



# Swelling restraint of thermally modified ash wood perpendicular to the grain

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## Abstract

The paper reports on creep of ash wood (*Fraxinus excelsior* L.) thermally modified at 180 and 200 °C, and subsequently subjected to compression in tangential and radial directions and simultaneously wetted, from the moisture content (MC) of 6% to above the fibre saturation point (FSP). The compressing load made 0.00, 0.25, 0.50 and 0.75 of impact stress at the proportional limit ( $R_c$ ). The compression stress needed to restrain the swelling of wood, the so-called swelling pressure, was indirectly determined from isochrones of mechano-sorptive creep. The most important finding was that thermal modification reduces the strain of ash wood subjected to compression perpendicular to the grain to a degree proportional to the mass loss. The compression stress needed to restrain the swelling of thermally modified wood is ca. 10 and 20% smaller in the tangential and radial directions, respectively. This effect leads to a reduction in the anisotropy of swelling pressure of thermally modified wood perpendicular to the grain. Moreover, although upon thermal modification the mass loss of wood takes place, at the MC of 6% it shows practically the same modulus of elasticity (MOE) and  $R_c$  as the unmodified wood. After wetting to MC higher than the FSP, the thermally modified wood at 200 °C shows significantly higher MOE and  $R_c$  than the wood modified at 180 °C and untreated wood. Reduction of wood hygroscopicity, an inevitable effect of thermal modification, also reduces the range of changes in mechanical properties of wood caused by the increase in its MC to the FSP.

## 1 Introduction

The effects of thermal modification of wood on its chemical structure and properties have been extensively studied. It has been established that the effects, depending on the process parameters (i.e. temperature and duration of treatment) and wood species, lead to changes in wood colour (Icel et al. 2015; Candelier et al. 2016), and a decrease in durability (Mazela et al. 2004; Sivrikaya et al. 2015) and hygroscopicity (Olek et al. 2013; Icel et al. 2015). Although thermal modification always leads to a reduction in wood density, sometimes an increase in its compression strength parallel and perpendicular to the grain or an increase in bending strength, modulus of elasticity (MOE) and hardness is observed. It depends on the wood species and parameters of

modification (Boonstra et al. 2007; Shi et al. 2007; Gündüz et al. 2008, 2009; Korkut and Hiziroglu 2009; Icel et al. 2015; Korkut and Aytin 2015; Moliński et al. 2016). Other wood properties including tensile strength parallel and perpendicular to the grain, shearing strength, dynamic bending strength and impact strength always deteriorate upon thermal modification (International ThermoWood Association 2003; Windeisen et al. 2009). Thermal modification of wood has also been found to result in a decrease in fracture toughness (Murata et al. 2013). Some divergence in the evaluation of the effect of thermal modification on mechanical strength of wood follows from the fact that many authors compare mechanical properties of modified wood with the reference data collected for reference wood after conditioning at the same air relative humidity (RH) and temperature. In other words, the mechanical parameters of modified wood are in fact determined at its lower moisture content (MC) than that of untreated wood. This fact has been confirmed by Arnold (2010) and Moliński et al. (2010, 2016).

Arnold (2010) studied the MC effect of thermally modified beech and spruce wood on the bending strength and revealed that the mechanical parameters of these two wood species after modification undergo smaller changes with

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increasing MC to the fibre saturation point (FSP) than the analogous parameters of unmodified wood. Similar conclusions have been drawn from the study of thermally modified beech wood subjected to compression parallel and perpendicular to the grain (Straže et al. 2016). The above authors studied the variation in wood mechanical properties over the entire hygroscopic range. The FSP of the thermally modified wood was found to be lower than that of unmodified wood; the former value was lower the higher the modification temperature (Straže et al. 2016). According to Moliński et al. (2016), who studied the mechanical properties of thermally modified and unmodified ash wood subjected to compression in the radial direction at the same and constant MC of 4 or 12%, the decrease in the mechanical properties with increasing MC was more pronounced for the modified than for the unmodified wood.

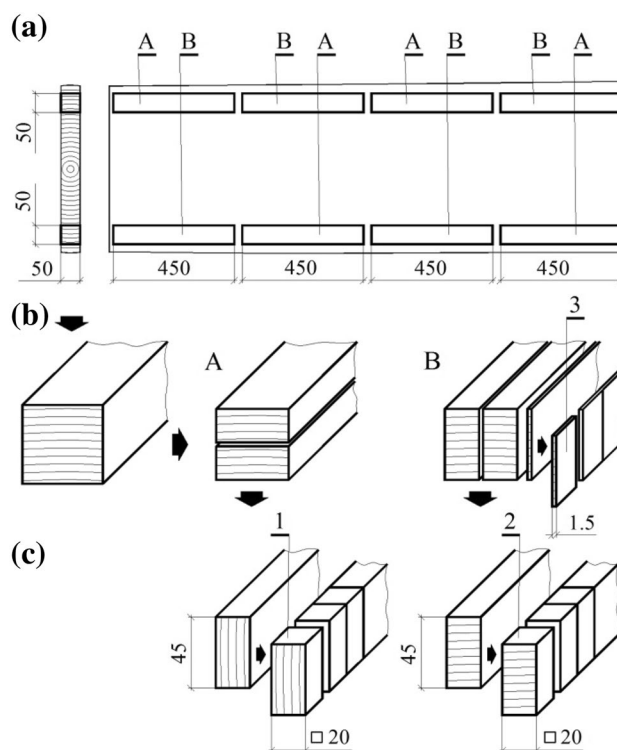
The above-mentioned studies concern the impact strength of thermally modified wood of different but constant MC. From the practical point of view it is important to establish the effect of changing MC of thermally modified wood on its behaviour under a long-term mechanical load (Arnold 2010), i.e. mechano-sorptive creep. To the best of the authors' knowledge, no data have been published on this subject, which has prompted us to fill this gap. The aim of this study was to determine the creep of thermally modified ash wood (*Fraxinus excelsior* L.) wetted under compression load in tangential and radial directions. The isochrones of mechano-sorptive creep were used to determine the stress needed to restrain the swelling, known in the literature as the wood swelling pressure (Perkitny 1951; Kollmann and Côté 1968). The wood swelling pressure, found indirectly on the basis of a linear relation between stress and strain caused by wetting of wood under compression load, provides values which better approximate the real values than direct measurements of this parameter (Moliński and Raczowski 1988; Moliński and Roszyk 2010).

In view of the fact that thermally modified ash wood is used for flooring, terrace and elevation boards and garden furniture, so the products are exposed to rainfall, determination of its swelling pressure, especially perpendicular to the grain, is very important as it describes the probability of crack formation or angle joint clearance.

## 2 Materials and methods

### 2.1 Material and samples

Samples were prepared in two stages. In the first stage, 8 pre-samples were cut out of a kiln-dried clear quarter sawn board (Fig. 1a). The dimensions of the pre-samples were  $50 \times 50 \times 450$  mm<sup>3</sup> in thickness, width and length, respectively. These pre-samples were cut into lamellae of ca.



**Fig. 1** Scheme of sample preparation: **a** initial cutting of pre-samples from the quarter-sawn board, **b** cutting lamellae before thermal modification (A—tangential and B—radial cutting pattern), **c** final cutting of the samples after thermal modification (1, 2—sample for density, impact compression and creep, 3—additional subsamples for sorption experiment)

25 mm in thickness in such a way as to obtain an exactly parallel or exactly perpendicular arrangement of annual rings with respect to the wide plane of the lamella. From the four pieces of elements, 8 lamellae of tangential cutting pattern (A) were cut off and from the remaining 4 elements, 8 lamellae of radial cutting pattern (B) were obtained (Fig. 1b). The lamellae were seasoned in the laboratory to MC of 10% for 3 months. Then, each set of lamellae (8 lamellae representing each anatomical direction) was divided into three parts: (a) modified at 180 °C, (b) modified at 200 °C and (c) control (unmodified). Sets (a) and (b) had an additional lamella to obtain samples for the determination of the mass loss (ML) caused by thermal modification. ML of the modified wood was determined with respect to the oven-dry mass of unmodified material.

Thermal modification of wood was performed under laboratory conditions taking into account the ThermoWood technology requirements (International ThermoWood Association 2003). The thermal modification of wood was made at two temperature options, 180 and 200 °C, for 3 h. Each modification process consisted of three phases. The initial phase of heating was made in moist air only, and superheated steam was added to the kiln after obtaining a

temperature of ca. 130 °C. The heating phase was ended when the target temperature was obtained and the modification had occurred. After completing the modification phase cooling started, firstly with superheated steam and next in moist air only.

In the second stage the lamellae were cut to obtain samples of the dimensions  $20 \times 20 \times 45 \text{ mm}^3$  (Fig. 1c) for the determination of wood density and its mechanical properties (impact strength, MOE) and creep. Prior to measurements, all samples were seasoned in separate desiccators at  $22 \pm 1 \text{ °C}$  over salt solutions to obtain the same MC of 6%. The air RH was 40, 55 and 58%, for the unmodified samples, and those modified at 180 and 200 °C, respectively. The seasoning lasted for 3 months. After this time the wood density was determined stereometrically. The dimensions of the samples were measured by a digital calliper ( $\Delta l = 0.001 \text{ mm}$ ). The mass was measured by a laboratory balance ( $\Delta m = 0.001 \text{ g}$ ). The MC of the samples was determined according to the oven-dry method.

The hygroscopic properties of wood in the adsorption and desorption experiment were determined for 13 subsamples of radial cutting pattern (B), for each option of thermal modification and control.

## 2.2 Adsorption and desorption experiment

The adsorption and desorption experiments were carried out using the previously prepared test setup, where the wood specimens were placed in a chamber, while the air RH was controlled by salt solutions (Majka and Olek 2007). The value of the relative error in MC determination during the experiment was accepted as an additional criterion of the wood samples' dimensions. Minimum mass of the samples was determined using the approach proposed by Taylor (1997). The dimensions of the wood specimens were ca.  $1.5 \times 30 \times 45 \text{ mm}^3$ , in the tangential (T), longitudinal (L) and radial (R) directions respectively. All sets of specimens (39) were stored in a desiccator and dried to a constant mass over phosphorus pentoxide ( $\text{P}_2\text{O}_5$ ). Temperature and air RH were measured by a thermo-hygrometer, type LB 706 (LAB-EL Reguły, Poland). The accuracy of temperature measurements was  $\pm 0.1 \text{ °C}$ , and that of air RH was  $\pm 2.0\%$  (in the range of 10–90%). The use of salt solutions made it possible to achieve nine levels of air RH inside the chamber. All specimens were weighed at least twice after obtaining equilibrium at each air humidity level. At the end of the experimental procedure, all the samples were placed in a laboratory drier (at  $103 \pm 2 \text{ °C}$ ) and their oven-dry mass was determined. Measurements for each option of thermal treatment were made for 13 samples. Therefore, each value of the equilibrium moisture content (EMC) was a mean of 13 measurements. Sorption isotherms were determined at  $22 \pm 1 \text{ °C}$ .

## 2.3 Impact strength

In order to choose the absolute compression load in the creep phenomenon and to characterise the mechanical properties of the wood investigated, at first its compression strength ( $R_c$ )—which is the stress at the proportional limit—and MOE were determined. The measurements were made with the testing machine Z050TH (Zwick/Roell, Kennesaw, USA), according to the standard PN-77/D-04229 (1977) on twin samples of MC equal to 6% and above the FSP. To obtain the wet state, the samples were immersed in distilled water. The MOE was determined from the stress–strain relation, similar to Straže et al. (2016).

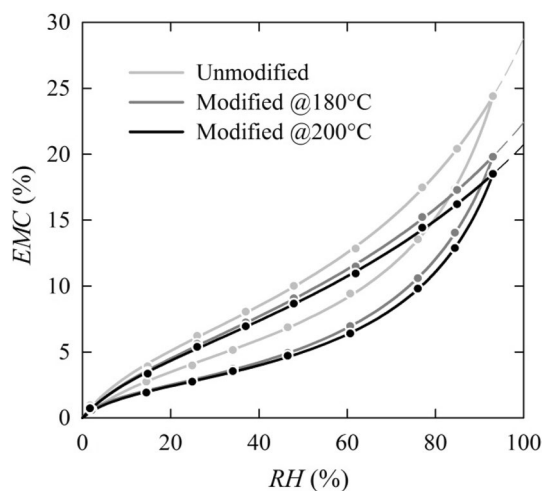
## 2.4 Creep

Creep of modified and unmodified samples was measured using a prototype creep testing machine (Moliński and Raczkowski 1982). The compression load was applied to the entire face surfaces of the samples in tangential or radial direction. The range of loading was chosen to permit observation of mechano-sorptive creep in a wide range of linear deformation. The stress ( $\sigma$ ) induced in the sample was 0.00, 0.25, 0.50 and 0.75 of the mean  $R_c$ , determined in a wet state ( $\text{MC} > \text{FSP}$ ). Immediately after loading of the sample (MC 6%) its wetting was started by immersion in distilled water of ambient temperature in a container so that the sample was submerged. The creep was determined by measurement of complex deformation of the wood sample in the direction of compression. The measurements were made by a digital displacement meter coupled with a computer. The measuring system Kest Electronics K1603 permitted recording of the results to the accuracy of 0.001 mm, at 15 s intervals. The measurements of dimension changes in load direction were continued until total swelling of the samples (72 h), established on the basis of their deformation at  $\sigma = 0.00 R_c$ .

## 3 Results and discussion

### 3.1 Adsorption and desorption

The adsorption and desorption isotherms for thermally modified and unmodified wood samples are presented in Fig. 2. The character of isotherms and the reduction of the EMC of ash wood strongly depend on the treatment temperature: the higher the heat treatment temperature, the lower the EMC (Metsä-Kortelainen et al. 2006). Thermal modification of wood at 180 or 200 °C for 3 h leads to a decrease in EMC corresponding to the air RH near saturation by 18 and 24%, respectively. Moreover, assuming the sorption model



**Fig. 2** Sorption isotherms of thermally modified ash (*Fraxinus excelsior* L.) wood obtained at a temperature of  $22 \pm 1$  °C (adsorption and desorption isotherms depicted with the GAB model; solid dots represent experimental data, dashed line represents extrapolation of fibre saturation point for RH = 100%)

proposed by Guggenheim–Anderson–de Boer (GAB) (Timmermann 2003), the theoretical MC of wood corresponding to the FSP (at air RH = 100%) was calculated:

$$EMC = M_m \frac{100 \cdot K \cdot C \cdot RH}{(100 - K \cdot RH) \cdot (100 - K \cdot RH + C \cdot K \cdot RH)} \quad (1)$$

where EMC; %—equilibrium moisture content, RH; %—relative air humidity,  $M_m$ ; %—monolayer capacity,  $C$ —equilibrium constant related to the monolayer sorption,  $K$ —equilibrium constant related to the multilayer sorption.

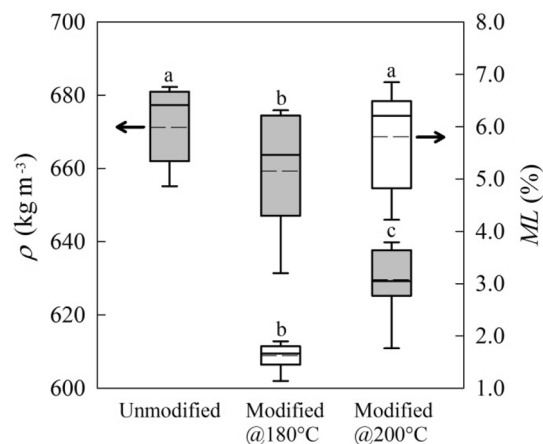
The calculated values of FSP for the unmodified sample and the samples modified at 180 and 200 °C were 28.8, 22.4 and 20.7%, respectively.

### 3.2 Mass loss

Thermal modification of wood resulted in the ML and thus in a decrease in density, to a degree dependent on the temperature of modification (see Fig. 3). The ML of the wood modified at 180 and 200 °C was 1.63 and 5.81%, respectively.

### 3.3 Impact strength

As follows from Table 1, the value of MOE and  $R_c$  in tangential direction at low MC (6%) remained unchanged, while the same parameters significantly increased for the sample under compression in radial direction ( $p < 0.05$ ). For the sample of MC above the FSP, modified at 200 °C, MOE



**Fig. 3** Density ( $\rho$ ) and mass loss (ML) of thermally modified ash (*Fraxinus excelsior* L.) wood; identical superscripts (a, b, c) denote insignificant ( $p < 0.05$ ) difference between mean values ( $n = 34$  for density and  $n = 10$  for mass loss for each set of samples) according to Tukey's HSD test, mean – dashed line

and  $R_c$  in both anatomical directions significantly increased (Table 1).

### 3.4 Creep

Figure 4 presents the creep of ash wood samples compressed perpendicular to the grain and simultaneously wetted from MC close to 6% to MC above the FSP. The strain ( $\epsilon$ ) at a given moment of creep was determined as a ratio of the resulting complex deformation related to its initial dimension in the loading direction. For both modified and unmodified samples, their deformation in tangential direction was greater than that in radial direction. Thermal modification was found to significantly reduce the swelling of wood (at  $\sigma = 0.00 R_c$ ) and the complex strain (at  $\sigma = 0.25\text{--}0.75 R_c$ ); the higher the modification temperature, the higher swelling reduction was observed. In all tests, the applied level of compression stress did not exceed the linear range of viscoelastic wood behaviour, as evidenced by the data presented in Fig. 5.

On the basis of the linear relations between the complex strain and the stress caused by compression loading (Eq. 2), the values of stress at which the dimensions of samples would remain unchanged despite wetting were determined. Table 2 presents the estimated values of Eq. 2 parameters for selected moments of the creep process:

$$\sigma = A \cdot \epsilon + B, \quad (2)$$

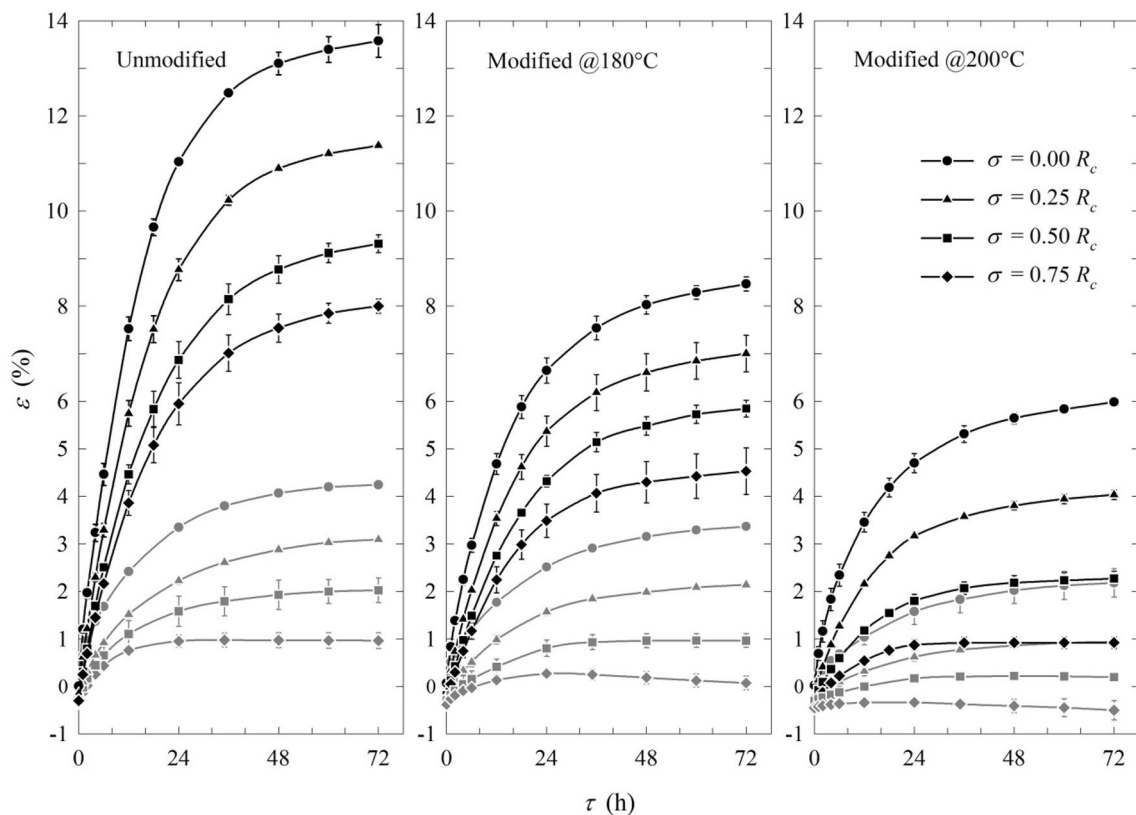
where for  $\epsilon = 0$  the term B is the estimated value of compression stress needed to completely restrain swelling.

The coefficients of determination ( $R^2$ ) take values close to unity, which confirms that practically all relations analysed were linear in the entire range of stress applied.

**Table 1** Modulus of elasticity (MOE) and compression strength perpendicular to the grain ( $R_c$ ) for thermally modified and unmodified ash (*Fraxinus excelsior* L.) wood for different moisture contents (MC)

MC	Treatment option	Tangential		Radial	
		MOE (MPa)	$R_c$ (MPa)	MOE (MPa)	$R_c$ (MPa)
6%	Unmodified	927 <sup>a</sup> ± 109	5.61 <sup>a</sup> ± 0.37	2289 <sup>ab</sup> ± 81	9.04 <sup>b</sup> ± 0.38
	Modified @180 °C	942 <sup>a</sup> ± 100	5.93 <sup>a</sup> ± 0.49	2246 <sup>b</sup> ± 78	9.82 <sup>a</sup> ± 0.36
	Modified @200 °C	909 <sup>a</sup> ± 58	5.60 <sup>a</sup> ± 0.47	2343 <sup>a</sup> ± 65	9.94 <sup>a</sup> ± 0.94
Above FSP	Unmodified	216 <sup>b</sup> ± 28	1.01 <sup>b</sup> ± 0.12	545 <sup>b</sup> ± 24	2.62 <sup>b</sup> ± 0.30
	Modified @180 °C	207 <sup>b</sup> ± 17	1.02 <sup>b</sup> ± 0.07	526 <sup>b</sup> ± 34	2.55 <sup>b</sup> ± 0.14
	Modified @200 °C	274 <sup>a</sup> ± 35	1.67 <sup>a</sup> ± 0.33	808 <sup>a</sup> ± 79	3.59 <sup>a</sup> ± 0.53

Mean values ( $n = 10$ ) ± standard deviations; identical superscripts (a, b, c) denote no significant ( $p < 0.05$ ) difference between mean values in columns according to Tukey’s HSD test



**Fig. 4** Strain (swelling) of thermally modified ash (*Fraxinus excelsior* L.) wood compressed perpendicular to the grain and wetted from MC=6% to MC above FSP, black dots and lines—tangential load,

gray dots and lines—radial load, dots represent mean ( $n=3$ ) values ± standard deviation

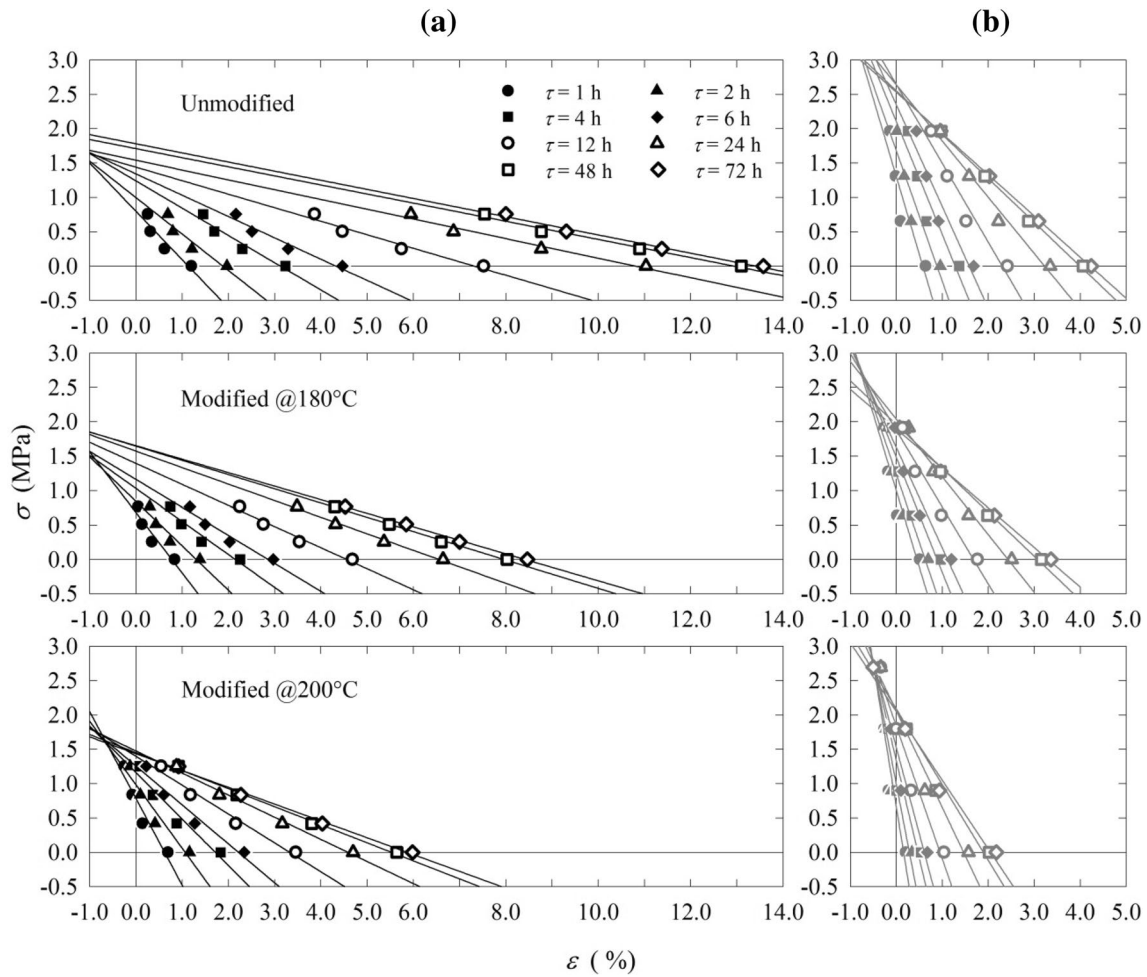
### 3.5 Swelling pressure

Swelling pressure is the theoretical value of stress needed to maintain the initial sample dimensions upon wetting, in other words, to completely prevent the wood from swelling (Moliński and Raczowski 1988; Krauss 2004; Roszyk et al. 2007; Moliński and Roszyk 2010). As follows from the data presented in Fig. 5, the stress needed to maintain the initial sample dimensions has a positive sign. It is obvious if it is taken into account that the compression load applied

must completely restrain the swelling of wetted samples. The stress needed to completely restrain swelling of thermally modified ash wood determined from the isochrones of its mechano-sorptive creep is shown in Fig. 6.

According to the data given in Fig. 6, the stress needed to restrain swelling (swelling pressure) is higher in the radial direction (Fig. 6b) than in the tangential direction (Fig. 6a). The compression stress needed to completely restrain swelling in the tangential direction of thermally modified wood is close to 1.5 MPa, practically independent of the modification



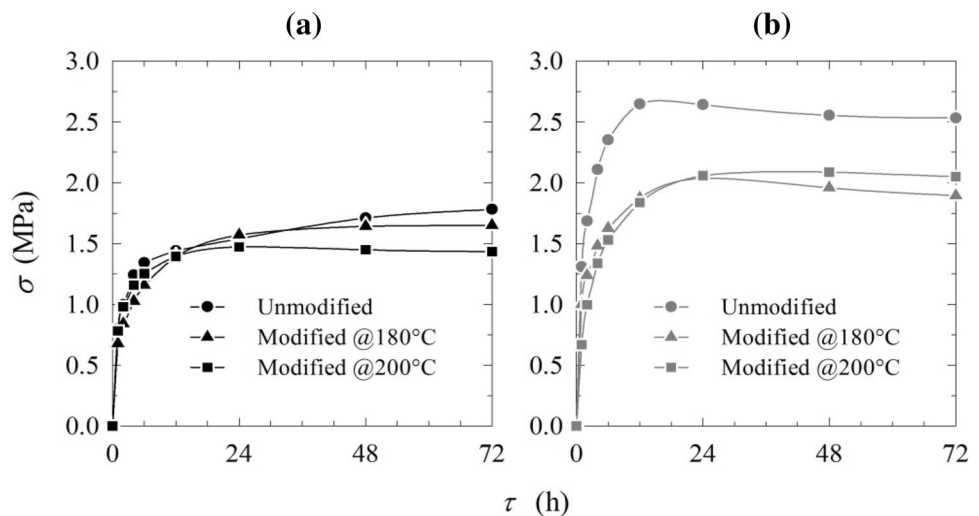


**Fig. 5** Stress ( $\sigma$ ) versus strain ( $\epsilon$ ) curves (isochrones) determined from creep of ash wood subjected to compression perpendicular to the grain and simultaneously wetted in the range MC=6% to MC above FSP, **a** tangential direction and **b** radial direction

**Table 2** Estimated coefficients of Eq. (2)

	$\tau$ (h)	Unmodified			Modified @ 80 °C			Modified @ 200 °C		
		A	B	R <sup>2</sup>	A	B	R <sup>2</sup>	A	B	R <sup>2</sup>
Tangential	1	-0.705	0.798	0.878	-0.878	0.679	0.883	-1.264	0.782	0.924
	2	-0.531	0.998	0.895	-0.646	0.842	0.902	-0.924	0.982	0.921
	4	-0.398	1.241	0.936	-0.480	1.029	0.931	-0.676	1.159	0.937
	6	-0.311	1.343	0.945	-0.407	1.160	0.948	-0.567	1.254	0.953
	12	-0.197	1.441	0.956	-0.306	1.393	0.970	-0.420	1.396	0.977
	24	-0.143	1.541	0.969	-0.240	1.571	0.991	-0.322	1.474	0.989
	48	-0.132	1.711	0.985	-0.207	1.643	0.997	-0.263	1.450	0.993
	72	-0.133	1.782	0.988	-0.196	1.651	0.998	-0.245	1.434	0.994
Radial	1	-2.267	1.309	0.843	-2.224	1.002	0.874	-4.167	0.670	0.921
	2	-1.906	1.686	0.864	-1.977	1.240	0.889	-3.490	0.997	0.919
	4	-1.644	2.108	0.887	-1.672	1.484	0.906	-2.847	1.339	0.928
	6	-1.484	2.353	0.904	-1.460	1.627	0.927	-2.517	1.531	0.940
	12	-1.149	2.648	0.905	-1.117	1.876	0.959	-1.932	1.838	0.957
	24	-0.819	2.642	0.977	-0.839	2.038	0.986	-1.406	2.059	0.967
	48	-0.639	2.555	0.997	-0.638	1.959	0.992	-1.103	2.088	0.974
	72	-0.601	2.533	0.999	-0.575	1.896	0.995	-1.001	2.051	0.979

**Fig. 6** The stress ( $\sigma$ ) needed to completely restrain swelling of thermally modified ash (*Fraxinus excelsior* L.) wood under wetting from MC = 6% to MC above FSP; **a** tangential direction and **b** radial direction



temperature (in the investigated range) and only insignificantly lower by ca. 10% than the value for native wood. In the radial direction this difference is more pronounced. For the thermally modified wood, irrespective of modification temperature, the compression stress needed to restrain the swelling was about 2.0 MPa, while for the native wood it was 2.6 MPa (ca. 20%).

The compression stress needed to restrain the swelling, the so-called swelling pressure of wood, is a positive function of MOE (Keylwerth 1962). The reciprocal of the MOE is known as the compliance. The higher the compliance, the easier is the swelling restriction. However, the results obtained in the present study do not confirm this observation. Although the MOE is practically unchanged in the thermally modified wood sample of low MC (6%) and significantly higher MOE values of the wet sample (MC > FSP) thermally modified at 200 °C, the swelling pressure of the modified sample is lower than that for the unmodified sample and independent of the modification temperature. The latter observation can be explained by the fact that the FSP of the modified wood is smaller than that of the native wood (Fig. 2). Taking into account the fact that the initial MC of all samples was the same, the swelling of modified wood occurred in a narrower range of MC than that of the unmodified sample. However, it does not explain the variation in the mechanical parameters determined upon compression as a function of MC (Table 1). An increase in MC from about 6% to MC > FSP resulted in a decrease in the MOE of the unmodified sample and the sample modified at 180 °C by about 77%, both in tangential and radial directions. The decrease of the modulus of elasticity in the case of samples of wood modified at 200 °C was 70 and 66%, in the tangential and radial directions respectively. An analogous situation was noted for the values of stress at the proportional limit. On the other hand, the difference in FSP of the samples modified at 180 and 200 °C is insignificant (Fig. 2).

## 4 Conclusion

The above presented measurements, their analysis and interpretation permit the following conclusions to be drawn. Thermal modification of ash wood at 180 and 200 °C resulted in the ML of 1.63 and 5.81%, respectively. Despite this ML, the thermally modified ash wood of MC 6% is characterised by practically the same values of MOE and Rc as the unmodified wood. After wetting to MC above the FSP, the wood thermally modified at 200 °C shows significantly higher MOE and Rc than the analogous values for the unmodified wood and the wood modified at 180 °C. The results of the sorption experiment and determinations of the MOE and Rc of wood of different MC prove that the reduction of wood hygroscopicity, which is an inherent effect of thermal modification, is also a reason to limit the range of changes (decrease) in mechanical properties of wood caused by the increase of its moisture to the FSP. Thermal modification exerts an impact on the swelling pressure of wood in the directions perpendicular to the grain to a degree dependent on the temperature of modification. The swelling pressure of thermally modified ash wood is lower by ca. 10 and 20%, in the tangential and radial directions, respectively, compared to the corresponding value of the unmodified wood. This effect results in a decrease in the anisotropy of swelling pressure of thermally modified ash wood. The observed effect reduces the anisotropy of the swelling pressure of thermally modified wood in the transverse directions.

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