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THE INFLUENCE OF MICROFIBRIL ANGLE ON CREEP SCOTCH PINE WOOD UNDER TENSILE STRESS ALONG THE GRAINS

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ABSTRACT

The influence of microfibril angle on the process of creep of microtome wood (*Pinus sylvestris* L.) samples of constant but varying in different samples moisture content, under different tensile stress levels was studied. The wood density was proved not to be a reliable indicator of its technological quality. Mechanical parameters of wood subjected to tensile stress along the grains were shown to be determined by the microfibril angle in S2 layer of the secondary cell wall. Increase in MFA from 10 to 18° was found to cause a linear increase in total creep irrespectively of tensile stress applied and moisture content. For higher MFA the total deformation of wood is greater than that following from linear approximation of the relation for MFA in the range 10-18°. The intensity of specific wood creep increases under tensile stress along the grains and of high moisture content with increasing MFA. For the wood samples of constant low moisture content such a correlation is not always found.

KEYWORDS: Pine wood, tensile stress, microfibril angle, creep of wood.

INTRODUCTION

Wood is a natural composite material in the nanoscale built of elementary cellulose fibres set in a matrix made of hemicelluloses and lignin. Hemicelluloses localised among the elementary cellulose fibres act as a kind of bridge between cellulose and lignin (Fengel 1970, Via et al. 2007). Cellulose being a linear polymer is assumed to be responsible for the elastic properties of cell wall under tensile stress acting in parallel or almost parallel to the microfibrils, mainly in the thickest S2 layer. With increasing microfibril angle (MFA) to the longitudinal axis of the wood cells, the matrix made of hemicelluloses and lignin gain increasing influence on the mechanical properties of the cell walls (Bergander and Salmén 2002, Barnett and Bonham 2004, Gindl and Schöberl 2004, Salmén 2004). This observation prompted a supposition that the effect of lignin

on the strength and stiffness of the bulk wood tissue should be greater in juvenile than in mature wood as in juvenile wood MFA values are greater. Moreover, in juvenile wood greater MFA is accompanied by a lower content of cellulose and greater content of lignin (Hori et al. 2003). The arrangement of microfibrils in a cell wall is a very important factor determining the properties of bulk wood tissue and individual fibres (Bendtsen and Senft 1986, Cave and Walker 1994, Xu et al. 2004). The wood stiffness and strength is determined by the quality of cell walls, thus by the orientation of microfibrils and their packing in unit volume, expressed by wood density. According to Evans and Ilič (2001), who studied wood of *Eucalyptus delegatensis*, the orientation and packing can be in as much as 96 % responsible for the variation in the modulus of elasticity. Many authors (Bendtsen and Senft 1986, Cave and Walker 1994, Booker et al. 1998) even claim that the mechanical properties of wood are to a greater degree determined by MFA than wood density. Moliński and Krauss (2008) who studied changes in density, strength and modulus of elasticity in earlywood and latewood as a function of the cambial age of annual rings in pine tree, reported an increase in the mechanical parameters of earlywood as a function of cambial age despite a significant decrease in its density. They have also shown that the changes in the modulus of elasticity of cell walls as a function of cambial age are of the opposite character to the changes in mean MFA in S2 layer of cell wall.

Investigation of relations between MFA in tracheid walls and mechanical parameters of wood subjected to tensile stress along the grains has shown that the strength and modulus of elasticity of cell walls are greater for lower values of MFA (Cave 1976, Dinwoodie 1981, Cave and Walker 1994, Reiterer et al. 1999, Groom et al. 2002, Moliński and Krauss 2008, Krauss 2010, Roszyk et al. 2010a). According to Reiterer et al. (1999), who studied microtome samples of pine wood under tensile stress along the grains, with MFA increased from 5 to 50°, the maximum value of the strain increases from 0.5 to 11 %, at the accompanying decrease in strength from 220 to 35 MPa. In the experiments on individual fibres subjected to tensile stress (Page et al. 1972, 1977, Page and El-Hosseiny 1983, Mott et al. 2002) also a positive correlation was found between MFA and their stretchability. Sedighi-Gilani and Navi (2007) suggested that the viscoelastic behaviour of wood subjected to tensile stress along the grains is related to local damage to the matrix and reorientation of cellulose fibres. Changes in MFA in the samples subjected to tensile stress have also been reported on their cyclic loading and these changes were interpreted as responsible for the fact that the wood samples were not weakened even upon loading exceeding the proportionality range (Keckes et al. 2003).

Ultrastructure of the cell wall determines to a high degree the behaviour of wood subjected to long term loading. Already in 1972, El-Osta and Wellwood noted that the deformation on short-time creep were proportional to MFA. This observation was confirmed by Kojima and Yamamoto (2004a), who analysed the parameters of the three-parameter model (Voigt element plus a spring), approximating the experimental data on creep of microtome samples of *Cryptomeria japonica* D.Don wood at the moisture content close to the fibre saturation point (FSP) and under tensile stress bordering on the limit of proportionality and proved that with increasing MFA, the responsibility of the viscous behaviour of the matrix filling the cellulose skeleton for the deformation caused by creep increases. For low MFA values, the process of creep is determined first of all by the local viscoelasticity of the cellulose skeleton (in amorphous regions) (Kojima and Yamamoto 2004b, 2005). Recently, Roszyk et al. (2010b), studied creep in microtome samples of pine earlywood under tensile stress and wetting by humid air and noted that the relative susceptibility to creep ($J(t)/J_0$) was practically constant in the samples whose mean MFA in S2 layer varied from 12 to 18°. At higher MFA the susceptibility increased with increasing mean MFA. Moreover, according to the same authors, the contribution of

instantaneous recovery (recorded directly after release of the load) in total deformation (recorded at the end of the process of hygromechanic creep) was the greater the lower the mean MFA in a given sample. The contribution of permanent deformation in total deformation increased with increasing MFA. These observations can indicate a reorientation of microfibrils in the cell wall, caused by local damages to the viscous matrix incrusting the cellulose skeleton and breaking of hemicellulose and cellulose bondings (Burgert 2006).

Olsson et al. (2007), studied creep of individual tracheids of pine wood (*Picea abies* [L.] Karst.) and reported that the deformation caused by wood creep both at a constant (RH = 80 %) and cyclically changed relative humidity of surrounding air (RH 80-30 %) increased with increasing tensile stress and with decreasing modulus of elasticity. According to many authors, the relation between the modulus of elasticity of wood or wood cell walls and MFA values (determined under tensile stress) is nonlinear (e.g. Cave and Walker 1994, Reiterer et al. 1999, Evans and Ilič 2001, Yang and Evans 2003, Krauss 2010). However, Via et al. (2009), who studied wood of longleaf pine (*Pinus palustris*), established that this correlation for MFA varying from 40 to 5° is equally strong when approximated by an exponential function or a linear one. The same authors also proved that for the same range of MFA variation (40 to 5°) the modulus of elasticity of this wood increases 4 times Cave and Walker (1994) for a similar range of MFA variation at the border of juvenile and mature wood of *Pinus radiata* observed 5 times increase in stiffness. In view of the above, it is important to supplement the existing information with that on the influence of MFA on the process of creep under tensile stress of different values. To the best of our knowledge no data have been published on this relationship. The aim of our study was to establish the influence of MFA on creep in microtome samples of wood induced by tensile strength of different values.

MATERIAL AND METHODS

Creep of wood was studied on microtome samples of the size 120(L)x7(T)x0.2-0.3 mm (R). Samples of similar size have been used in earlier studies (Robson 1989, Reiterer et al. 1999, Moliński et al. 2008, Roszyk et al. 2010a,b). The samples to be studied were obtained by a slide microtome from a plank cut out of fresh pine wood along the ray (*Pinus sylvestris* L.) of tangent width of 7 mm. The plank was localised immediately above the fillet (of 5 mm in width) for which the changes in MFA had been studied along the width of selected annual rings. The measurements were performed on microscopic preparations with the help of a computer image analyser and after earlier visualisation of microfibrils in cell walls. The fillet was heated in a 20 % solution of $Cu(NO_3)_2$ at 80°C for 24 hours, then washed with distilled water and to stop the salt activity, it was boiled in distilled water at 100°C for 2 hours. After the above-described preliminary preparation, from the same annual rings from which the samples for wood creep and immediate tensile strength were collected, some tangent slices were obtained by slide microtome. The slices were of about 20 μm in thickness and were used to make microscopic preparations (Moliński et al. 2008, Krauss 2010, Roszyk et al. 2010a).

The plank was divided into two equal parts of 120 mm in length and with the use of a slide microtome the samples to be studied were sliced. For investigation of creep and determination of tensile strength only the samples cut off from the earlywood zone were used. A scheme illustrating the procedure of obtaining the samples for MFA measurements is shown in Fig. 1.

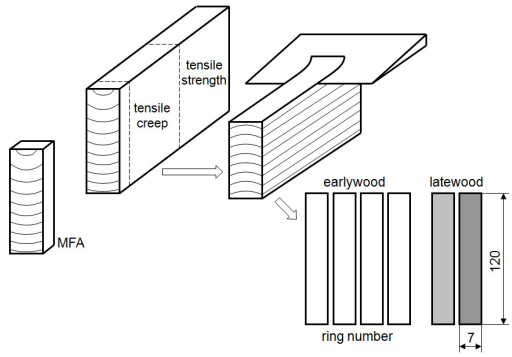

Fig. 1: Schematic procedure of sample preparation.

Having determined MFA changes in individual annual rings (Moliński et al. 2008, Krauss 2010, Roszyk et al. 2010a) mean MFA values for each sample were calculated. Then they were labelled to identify the annual ring of their origin and their moisture content was equilibrated with the surrounding air humidity (MC ~ 7 %), and the linear dimensions of the samples were measured. The thickness in the radial direction was measured at three sites at the middle of the length and at 2 cm from the middle on both sides with the use of an electronic micrometer to the accuracy of 0.001 mm. The width in tangential direction and the length were measured by a Biotronic increment meter, to the accuracy of 0.01 mm. The samples were also weighted to the accuracy of 0.0001 g. On the basis of these measurements the sample densities were calculated. At the next stage the ends of the microtome preparations were covered with plates made of hardboard of the size 20x20x3 mm, glued on both sides. This strengthening of the mounting elements protected the samples against crushing in the holders of testing machines (Roszyk et al. 2010a, b).

Tensile strength along the grains was measured on the samples twin to those on which the creep process was studied, obtained from the same annual rings and in direct neighbourhood along the length of the material. Measurements of tensile strength were made on a testing machine ZWICK ZO50TH using an extensometer ZWICK 066550.02, whose measuring base was set for 30 mm. After mounting in the machine holders the samples were subjected to tensile stress at the rate 1 $mm.min^{-1}$. Preliminary fed with the sample size the programming of the machine permitted calculation of the tensile strength and modulus of elasticity. The measurements were performed at the wood moisture content of 7 %. The creep of the samples was measured by a prototype creep device which construction is schematically presented in Fig. 2. The stretching stress constant in time was induced by mounting a load chosen according to the cross-section of a sample. The stretching stress applied in the study caused 15, 30 or 45 % of the mean tensile strength determined at the moisture content of 7 % for samples of similar MFA values. It means that the samples in which MFA values were considerably different were subjected to different stress, but the stress values corresponded to the presumed level of stress relative to the tensile strength. The tensile stress values selected ensured the linear viscoelastic creep at a constant moisture content of the sample (Poliszko and Raczkowski 1984, Roszyk 2006).

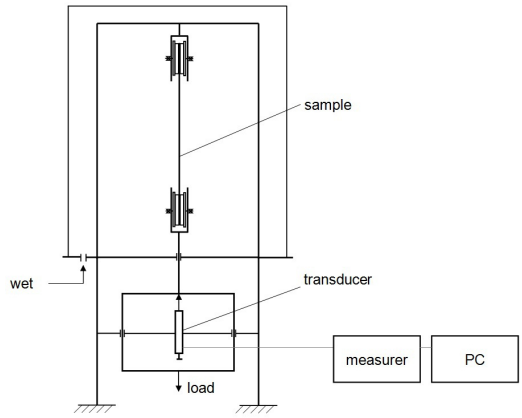

Fig. 2: A schematic diagram of the prototype laboratory creep-testing machine.

For each tensile stress level chosen, two variants of the experiment aimed at the study of creep were realised; in the first – the moisture content of a wood sample corresponded to the hygroscopic equilibrium in laboratory conditions (MC ~ 7 %), while in the second – the moisture content of wood samples was maintained at 27 %. In the first variant, after mounting the sample in the holder of the creep machine, it was subjected to an adequate tensile stress and its extension was recorded. In the second variant, after mounting the sample, the chamber with the sample was blown with the air preliminary blown through distilled water of room temperature. The process of sample wetting was controlled by measurements of mass of a control sample placed in the creep machine chamber and hang on a laboratory balance. Measurements of creep on the samples of the higher moisture content were performed two hours after stabilisation of the control sample mass. Extension of the sample was measured using electronic displacement sensors MEGATRON CR1825K, coupled to a PC unit. Measuring system KEST ELECTRONICS K1603 permitted recording the results with the accuracy of 0.001 mm at 15 second intervals.

RESULTS AND DISCUSSION

The main parameters characterising the experimental material are given in Tab. 1, where is also given the mean MFA of individual samples. The results show great scatter for samples of similar mean MFA values. Thus, it can be concluded that the scatter of tensile strength and modulus of elasticity within particular groups of samples is related to the variation in wood density. In view of the above, for the experiment aimed at determination of creep we used only the samples representing particular groups characterised by very close mean MFA whose density did not deviate from the mean value by more than ±10 $kg.m^{-3}$. Analysis of the results of measurements showed that the tensile strength and modulus of elasticity decrease with increasing wood density (Fig. 3). The fact that the earlywood density in mature tissue is lower than in the juvenile tissue has been shown also for *Tsuga heterophylla* (Panshin et al. 1964), *Picea mariana* (Zhang 1998) and *Larix kaempferi* (Koizumi et al. 2005). Reduction of the values of mechanical parameters of wood with its increasing density proves that the wood density cannot be treated as a reliable indicator of wood technological quality. Thus, our results have fully confirmed the earlier reports (Bendtsen

and Senft 1986, Cave and Walker 1994, Moliński and Krauss 2008, Roszyk et al. 2010b).

Tab. 1: Density, mechanical strength and modulus of elasticity of wood of MC 7 %, determined upon tensile stress applied along the grains and the mean MFA in particular samples.

Typ of wood tissue	Density (kg.m^{-3})			Tensile strength (MPa)			Modulus of elasticity (GPa)			Average MFA (°)		
	x_{min}	X_{AV}	x_{max}	x_{min}	X_{AV}	x_{max}	x_{min}	X_{AV}	x_{max}	x_{min}	X_{AV}	x_{max}
Juvenile wood	355	383	466	16.6	22.4	34.1	3.3	3.4	5.9	20	20	20
	264	328	448	21.7	25.2	35.6	2.9	3.1	3.4	18	18	19
	203	283	332	23.9	28.5	38.9	3.5	4.4	4.8	16	16	17
Mature wood	219	256	312	30.8	39.6	48.2	4.4	4.6	6.2	12	13	14
	236	245	252	32.0	41.8	54.8	4.1	5.3	7.1	10	10	11

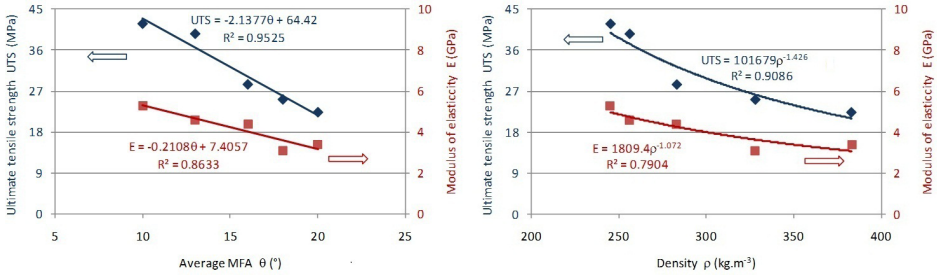

Fig. 3: Correlations between tensile strength, modulus of elasticity and mean MFA and density of wood samples studied.

As follows from Fig. 3, the mechanical parameters of the wood studied are determined by the MFA values. The modulus of elasticity and tensile strength of the samples studied are inversely proportional to the mean MFA values and these correlations are characterised by high coefficient of determination R^2. The influence of MFA on the tensile strength and modulus of elasticity is illustrated by the upper plot in Fig. 3, while the lower shows the analogous dependencies on wood density. If the densities of the samples studied were the same, the influence of MFA on the two mechanical parameters would be even greater.

As follows from the above results, MFA must significantly influence the creep process. Exemplary curves describing the creep of samples subjected to different tensile stress and characterised by the same mean MFA of 6° at a constant but different for different samples moisture constant (either ~7 % or ~27 %) are shown in Fig. 4. Deformation of samples (strain, ε) was expressed as the ratio of the extension of the sample in a given experiment ($\Delta l(t)$) to its initial length measured between the machine holders (l_0).

To illustrate the scatter of results the minimum and maximum values of the parameter measured are given along with the curve showing the character of changes in the mean values of these parameters, calculated on the basis of three repetitions. Small diversity of the values of deformation for the samples studied for different levels of tensile stress applied confirms correct

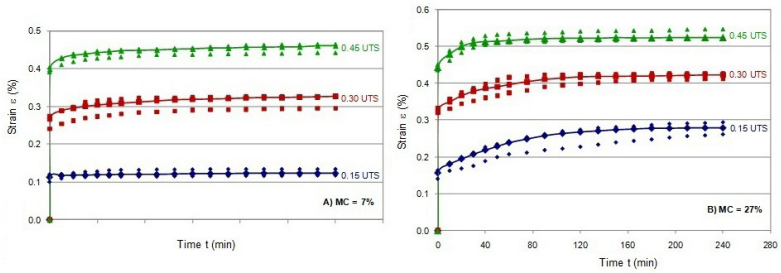

Fig. 4: Time dependencies of strain of the samples characterised with MFA of 16° and of either low A) or high B) moisture content, at constant for a given sample but different for different samples tensile stress applied along the grains.

choice of the samples and correct range of tensile stresses applied. According to the data, an increase in the tensile stress applied causes intensification of creep in the samples studied, which is greater at higher moisture content. For the samples with other mean MFA the effect of tensile stress on their creep was similar. The above observations are illustrated by the dependence of isochronal mean values of total deformation, recorded for particular groups of samples after 4 hours of their creep on the level of tensile stress, shown in Fig. 5. As follows, the total deformation recorded in the last phase of the process of creep is a linear function of the tensile stress applied and this correlation is characterised by high coefficients of determination (R^2). Moreover, this figure confirms the earlier observations as to the effect of MFA on creep of wood subjected to tensile stress along the grains (El-Osta and Wellwood 1972, Kojima and Yamamoto 2004a, Roszyk et al. 2010b). With increasing mean MFA the magnitude of deformations increases.

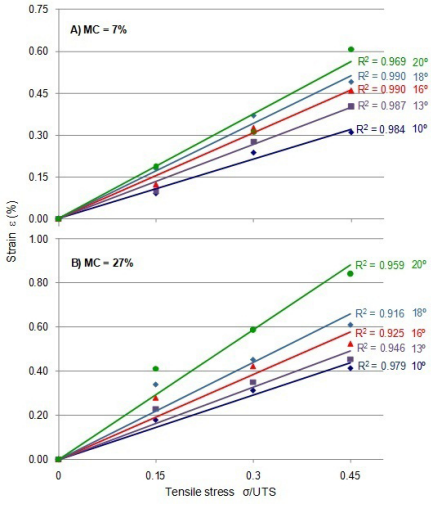

Fig. 5: Tensile stress applied along the grains versus total wood deformation (t = 4 h) for samples of a constant low A) or high B) moisture content versus mean MFA for particular samples.

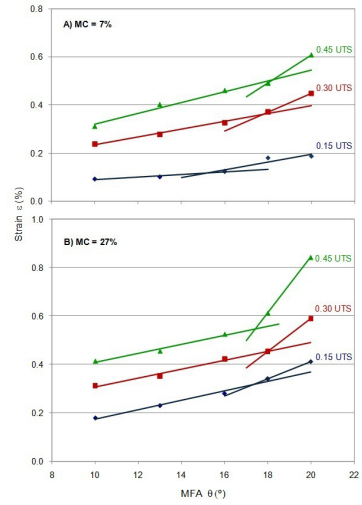

Fig. 6: The influence of MFA on total deformation (t = 4 h) of samples subjected to tensile stress of different values.

According to the results of our experiment, an increase in the mean MFA from 10 to 18°results in a practically linear increase in total deformation irrespective of the tensile stress applied and wood moisture content (Fig. 6). For higher MFA, e.g. of 20°, the total deformation was greater than it would be according to the linear approximation of the relation for MFA from the range 10-18°. This observation is in agreement with that reported by Roszyk et al. (2010b) who analysed the influence of MFA on hygromechanical creep of microtome wood samples under tensile stress. After analysis of recovery, this effect was interpreted as a result of microfibril reorientations.

Decreasing MFA values in cell walls of the reaction wood of spruce (*Picea abies*) upon tensile stress application along the grains was noted by Burgert (2006). He claimed that this effect was significant only in the range of tensile stress above the limit of proportionality. In this paper the tensile stress values were much lower (up to 45 % of ultimate tensile strength determined at the moisture content of about 7 %). The MFA decrease in the conditions of wood creep should be observable only in the range of specific creep, that is with disregard of instantaneous deformations. The specific creep obtained for the samples studied of moisture contents of 7 % or 27 % are presented in Fig. 7. The time dependencies of specific creep were made only for the mean values of specific creep. As indicated by the plots, the deformation of the samples subjected to tensile stress at MC of 27 % is greater than at MC of 7 %.

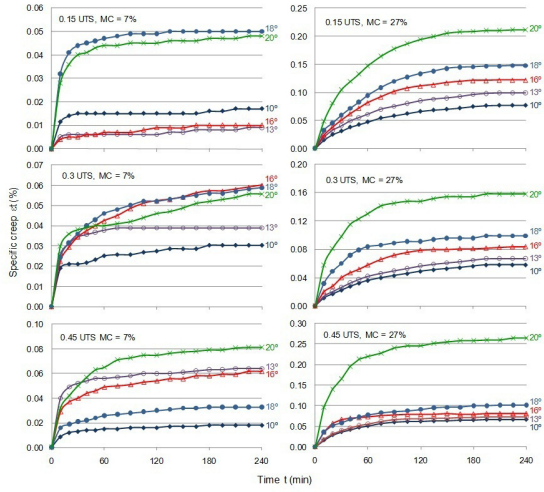

Fig. 7: Plots of specific creep of samples subjected to different tensile stress versus mean MFA for the samples of low (7 %) and high (27 %) moisture content.

Moreover the intensity of specific creep at MC of 27 % for particular ranges of tensile stress applied is the greater the higher the mean MFA value for the samples studied. Moreover, for the samples of MFA of 20° the deformation caused by creep is much greater than for the samples of MFA ≤ 18°, see Fig. 6. For the samples of MC of 7 %, the deformation caused be specific creep tends to increase with increasing tensile stress applied, but the creep curves for different MFA and all tensile stress applied are rather randomly distributed. No distinct effect of MFA on creep of wood of low moisture content can be explained by a higher energy needed to initiate the mobility of cellulose chains in the matrix surrounding them than in the wood of higher MC. Differences

in the character of the curves of time dependencies of specific creep for the two levels of moisture content are interpreted as a result of the water induced plasticity. In wet wood the hemicelluloses directly surrounding cellulose undergo softening already at room temperature (e.g. Salmén 1984). Therefore, in the wood of MC of 27 % the possibility of microfibril migration is much greater than in the wood of MC of 7 %, and moreover it is the easier the greater MFA. The differences in wood creep values not always can be attributed to different tensile stress and can also be related to variation in mechanical parameters of cell walls at the same MFA values (Cave and Walker 1994, Krauss 2010, Roszyk et al. 2010a). It should be emphasised that the results presented have not exhausted the problem of our concern and the study should be continued.

CONCLUSIONS

Analysis of the above presented and discussed results permits drawing the following conclusions:

1. Wood density cannot be treated as reliable descriptor of its technological quality. Mechanical parameters of wood subjected to tensile stress along the grains are mainly determined by the microfibril angle in S2 layer of secondary cell wall.
2. Increase in MFA from 10 to 18° causes a linear increase in total deformation. For higher MFA the deformations are greater than that following from approximation of the above linear dependence for MFA from 10-18°.
3. Intensity of specific creep of wood of constant high moisture content and subjected to tensile stress is the higher the greater the MFA in S2 layer of secondary cell wall. For wood of low moisture content such a correlation is not always noted, which can be related to a greater energy needed for initiation of cellulose chains mobility in the surrounding matrix.

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