VARIATION IN THE MICROFIBRIL ANGLE IN TANGENTIAL WALLS OF PINE WOOD TRACHEIDS (*PINUS SYLVESTRIS* L.)

Andrzej Krauss Poznan University of Live Sciences, Department of Wood Technology, Poznan, Poland

ABSTRACT

This paper presents results of measurements of the radial variation in microfibril angle (MFA) in the tangential walls of early and late tracheids in pine wood (*Pinus sylvestris* L.). In the walls of early tracheids the mean MFA values is on average ca.5° greater than in those of late tracheids. The difference in the mean MFA values between early and late tracheids increases with increasing cambial age of the annual rings. In the first near-pith annual rings the MFA of earlywood is by about 25 % greater than that of latewood. In the mature wood the mean MFA of earlywood is by 60 % greater than that of latewood.

KEY WORDS: juvenile wood, mature wood, individual rings, microfibril angle

INTRODUCTION

Recently, in wood science much interest is paid to the relations between the ultrastructure of the cell wall and wood properties. Cellulose in the form of microfibrils is responsible for the strength and elasticity of the cell walls. In coniferous species with growing maturity of wood tissue the mean value of the microfibril angle (MFA) decreases in the S2 layer of the secondary cell wall with respect to the longitudinal cell axis (Donaldson 1992, Lichtenegger et al. 1999). The orientation of microfibrils in the S2 layer, making from 79 to 89 % of the cell wall thickness, has been identified as the main factor determining mechanical properties of wood (Cave and Walker 1994). The strength of the bulk wood tissue depends on the MFA and at the same deformation the greatest strain suffers the walls of the smaller MFA (Reiterer et al. 1999). In view of the above, interest should be paid not only to the variation in the microfibril orientation along the radius of trees but also in individual annual rings. No such data are available for pine wood grown in Poland, as Polish pine trees are dominating in tree stands.

A number of methods for MFA determination have been proposed. The indirect methods are based on the use of X-ray technique (transmission or reflection X-ray diffraction) and the direct ones employ light or electron microscopy. The indirect methods permit determination of the mean MFA values for a wood sample whose thickness is of an order of millimetres, whereas the

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direct methods permit measurements of individual MFA in individual tracheids. When a large number of measurements must be performed the light microscopy is used. It is easy and ensures a wide field of view, in contrast to the electron microscopy, which is particularly important for measurements in microtome samples (Sarén and Serimaa 2006). The direct methods require preliminary preparation of the samples to expose the course of microfibrils, which can be achieved by treatment to the fungi enzymes (Anagnost et al. 2002, Khalili 2001) or destruction of cell walls by salt solutions (Wang et al. 2001, Moliński et al. 2007). In the wood treatment with fungi enzymes with decreasing loss of the wood mass the number of visible microfibrils increases but the slicing of preparations is difficult and the treatment takes a long time. The visual exposure of microfibrils proposed by Wang et al. (2001) employs the treatment of wood samples with a saltsolution and followed by the application of ultrasounds. There is also a method proposed by Moliński et al. (2007) which is a modification of the method proposed by Wang. In this method small samples of bulk wood are subjected to a solution of copper nitrate for a time and at a temperature adjusted to the tree species. This procedure does not require a preliminary slicing of microtome preparations and the later application of ultrasounds. The samples for this treatment have the following dimensions 7(L) x 5-10 (T) mm. The samples are cut along the radius, from which later tangential preparations are obtained.

MATERIAL AND METHODS

Material for the study was cut out at the breast height from a pine tree (Pinus sylvestis L.) of 115 years from the management stand owned by the Poznań University of Life Sciences. From the radial sample in which the wood macrostructure parameters were marked, a stripe of the size of 10 mm in the tangential direction and 7 mm along the grains was cut off. The stripe was divided into sections covering the zones of juvenile, maturating and mature wood. In each zone the values of the MFA were measured in 3 annual rings characterised by the proper structure of wood tissue. In each annual ring selected the tangential preparations of 30 µm in thickness were sliced off with a sliding microtome made by Leica, at every 0.5 mm To expose the microfibrils, the samples were treated with a 25 % solution of $Cu(NO_3)_2 \cdot 3H_2O_3$, in which the samples were heated at 85 °C for 15 hours. The MFA values were measured under a light microscope Motic Digital Microscope working with a PC unit with the Motic Image Plus 2.0 ML software allowing the MFA measurements to the accuracy of 0.01°. In each preparation at least 30 angles were measured and no more than 2 angles were measured in one tracheid. Statistical analysis was performed with the software Statistica 8 PL StatSoft [®] Polska. The aim of the study was to determine the variation in the MFA in the S2 layer in the tangential walls of early- and latetracheids in pine wood (Pinus sylvestris L.) grown in Poland.

RESULTS AND DISCUSSION

Tab. 1 presents the values of indices characterising the samples used in the experiment together with the mean values and ranges of variation of the MFA in the tangential walls of tracheids in annual rings. On the basis of the differences in the macrostructural features in the annual rings, three zones of the MFA measurements were distinguished.

Cross		Width of	Percentage of	Basic specific	Cambial	Mean MFA, Θ [deg.]		
sectional zone (annual rings)		annual rings	late- wood (%)	gravity (kg.m ⁻³)	age of annual ring	Earlywood,	Latewood,	Θ_{ew} - Θ_{lw}
		(mm)				Θew	Θ _{lw}	[deg.]
		Mean ± S.E. (Range)				Mean ± S.E. (Range)		
					3	21,3±0,24	15,7±0,38	5,6
						(12,1-35,2)	(8,1-23,9)	
Juvenile wood		5,2±0,26	16±2,1	390±14	6	20,7±0,24	16,9±0,42	3,8
						(9,3-33,4)	(7,1-26,1)	
		(3,9-6,6)	(8-28)	(370-410)	0	18,9±0,28	15,7±0,33	2.2
(1-12)					0	(11,8-25,6)	(8,4-23,9)	3,2
					mean	20,3±0,16	16,1±0,22	4,2
						(9,3-35,2)	(7,1-26,1)	
					15	19,1±0,20	12,5±0,26	6,6
						(12, 5-25, 7)	(8,0-21,3)	
Maturating wood		4,9±0,21 (4,3-5,8)	35±2,5 (26-43)	390±8 (380-400)	16	18,6±0,23	11,8±0,21	6,8
						(11,3-25,5)	(7,0-17,7)	
(13–18)					18	17,7±0,2	13,4±0,41	4,3
						(10, 1-28, 8)	(7,4-20,1)	
					mean	18,5±0,14	12,4±0,16	6,1
	(10, 1-28, 8)					(7,0-21,3)		
Mature wood		2,2±0,06	38±0,7 (21-54)	450±29 (390-500)	103	15,6±0,41	9,4±0,28	6,2
						(4,9-25,9)	(4,6-15,9)	
					106	15,7±0,43	9,5±0,34	6,2
	wood					(6,2-29,0)	(2,8-22,0)	
(10 1	112)	(0,9-4,2)			100	15,2±0,69	10,3±0,41	4.0
(19-112)					109	(5,7-25,4)	(2,4-20,3)	4,9
					maan	15,5±0,27	9,7±0,20	5.0
					mean	(4.9-29.0)	(2.4-22.0)	3,8

Tab. 1: Radial variation in the MFA and indicators of the macrostructure of annual rings

The results revealed differences in the mean MFA values between the early- and latewood, in all zones. The differences in the juvenile wood ranged to about 4°, while in the maturating and mature wood – to about 6°. These values indicate that the heterogeneity of juvenile wood is lower than that in the maturating and mature wood, which is in agreement with the literature reports, e.g. by Lichtenegger et al. (1999) who established that in the juvenile wood the variation in the MFA within single rings is small. Analysis of the MFA changes along the radius revealed a decrease in the mean MFA over the juvenile and mature wood on average by 4.8° in earlywood an by 6.4° in latewood. In the juvenile pine wood the mean MFA values are relatively small, e.g. by about 12° smaller than those in the juvenile larch tree (Moliński et al. 2007). This result testifies to a relatively good quality of pine trees grown in Poland. The ranges of the MFA variation are rather wide and with increasing distance from the pith these ranges shift toward lower values. In general, the determined MFA values vary within the range of 10-30°, typical of the MFA in the S2 layer, (Wagenfűhr 1999). The extreme values of the MFA of 2.4° and 35.2° were noted in latewood of the ring 109 and in earlywood of the ring 3, respectively.

Fig.1 illustrates the changes in the MFA typical of the annual rings at different distances from the pith. The character of the MFA changes is in agreement with the results reported by other authors, e.g. Abe at al. (1992). It is because the microfibrils form higher angles in the cell walls produced at the beginning of the vegetation period as compared at the end of the period. In the rings close to the pith, the mean MFA values almost linearly decreas in individual rings along its width. This observation is in agreement with the results of Anagnost et al. (2002). In the rings further from the pith, the intermediate zone between early- and latewood becomes well distinguished, in particular in mature wood. The mean values of MFA are more or less

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constant in earlywood and latewood. However, the values in earlywood are clearly higher as compared to latewood. In the transition zone the values are intermediate.



Fig. 1: Variation of the MFA along the width of selected annual ring of pine



Fig. 2: Radial variation of the MFA in early and late pine wood

Fig. 2 presents the MFA changes versus the distance from the pith in a logarithmic scale. With the increasing cambial age of the rings the values of MFA decrease, which was also observed for other species (Cave and Walker 1994, Sahlberg et al. 1997, Lichtenegger et al. 1999, Anagnost et al. 2002). The correlation between the mean MFA and the position of the early- and latewood along the radius is high (R^2 =0.84/0.74). The greater absolute value of the slope of the above linear dependence obtained for latewood indicates that the decrease in the MFA with increasing distance from the pith is more intense in latewood than in earlywood. This fact confirms that mature wood is characterised by greater heterogeneity than juvenile wood.

CONCLUSIONS

The mean MFA values in the tangential walls of tracheids in early- and latewood in pine tree decrease with increasing cambial age of the annual rings. In the walls of early tracheids the mean MFA values is ca. 5° greater than in those of late tracheids. The difference in the mean MFA values between early and late tracheids increases with increasing cambial age of the annual rings. The MFA variation in the cell walls in individual rings can be an indicator of wood heterogeneity and differences in its quality.

REFERENCES

- 1. Abe, H., Ohtani, J., Fukazawa, K., 1992: Microfibrillar orientation of the innermost surface of conifer tracheid walls, IAWA Bull.,13, Pp. 411-417
- 2. Anagnost, S.E., Mark, R.E., Hanna, R.B., 2002: Variation of microfibril angle within individual tracheids. Wood and Fiber Sci., 34(2): 337-349
- 3. Cave, I.D., Walker, J.C.F., 1994: Stiffness of wood in fast-grown plantation softwoods. Forest Prod J. 44(5): 43-48
- 4. Donaldson, L.A., 1992: Within- and between-tree variation in microfibril angle in Pinus radiate. New Zealand of Forestry Sci., 22: 77-86
- Khalili, S., Nilsson, T., Daniel, G., 2001: The use of rot fungi for determining the microfibrillar orientation in the S₂ layer of pine tracheids. Holz als Roh- und Werkstoff, 58: 439-447
- Lichtenegger, H., Reiterer, A., Tschegg, S., Fratzl, P., 1999: Variation of cellulose microfibril angles in softwoods and hardwoods – a possible strategy of mechanical optimization. J. of Structural Biology 128: 257-269
- Moliński, W., Fabisiak, E., Roszyk, E., 2007: Właściwości drewna modrzewia (*Larix decidua* Mill.) z uprawy plantacyjnej. Technologia drewna wczoraj, dziś i jutro. Praca zbiorowa pod red. W. Strykowski, ITD Poznań. Pp. 251-260
- Reiterer, A., Lichtenegger, H., Tschegg, S.E., Fratzl, P., 1999: Experimental evidence for a mechanical function of the cellulose spiral angle in wood cellulose walls. Philos. Mag., A 79: 2173-2186
- Sahlberg, U., Salmen, L., Oscarsson, A., 1997: The fibrillar orientation in the S₂-layer of wood fibres as determined by X-ray diffraction analysis. Wood Sci. Technol. 31: 77-85
- 10. Sarén, M-P., Serimaa, R., 2006: Determination of microfibril angle distribution by X-ray diffraction. Wood Sci. Technol. 40: 445-46
- 11. Wang, H.H., Drummond, J.G., Reath, S.M., Hunt, K., Watson, P.A., 2001: An improved fibril angle measurement method for wood fibres. Wood Sci. Technol., 34: 493-503
- 12. Wagenführ, R., 1999: Anatomie des Holzes. DRW- Verlag, 188 pp.

Andrzej Krauss Poznan University of Live Sciences Department of Wood Technology Ul. Wojska Polskiego 38/42 60 – 627 Poznan Poland