashby 1997),

$$\boldsymbol{\rho}_{\mathbf{w}} = \boldsymbol{\rho}_{\mathbf{ew}} \times \boldsymbol{u}_{\mathbf{ew}} + \boldsymbol{\rho}_{\mathbf{lw}} \times \mathbf{u}_{\mathbf{lw}} \tag{1}$$

where ρ_{ew} and ρ_{lw} stand for the density of earlywood and latewood, respectively, and u_{ew} and u_{lw} stand for the earlywood and latewood content, respectively, in annual rings.

Table 1. Parameters of Wood Selected for the Study

Ring Number	Percentage Latewood (%)	Density, ρ (kg/m^3)				
		EW		LW	EW + LW	
					Measured	Calculated
31 to 39	36.7	X	352	655	476	463
		±S	26	66	26	
		V (%)	8.3	11.2	5.5	
43 to 49	41.7	X	332	717	509	493
		±S	50	85	16	
		V (%)	14.9	11.9	3.2	
60 to 66	43.5	X	290	669	467	455
		±S	58	96	7	
		V (%)	19.9	14.3	1.6	

EW: Earlywood, LW: Latewood, X: Mean Value, ±S: Standard Deviation, V: Variation Coefficient

The latewood density ranged from 1.9 (annual rings 31 to 39) to 2.3 (rings 60 to 66) times higher than that of earlywood; for all rings, it was 2.1-fold higher. The density of the wood determined for the entire selection of annual rings was slightly higher than the actual weighted means of the earlywood and latewood, from 2.6% for rings 60 to 66 to 3.1% for rings 43 to 49.

Similarly, the tensile strength, modulus of elasticity, and strain at failure were determined for the samples of 8% MC (Table 2) and MC higher than FSP (Table 3). The tensile strength and modulus of elasticity of latewood of MC 8% were on average nearly 3 times higher than the corresponding values for earlywood of the same MC. The difference was smaller for wet wood (2.3-fold higher in latewood). The strain at failure values for the earlywood and latewood were similar, except for wet earlywood, which had the highest recorded value.

Table 2. Mechanical Parameters of Wood at Low Moisture Content (8%)

Parameter	Ring Number	EW		LW	EW + LW	
					Measured	Calculated
Tensile Strength, TS (MPa)	31 to 39	X	51.9	150.1	99.3	87.9
		±S	9.5	30.2	8.9	
		V (%)	18.3	20.1	8.9	
	43 to 49	X	56.9	136.0	109.5	89.9
		±S	6.3	32.1	9.2	
		V (%)	11.1	23.6	8.4	
	60 to 66	X	52.2	174.4	100.0	105.4
		±S	10.1	35.1	12.2	
		V (%)	19.3	20.1	16.7	
Modulus of Elasticity, MOE (GPa)	31 to 39	X	6.5	14.9	10.6	9.6
		±S	1.0	3.0	0.9	
		V (%)	15.8	20.3	8.3	
	43 to 49	X	5.4	17.0	11.8	10.2
		±S	0.5	3.1	0.5	
		V (%)	10.3	18.6	8.4	
	60 to 66	X	6.0	20.0	11.3	12.1
		±S	1.2	3.6	0.6	
		V (%)	20.0	17.8	6.0	
Strain at Failure, ε (%)	31 to 39	X	0.84	1.31	1.03	1.01
		±S	0.17	0.20	0.07	
		V (%)	20.1	15.4	6.5	
	43 to 49	X	1.18	0.96	1.05	1.09
		±S	0.13	0.28	0.07	
		V (%)	11.1	29.2	6.8	
	60 to 66	X	0.98	1.01	0.78	0.99
		±S	0.15	0.20	0.12	
		V (%)	16.0	20.3	16.0	

EW: Earlywood, LW: Latewood, X: Mean Value, ±S: Standard Deviation, V: Variation Coefficient

In general, the results obtained for earlywood and latewood at 8% MC were characterized by smaller variation coefficients than those for wet wood (MC > FSP). The variation coefficients determined were similar to those reported by Jeong *et al.* (2009). However, the values that were obtained for the radial samples, including both earlywood and latewood, were the most similar. The average tensile strength and modulus of elasticity determined for these samples were higher than those calculated from the weighted averages of earlywood and latewood treated separately. This relationship was observed for wood of low and high MC. Irrespective of the MC, the mean tensile strength along the grains for all of the annual rings was 8.5% higher than the mean weighted tensile strength measured for earlywood and latewood separately. Similarly, the modulus of elasticity measured for the dry wood was about 5.5% higher than the calculated one, while for wet wood it was 10.5% higher than the calculated value. This relationship did not hold for the values measured for the annual rings 60 to 66, for which the tensile strength and modulus of elasticity in both dry and wet samples were lower than those calculated from the weighted means.

Table 3. Mechanical Parameters of Wood at High Moisture Content (MC > FSP)

Parameter	Ring Number	EW		LW	EW + LW	
					Measured	Calculated
Tensile Strength, TS (MPa)	31 to 39	X	35.0	74.3	60.1	49.4
		±S	8.9	7.4	8.8	
		V (%)	25.5	20.7	14.6	
	43 to 49	X	39.9	73.9	58.8	54.1
		±S	9.2	16.0	6.8	
		V (%)	23.2	21.6	11.5	
	60 to 66	X	31.5	85.9	54.6	55.2
		±S	6.9	19.3	7.1	
		V (%)	21.8	22.4	20.6	
Modulus of Elasticity, MOE (GPa)	31 to 39	X	4.2	9.0	6.5	6.0
		±S	1.2	2.1	1.3	
		V (%)	28.9	23.3	20.1	
	43 to 49	X	3.6	9.0	6.8	5.9
		±S	0.5	1.8	1.3	
		V (%)	13.6	20.8	19.8	
	60 to 66	X	3.7	8.8	6.5	5.9
		±S	1.0	2.0	1.7	
		V (%)	28.3	22.9	26.2	
Strain at Failure, ε (%)	31 to 39	X	1.19	0.65	1.33	0.99
		±S	0.40	0.23	0.40	
		V (%)	33.5	36.7	30.4	
	43 to 49	X	1.40	0.92	1.07	1.20
		±S	0.44	0.26	0.27	
		V (%)	31.8	28.0	25.2	
	60 to 66	X	1.23	1.16	0.67	1.20
		±S	0.36	0.45	0.18	
		V (%)	29.5	39.4	27.3	

EW: Earlywood, LW: Latewood, X: Mean Value, ±S: Standard Deviation, V: Variation Coefficient

The weighted mean values of the strain at failure calculated for earlywood and latewood in solid wood were in general higher than those measured for radial samples, for dry wood by about 8% and for wet wood by about 10%. However, in the samples from the annual rings 31 to 39, the opposite result was observed for wet wood.

The difference in the tensile strength and the modulus of elasticity between the dry earlywood and latewood was greater than the difference in their densities, which confirms that parameters other than density influence the mechanical parameters of wood. The most important of these other parameters is the MFA (Cave 1976; Dinwoodie 1981; Cave and Walker 1994; Reiterer *et al*. 1999; Groom *et al*. 2002; Krauss *et al*. 2011). For the same reason, the parameters determined for the tangent samples varied more than those determined for the radial samples. The MFA values were found to vary significantly along the width of individual annual rings, especially in mature wood; this factor has been taken into account (Abe *et al.* 1992; Lichtenegger *et al.* 1999; Anagnost *et al.* 2002; Fabisiak and Moliński 2007a, 2007b; Fabisiak *et al.* 2008, 2009). Representative changes in the mean value of MFA in the walls of tracheids of mature Scots pine were calculated as a function of their position in a given annual ring (Fig. 2). After visualization of microfibrils in cell walls, MFA measurements were made on tangentially sliced microscopic preparations of about 20 µm in thickness with the help of a computer image analyzer. The method of measurement has been described in earlier works (Moliński and Krauss 2008; Krauss 2010; Roszyk *et al.* 2010, 2012).

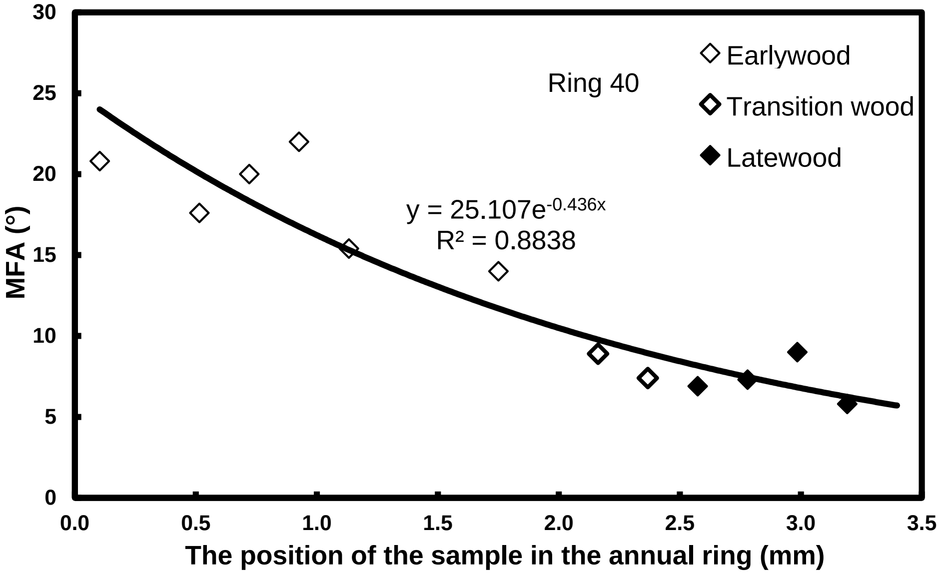

Fig. 2. MFA values in *Pinus sylvestris* L. mature wood annual rings (based on Roszyk 2014)

The relationship between tensile strength and modulus of elasticity determined along the grains for earlywood and latewood in wet and dry states is a result of the already known effect of moisture on these parameters for earlywood and latewood (Biblis 1969; Helińska-Raczkowska and Raczkowski 1979; Roszyk *et al.* 2013; Roszyk 2014). In earlywood, the increase in the MC from 8% to the wet state causes a decrease in the values of tensile strength and modulus of elasticity by about 30%. Upon the same increase in MC in latewood, the analogous decrease reached even 50%. For the samples comprising earlywood and latewood and subjected to tensile stress, the decrease in these parameters was over 40%. In view of the fact that in these samples the contribution of latewood was smaller than that of earlywood (Table 1), the above results imply that the effect of MC on the mechanical parameters studied is determined by the latewood. The greater influence of the MC on the tensile strength and elasticity of latewood than on those of earlywood should be related to the orientations of microfibrils in these wood tissues. In the walls of the earlywood tracheids, the mean MFA is much greater than in the walls of the latewood. At a low MFA, the mechanical parameters are determined mainly by the cellulose system. The matrix encrusting the cellulose skeleton has a greater impact at a higher MFA (Via *et al.* 2009; Klüppel and Mai 2012). According to Dong *et al.* (2010), the mechano-sorptive effect of wood is greater with smaller MFA values. However, the mechanism of interaction between the matrix (hemicelluloses and lignin) and the cellulose skeleton has not been fully established yet (Jin *et al*. 2015). Nevertheless, the results of this study are fully consistent with previous observations. The different effect of the MC on earlywood and latewood explains the greater difference in the tensile strength of these zones in dry wood compared with wet wood.

The results obtained for the strain at failure were also consistent with earlier reports. The values of the strain at failure at low MC are comparable for earlywood and latewood (Roszyk 2014). With increasing MC, the strain at failure increases only for the wood of a relatively high MFA, which explains why the results for wet earlywood obtained in this paper were on average higher than the other values. The plasticization of matrix seems to affect the strain at failure of wet samples, but it has little effect on this parameter for dry wood samples (Klüppel and Mai 2012).

When the experimental values of wood containing earlywood and latewood zones were compared with the values calculated from the rule of mixtures, the differences were negligible. Thus, the applicability of the law of mixtures to the mechanical parameters of earlywood and latewood subjected to tensile stress was confirmed. The observed and expected greater variation of results obtained only for earlywood or only for latewood in comparison to that obtained for the samples comprising both earlywood and latewood hampered deeper analysis. In a similar study performed for spruce wood (Lanvermann *et al.* 2014), the tensile strength decreased when the MC increased from 9.3% to 23.7%. Because of the high variation in the results, it was impossible to find any correlation between the values of the parameters and the position in the annual ring. The authors hope that this study will stimulate further research work aimed at explanation of the possible synergistic effect of the earlywood and latewood on mechanical parameters of solid wood in different states of MC.

CONCLUSIONS

1. The influence of moisture content (MC) on the mechanical properties of earlywood and latewood subjected to tensile stress is different. In latewood this influence is more pronounced on the tensile strength and modulus of elasticity, while in earlywood the greater effect is observed on the strain at failure. The differences can be explained by smaller MFA values in latewood tracheids than in earlywood ones.
2. The effect of MC on the mechanical properties of solid wood of Scots pine (comprising both earlywood and latewood) subjected to tensile stress is determined by the changes in the properties of latewood than in those of earlywood, despite the smaller contribution of the former.
3. The tensile strength of wood along the grains, its modulus of elasticity, and the strain at failure in this direction, calculated according to the rule of mixtures for earlywood and latewood, do not differ significantly from the experimentally determined values for wood comprising of both these zones. This observation applies to the wood of both low and high MC.

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