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The effect of ultrastructure and moisture content on mechanical parameters of pine wood (*Pinus sylvestris* L.) upon tensile stress along the grains

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Abstract: Selected mechanical parameters of earlywood and latewood from *Pinus sylvestris* subjected to tensile stress were measured as a function of moisture content. Microfibril angles (MFAs) to the longitudinal axis of the cell in the S2 layer of tangent cell walls in the same wood samples were also measured. The tensile strength and modulus of elasticity of earlywood were practically independent of its moisture content, while the same parameters of latewood significantly decreased as its moisture content increased in the hygroscopic range. For earlywood, the strain at break was greater the higher its moisture content. For latewood, no notable effect of moisture content on the strain at break was observed. The influence of moisture content on mechanical parameters measured upon tensile stress was proven to be determined by mean MFA values. This influence was the most pronounced for low MFA values, up to about 13°, while for mean MFAs from 13° to 22° the tensile strength and modulus of elasticity were practically constant and independent of moisture content.

Key words: Earlywood, latewood, microfibril angle, moisture content, pine wood, tensile stress

1. Introduction

It has been known for a long time that the effect of moisture on mechanical parameters of wood during tensile stress is different for early and latewood. As follows from the studies from the 1960s on pine wood (*Pinus taeda* L.), an increase in the moisture content from an air-dry state to a wet state leads to a decrease in the tensile strength of only 13% for earlywood and as much as 30% for latewood (Biblis, 1969). According to the measurements performed on Douglas-fir wood (*Pseudotsuga menziesii* Franco), the tensile strength of earlywood in an air-dry state is practically the same as in a wet state, and its linear modulus of elasticity in a wet state is about 20% lower than in an air-dry state. Specific work to ultimate load of wet earlywood can be 16% higher than that of air-dry wood. The tensile strength and modulus of elasticity of wet latewood are over 40% lower and the specific work to ultimate load is 50% lower than their correspondents in air-dry latewood (Helińska-Raczkowska and Raczkowski, 1979). Recently, Roszyk et al. (2013) showed that for, pine wood (*Pinus sylvestris* L.), the main mechanical parameters of earlywood subjected to tensile stress (immediate strength, modulus of elasticity, stress at the proportional limit) are practically independent of its moisture content. However, they reported that wet earlywood becomes damaged at a higher strain than air-dry wood. Although the main parameters of the air-dry latewood subjected to tensile stress along the grains are much higher than in the wet state, the wood is damaged at comparable values of strain.

The reasons for different response to changes in the mechanical parameters of early- and latewood subjected to tensile stress along the grain depending on the moisture content should be probably looked for in the ultrastructure. As demonstrated for earlywood of Sugi wood in Japan (*Cryptomeria japonica* D.Don), the arrangement of microfibrils in the S2 layer of the secondary cell wall with respect to its longitudinal axis determines the influence of the effect of moisture content on modulus of elasticity (Kojima and Yamamoto, 2004a). For wood with low microfibril angles (MFAs), reduction in the modulus of elasticity with increasing moisture content of the wood is much greater than that of wood with intermediate MFA values. For low MFAs, the performance of wood subjected to tensile stress is determined by cellulose in the form of microfibrils in which the crystalline regions show high stiffness in the direction parallel to the microfibrils. With increasing MFAs, the effect of the matrix encrusting the cellulose skeleton, i.e. hemicelluloses and lignin, on the mechanical properties of cell walls increases (Bergander and Salmén, 2002; Barnett and Bonham, 2004; Gindl and Schöberl, 2004; Salmén, 2004).

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It is known that in the walls of cells produced at the beginning of the vegetation period (earlywood), the MFA is usually greater than in latewood (Preston, 1934; Abe et al., 1992; Sarén et al., 2001, 2004; Anagnost et al., 2002; Fabisiak and Moliński, 2007a, 2007b; Krauss, 2007, 2010). Thus, the longitudinal tensile strength and elasticity constants in latewood are usually higher than those in earlywood (Wimmer et al., 1997; Moliński and Krauss, 2008; Krauss, 2010).

In view of the above, we investigated the effect of ultrastructure on the fundamental mechanical parameters of woods of different moisture contents subjected to tensile strength along the grains. The results are expected to provide valuable information on the phenomenon of mechanical strength upon moisture adsorption and on interpretation of the hygro-mechanical behavior of wood (hygro-mechanical wood creep) (Kojima and Yamamoto, 2004b, 2005; Roszyk et al., 2010, 2012).

2. Materials and methods

2.1. Sample preparation

Measurements of the fundamental mechanical parameters of wood of different moisture contents subjected to tensile stress were made on microtomed samples of 90 × 10 × 0.25 mm (L × H × W) in size. Similar samples were used in earlier studies on similar problems (Helińska-Raczkowska and Raczkowski, 1979; Robson, 1989; Reiterer et al., 1999; Moliński and Krauss, 2008; Krauss, 2010; Roszyk et al., 2010, 2012, 2013).

The subject of measurements was pine wood (*Pinus sylvestris* L.) obtained in the form of a heart plank of tangent thickness of 60 mm and length of 70 cm, characterized by a linear arrangement of fibers in the radial plane. The heart plank was cut along the pith; from its northern part, a board of the tangent thickness of 10 mm was obtained. From the front of the board a strip of wood of 10 mm in length was cut off for measurements of macrostructural parameters and MFA. The remaining part of the board was divided into laths of 90 mm in length and 45 mm in width, and each of the laths was labeled. After wood plasticization was achieved by boiling in distilled water for 30 h, samples from previously selected annual rings were sliced off with a sledge microtome. Finally, about 100 samples were obtained from earlywood and another 100 from latewood. The process of sample preparation is given in Figure 1. The annual rings from which the samples were cut out were chosen on the basis of their widths along the ray and the proportion of latewood. Microtomed samples were obtained from mature tissue and from the annual rings wide enough that a few samples could be obtained from the earlywood and latewood (rings: 21, 38, 39, 40, 62).

2.2. Wood preparation for tensile test

We prepared 4 samples neighboring along the ray in a given ring and equally distant from the border of the preceding ring. Each of the samples was prepared to have different moisture content by the following treatment:

1) Moisture content = approximately 8%, conditioned at laboratory [temperature (T) = 20 °C, relative humidity (RH) = 42%];
2) Moisture content = approximately 18%, conditioned over oversaturated water solution of KCl (T = 20 °C, RH = 85%);
3) Moisture content = approximately 25%, conditioned over oversaturated water solution of K_2SO_4 (T = 20 °C, RH = 98%);
4) Moisture content ≥ fiber saturation point (FSP), soaking in distilled water.

To reach the target moisture content, the transverse sizes of the samples were measured. Thickness was measured to the accuracy of 0.001 mm by a micrometer screw, and width to the accuracy of 0.1 mm by a Brinell glass. So as to calculate wood density, 8% moisture content

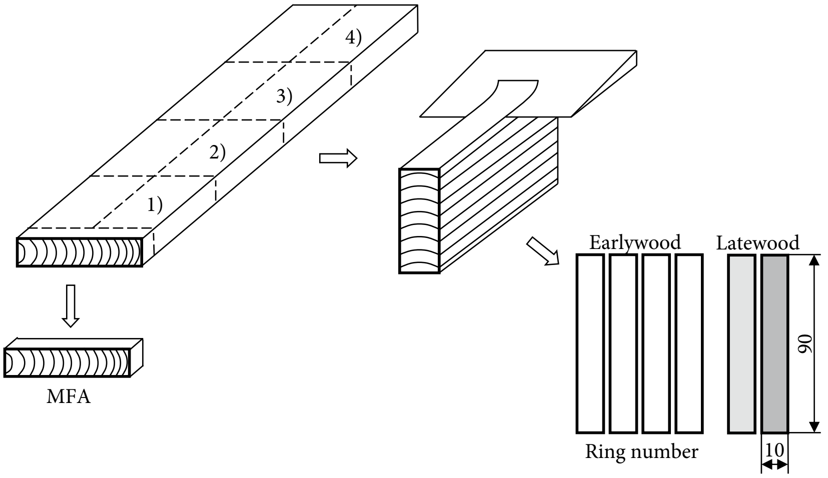

Figure 1. Sample preparation.

samples were also subjected to measurements of length to the accuracy of 1 mm by a measuring rule and mass to the accuracy of 0.0001 g by analytical balance. Before tensile stress application, the ends of the microtomed samples were protected against damage in a mounting gripper (Moliński and Krauss, 2008; Krauss, 2010; Roszyk et al., 2010, 2012, 2013). Pieces of hardboard of 2 cm in length, 3 mm in thickness, and 2 cm in width were glued to the ends of the samples with polyvinyl acetate glue or single-component polyurethane water-repellent glue for the samples of higher moisture content.

2.3. Measurement of mechanical parameters

Tensile strength tests were made on a testing machine (ZWICK ZO50TH) with the use of a BTC-EXMARCO.001 extensometer (Figure 2). The testing machine computer was fed with the transverse size of a given sample and the extensometer base (25 mm) and then the tensile stress was applied at the rate of 0.5 mm/min. We only measured the samples broken in more or less the middle of their length. To prevent the samples from drying, they were kept in special foil envelopes. When the sample was placed inside the envelope, the excess air was removed and the envelope was sealed by a welding machine. The envelopes were larger than the samples to make sure that they did not influence the stress or strain (Roszyk et al., 2013).

2.4. Measurements of moisture content and MFA

The actual moisture content of each sample was measured directly prior to testing by the gravimetric method with the use of balance plates.

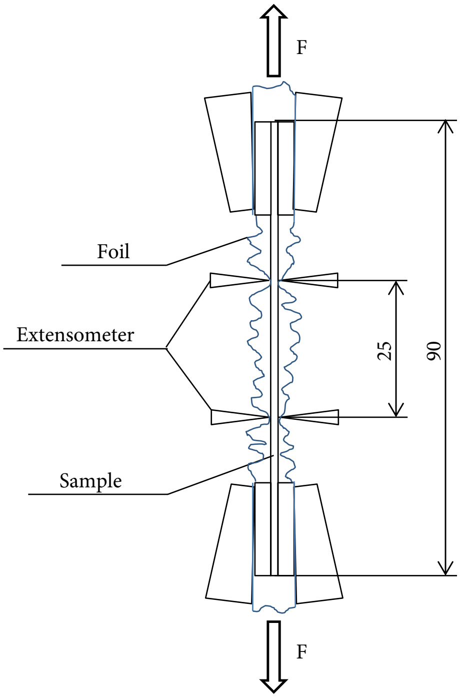

Figure 2. Measuring tensile stress and deformation.

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 (e.g., Moliński and Krauss, 2008; Krauss, 2010; Roszyk et al., 2010, 2012). A mean value of MFA was calculated in individual annual rings.

3. Results

The width of annual rings for tensile strength tests varied from 2 to 4 mm. The proportion of latewood in these rings varied from 25% to 37.5%. The mean density of earlywood determined at the moisture content of about 8% varied from 223 to 252 kg/m^3, while that of latewood was from 571 to 749 kg/m^3. The values of the above-mentioned parameters of samples are given in Table 1. Table 2 presents the mean values of MFA measured in the samples used for tensile strength tests and the basic fundamental data. According to Table 2, the mean values of MFA in the wood studied varied in a relatively wide range from 5.8° to 22°, thus almost 4-fold.

The influence of moisture content on tensile strength and modulus of linear elasticity of the earlywood is illustrated in Figure 3. This figure indicates that both parameters are practically independent of moisture content. No correlation was found between the mechanical parameters of earlywood and its moisture content. In contrast, tensile strength and modulus of elasticity in latewood decreased considerably as moisture content of the wood increased. This relation can be described by the power relations with determination coefficients (R^2) of greater than 0.6. According to the equations given in Figure 3, as the moisture content increased from an air-dry state to a wet state, the tensile strength of latewood decreased by about 68%, while the modulus of elasticity decreased by as much as 80%. The equations describe the variation of these 2 mechanical parameters of latewood in the entire range of moisture content variation. However, it is important to remember that the actual relation between the tensile strength and linear modulus of elasticity is restricted to the hygroscopic range to the FSP. To provide evidence for this, in Figure 4 the tensile strength and the modulus of elasticity of latewood are presented as a function of moisture content. The points corresponding to the results of measurements are divided into 2 groups, the first for the wood whose actual moisture content when subjected to tensile stress was in the hygroscopic range, and the second for the wood whose actual moisture content when subjected to tensile stress was close to or higher than the FSP. As follows from the arrangement of the data, only the bound water influences the mechanical parameters of wood.

Table 1. Parameters of wood selected for the study.

Cambial age of annual rings (years)	Ring width (mm)	Percentage of latewood (%)	Average density, ρ (kg/m³)	
			Earlywood	Latewood
21	3.0	33.3	226	749
38	4.0	37.5	232	719
39	3.7	35.0	242	647
40	3.5	28.6	223	639
62	2.0	25.0	252	571

Table 2. MFA values in earlywood and latewood (±S: standard deviation, V: variation coefficient).

Annual ring zone	MFA				
	Min.	Avg.	Max.	±S	V
	(°)				(%)
Earlywood	11.6	16.4	22.0	2.9	17.8
Latewood	5.8	9.0	12.3	1.7	18.8

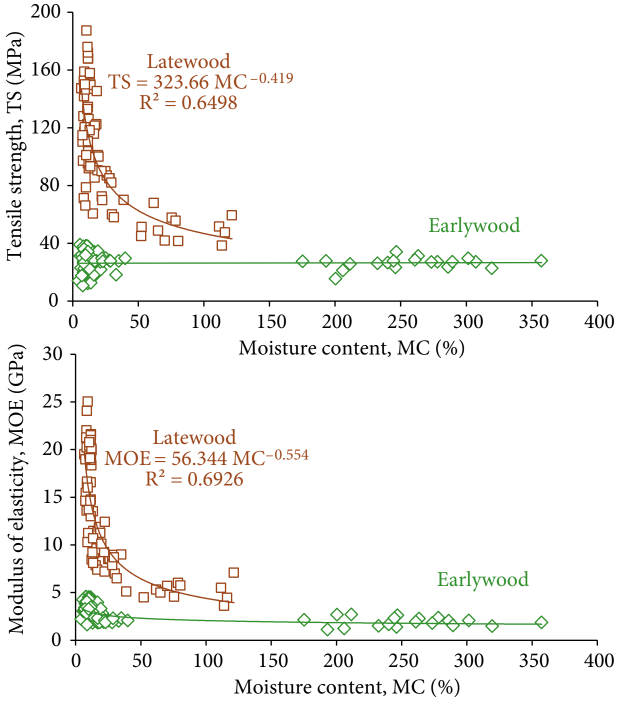

Figure 3. Relations between moisture content and mechanical parameters for earlywood and latewood from pine wood.

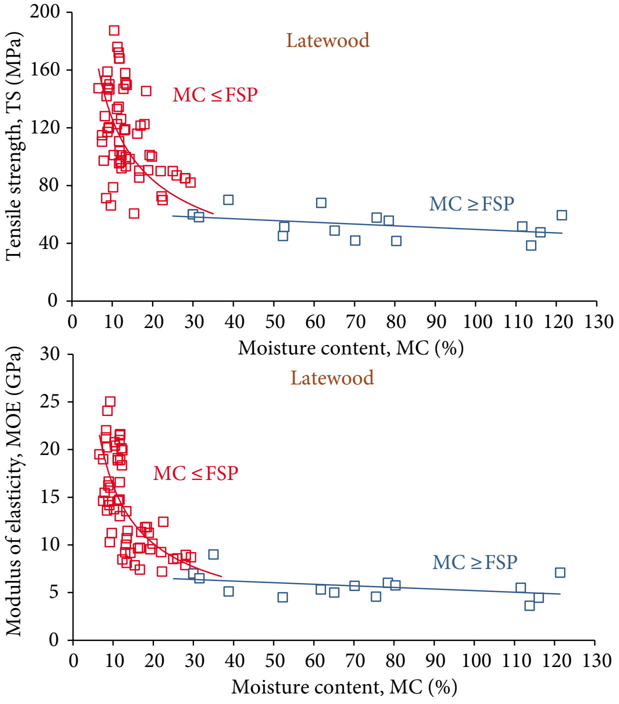

Figure 4. Tensile strength and modulus of elasticity for latewood inside and outside of the hygroscopic range.

Relative strain of the wood (with respect to the measuring base of the extensometer) at break as a function of moisture content was different for earlywood and latewood (Figure 5). For earlywood, the strain at break increased with increasing moisture content in the hygroscopic range, while for latewood no clear relation between these parameters was observed. Combined interpretation of the data on the relation between moisture content and tensile strength, modulus of elasticity, and strain at break indicates that although there was no direct influence of moisture content on tensile strength of earlywood, the strain at break increased as the moisture content increased up to the FSP. For latewood, it was the other way around: although its tensile strength and modulus of elasticity decreased as moisture content increased from the air-dry state to the FSP, the strain at which it broke did not depend on the moisture content.

4. Discussion

An observation similar to the one described above was studied between the density of early- and latewood and the mechanical parameters determined under tensile stress (Raczkowska and Raczkowski, 1979). A strong relation between Douglas-fir wood density and its tensile strength, modulus of elasticity, and specific work to ultimate load was demonstrated, although no distinct correlation was found between the wood density and its strain at break. Moreover, differences in the types of break were observed between earlywood and latewood; for earlywood, a typical break was an easy break, while for latewood it was more often a jagged break. This difference was explained by the fact that the tensile strength of earlywood depends first of all on the strength of the tracheid walls, while in latewood it is to a high degree also dependent on the strength of the links between neighboring tracheids or the links between particular layers of the cell wall.

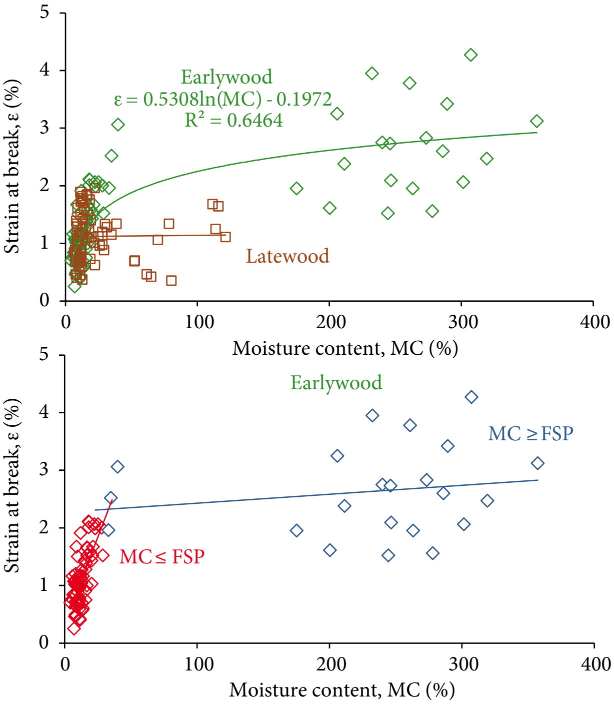

Figure 5. The influence of the moisture content of wood subjected to tensile stress along the grains on the strain at break.

According to other authors, the difference in tensile strength between early- and latewood has been determined not only by the wood density but also by MFA values (Mark, 1967; Mark and Gillis, 1973; Groom et al., 2002a, 2002b; Donaldson and Xu, 2005; Moliński and Krauss, 2008). To this end, we analyzed the mechanical parameters of earlywood and latewood as a function of MFA. The results are presented in Figure 6. As we have

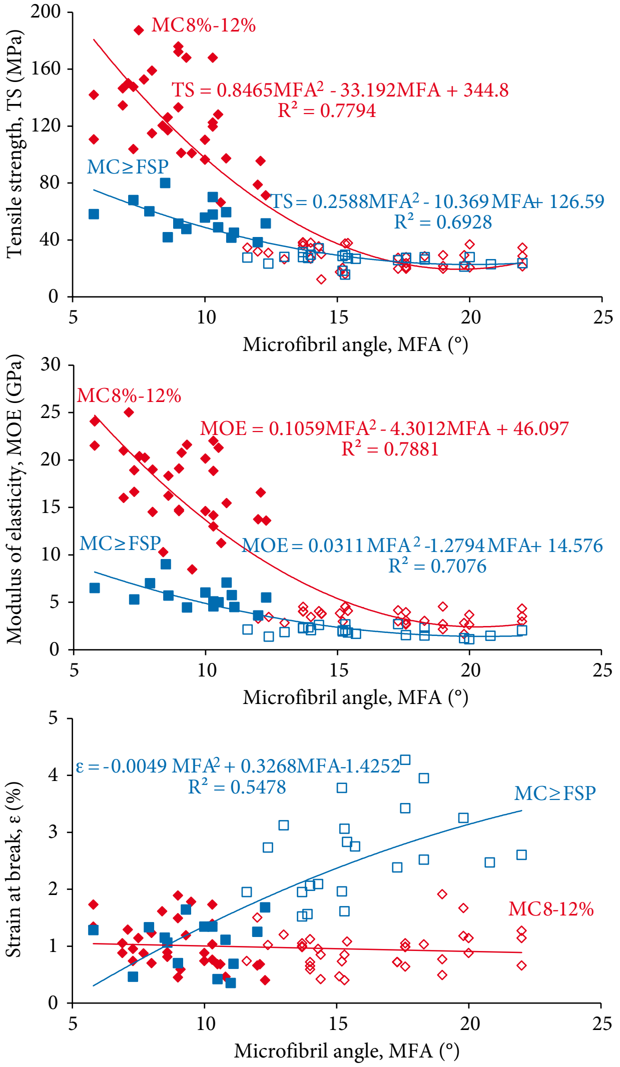

Figure 6. Relations between selected mechanical parameters of wood subjected to tensile stress along the grains in the air-dry state (moisture content = 8%–12%) and wet state (moisture content ≥ FSP) and the mean MFA values in the wood.

already evidenced the influence of moisture content on these parameters, Figure 6 refers to 2 different moisture content states, the air-dry state (moisture content = 8%–12%) and wet state (moisture content ≥ FSP). The results for earlywood are the empty symbols, while those for latewood are the corresponding filled ones. As presented in Figure 6, there is a strong correlation between the tensile strength and modulus of elasticity of wood, both in the air-dry and the wet state. Interestingly, the differences in the tensile strength and modulus of elasticity between earlywood and latewood, both in the air-dry and wet states, are smaller for higher values of MFA. The effect of MC on these mechanical parameters was detectable only for small MFAs, i.e. up to about 13°. For higher MFAs, the values of the above mechanical parameters remained constant and were independent of moisture content. However, the relation between strain at break and MFA is different. No correlation was found between the strains at break of the air-dry state, but for MFAs > 13°, the strain at break of the wet wood was found to increase with increasing MFA.

In conclusion, the above presented and discussed results have shown that the mechanical strength and modulus of elasticity of earlywood subjected to tensile stress along the grains were practically independent of its moisture content. In contrast, the mechanical strength and modulus of elasticity of latewood subjected to tensile stress along the grains decreased with increasing moisture content in the hydroscopic range, on average by 70% and 80%, respectively. Moreover, the strain at break of earlywood was higher the greater the moisture content. For latewood, no significant influence of moisture content on this parameter was found. The effect of moisture content on the mechanical strength of wood subjected to tensile stress along the grains depended on the mean MFA values in the tangent tracheid walls and was pronounced mainly for small MFAs of up to about 13°. It should be noted that the tensile strength and modulus of elasticity of wood whose mean MFA values vary between approximately 13° and 22° are practically constant and independent of moisture content.

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