

Figure 12. Comparison of restoring time, T_R

4 Conclusions

The performance of the proposed shear wall was investigated by computer simulation using the finite element method. The results could be concluded as follows:

The insertion of a visco-elastic gasket between a particleboard and its surrounding wooden frame could improve the performance of shear wall significantly. A good combination of particleboard with high shear rigidity, and a gasket with effective viscous damping, will result in an efficient distribution and high load bearing capacity. This design is especially useful for structures that are expected to withstand high external forces, such as those arising from typhoon or earthquake.

The performance of shear wall could be upgraded by adjusting the properties of the visco-elastic gasket. The adequate properties of visco-elastic gasket was found out to realize the expected attractions of shear wall, such as the high performance in loading damping, the capacity of energy dissipation and the ability of self restoring. Therefore, this new type of shear wall is available for improving the safety and performance of wooden houses. However, its validity of interpretation is necessary to be verified by the experimental investigations.

5 References

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Thermo-Hydro-Mechanical Densification and Influence of Post-Treatment on Set-Recovery

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Abstract

Thermo-Hydro-Mechanical (THM) densified spruce samples were pre-treated and post-treated under high-pressure saturated steam. Different post-treatment periods and temperatures (max. of 200°C) were used to study the permanent fixation. Pre-treatments before densification to compare the treatment homogeneity with post-treated samples were also achieved. This experience was executed by a pressure-controlled reactor developed for this task.

Soaking-drying cycles of post-treated samples were done to measure the set-recovery. In order to understand the shape memory of densified post-treated specimens at cellular level, transversal micrographs were made by confocal microscope. X-rays diffraction was also carried out to determine the crystallinity index (CrI) of cellulose.

The earlywood cells were deformed before latewood ones as its cell wall is thinner. During set-recovery test the earlywood cells are also the first ones to recover its initial shape due to higher inner stress and deformation. Increasing both the post-treatment temperature and duration results in increase of crystalline cellulose percentage at the expense of amorphous cellulose. Hydrolyse of glucosidic bonds of cellulose which releases the inner stress and new configuration of the amorphous phase seem to be responsible for this increase of crystallinity and for the shape stabilisation. Unfortunately, the higher the temperature and the longer the PT, the darker the colour of the samples according to chemical modifications. The results show that there is a compromise between the temperature and the duration of the PT. To avoid chemical degradations and darkening of the colour one could use lower temperature than 200°C and longer PT.

1 Introduction

A process to densify small specimens of different wood species by Thermo-Hydro-Mechanical (THM) means has been developed by Navi and Girardet (2000) [1]. They used a hydro-thermal chamber with pressured saturated steam at a maximum of 150°C. This apparatus was also used for saturated steam treatments to achieve permanent fixation of the compression set in wood. Although the so treated samples got significant

improvement in mechanical properties and showed a less hygroscopic behaviour when plunged into water, the steam-treatment periods to get permanent fixation were quite long (several hours). Inoue et al. (1993) [2] and Ito et al. (1998) [3,4] had recourse to higher temperature steam treatments (200°C) and needed only two minutes to obtain the same permanent fixation. As the maximum treatment temperature in our previous hydro-thermal chamber was only 150°C, a new steam reactor had to be conceived to reach 200°C. A brief description of it is given in the experimental part.

Tanahashi et al. (1989) [5] studies on the characterisation of steam-exploded wood showed that steaming of white birch (*Betula platyphilla*) wood results in an increase in crystallinity (CrI : from 51 to 67%), micelle width (from 25Å to 52Å), and microfibril width (32Å to 50Å), whereas the lengths of cellulose microfibrils were decreased (to about 2000Å). The conditions of steam-explosion is not far from those of thermo-hydro-treatment; therefore Tanahashi et al. results could explain partially the permanent fixation mechanism of densified wood with high temperature steam.

In this work the effect of pre-treatment and post-treatment conditions on the permanent fixation of the transformed shape and the crystallinity of cellulose of THM densified wood samples were studied. Following, after a brief explanation of THM densification and high temperature steam post-treatment, the results obtained from the set-recovery test, micrograph of cross-sections of the specimens and crystallinity index were reported. Our remarks were given in the conclusion.

2 Experimental

2.1 THM Densification

Small cylindrical specimens of spruce (ϕ : 30mm, h : 40mm) were densified under saturated steam at 140°C in the same hydro-thermal chamber as the one used in Navi and Girardet (2000). A press was used to compress the steam-plasticized samples in radial direction under controlled displacement mode.

The value of the compression set (C_1) is defined as :

$$C = \frac{R_0 - R_c}{R_c} \times 100 (\%) \quad (1)$$

where R_0 and R_c are the thickness of the samples before and after compression respectively.

A diagram of the THM procedure is given in Fig. 1. It consists in five steps: 1 - sample plasticisation at 140°C saturated steam during 20 minutes; 2 - compression by 1mm/min until 12 kN; 3 - drying by rapid steam depressurisation and pressurised air cooling; 4 - cooling; 5 - unloading. The compressed thickness (R_c) is measured after two weeks at room temperature under 60% RH.

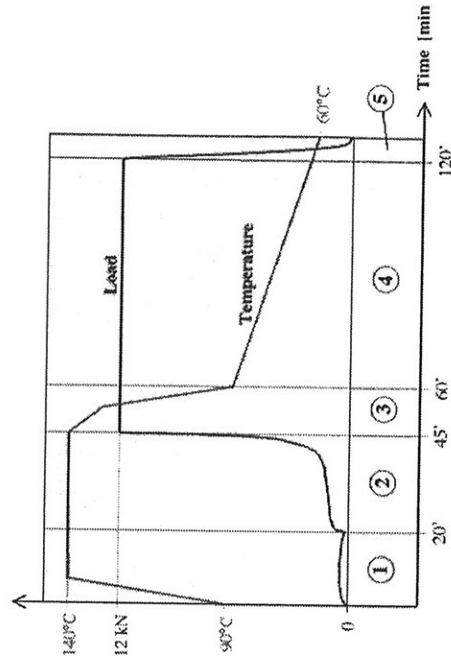


Figure 1. Diagram of the THM densification procedure

2.2 Post-treatment

Post-treatment is defined as a steam treatment of a specimen after its densification. Each densified sample was post-treated in a reactor under saturated steam between 140°C and 200°C during different lengths of time. To keep its compressed shape the samples were placed in a mould. To see the reproducibility and the dispersion of the results each type of post-treatment was done twice. The temperature and duration of post-treatment are shown in Tab.2 and Tab.3.

The THM post-treatment reactor is constituted of one pressure cell where the sample is treated. Water input and steam output are directly connected with the pressure cell. As the post-treatment is controlled by pressure inside the reactor the corresponding temperature at saturated condition was calculated by Clausius-Clapeyron equation given by :

$$\ln p_0 = 13.96 - \frac{5205}{T} \quad (2)$$

where p_0 is the pressure in kg/cm² and T the temperature in Kelvin.

2.3 Comparison between pre-treated and post-treated densified wood

Pre-treatment is defined as a steam treatment of the specimen prior to its densification. Two samples pre-treated and post-treated at 174°C during 16 min were compared. The results are shown in Fig. 3.

2.4 Confocal microscopy observations and cellulose CrI evaluations with X-rays diffraction method

For confocal microscope observation, the samples were impregnated under vacuum at 60°C during 4 hours with a special resin (100g Araldit DY 026 SP, 75g hardener HY

2954) and polished. For X-rays diffraction 1 cm² squared and 2 mm thick specimens with flat surface were prepared.

3 Results

3.1 Densification

Before and after compression the mass, the thickness and the diameter of each sample were measured to calculate its mean density and mean compression set value. Tab. 1 presents the results of densification.

Table 1. Samples mean values before and after compression

	Mass [g]	Thickness [mm]	Volume [mm ³]	Density [g/cm ³]	C [%]
Initial state values	11.18	40.11	28.353	0.394	—
Densified state values	11.68	13.16	9.126	1.280	67.2

Each THM post-treated sample has undergone a set-recovery test, an impregnation for confocal microscope observations and X-rays diffraction to determinate its cellulose crystallinity index (CrI). Therefore the THM post-treated samples were divided in 4 equal parts with a band saw.

3.2 Set-recovery test

In order to determine the effect of THM post-treatment on recovery of compressive transformed shape, a set-recovery test was conducted on each sample. It consists in three soaking-drying cycles (1 cycle = 3 days in distilled water at 60°C followed by 1 day oven-drying at 30°C). After these three cycles the samples were oven-dried at 30°C during two days and finally the recovered thickness (R_c) measured. The set-recovery results for each different temperature of PT are given in Fig. 2 and Tab. 2. The set-recovery (R₁) is defined as :

$$R_1 = \frac{R'_c - R_c}{R_0 - R_c} \times 100 \quad (\%) \quad (3)$$

Table 2. Set-recovery values function of post-treatment temperature and processing time

non post-treated	time	140°C			160°C			180°C			200°C		
		60'	120'	180'	30'	60'	90'	8'	16'	24'	2'	4'	8'
—	74.5 %	18.7 %	2.2 %	0.9 %	2.1 %	0.7 %	-1.9 %	1.7 %	-1.9 %	-2.4 %	0.7 %	-1.4 %	-2.3 %

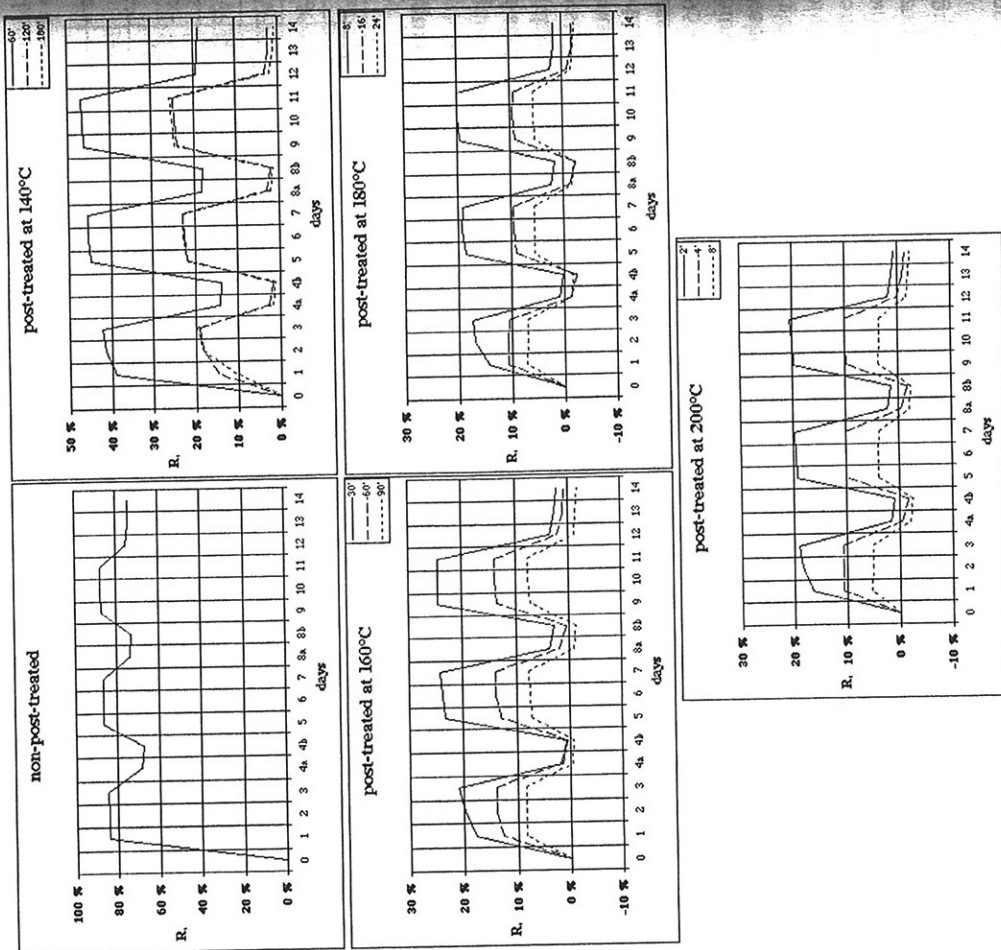


Figure 2. Swelling and contraction curves at radial of densified specimens

3.3 Comparison between pre-treated and post-treated densified wood

The two pre-treated and two post-treated samples underwent the same set-recovery test as is explained in § 3.2 in this case but 10 soaking-drying cycles were done. Fig. 3 shows the evolution of each sample thickness.

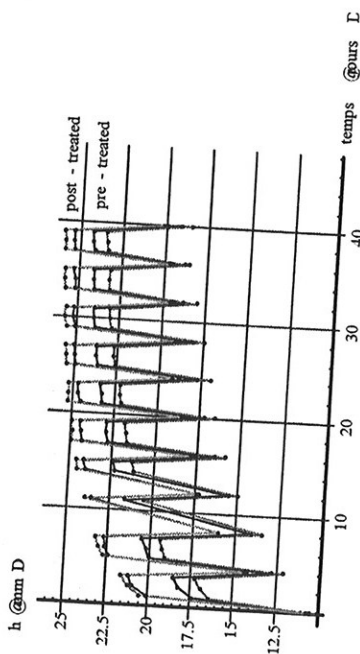


Figure 3. Thickness evolution of the pre-treated and post-treated samples (174°C, 16')

3.4 Cellulose crystallinity index (CrI) evaluations with X-rays diffraction method
 With intent to explain the permanent fixation of compressed wood, cellulose crystallinity was investigated with X-ray diffraction method before and after post-treatment. The method to determinate the crystallinity index (CrI) is given in Wellwood et al. (1975) [6]. The only difference is that we performed X-ray diffraction with massive spruced instead of milled ramie. The empirical method proposed by Segal et al. (1959) [7] was used to calculate CrI, except that the background was designated arbitrarily as the line drawn between 9° and 30° (2 θ) (cf. Fig. 4). This method was chosen as it provides a saving in time and energy in our case.

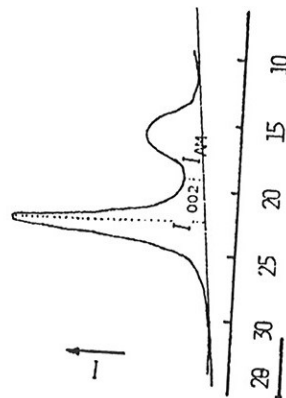


Figure 4. Typical diffractometer trace of cellulose that indicates location of intensity values I_{002} and I_{AM} used in calculating CrI by peak height

$$\text{CrI} = \frac{I_{002} - I_{AM}}{I_{002}} \cdot 100 \quad (\%) \quad (4)$$

where I_{002} is the count (maximum) intensity (in arbitrary units) at the angle of (002) lattice diffraction, and I_{AM} is the count intensity, from amorphous background scatter, in

the same units, at $2\theta = 17.5^\circ$ (approximately). The results of cellulose CrI are presented in Tab. 3.

Table 3. Crystallinity index of cellulose function of post-treatment temperature and lengths of time

non post-treated	140°C		160°C		180°C		200°C	
	60'	120'	30'	60'	8'	16'	2'	4'
—	77.8 %		80.9 %	81.8 %	81.8 %	83.1 %	84.4 %	84.4 %
			80.4 %	81.1 %	84.3 %	85.3 %	87.5 %	87.5 %
			83.0 %	84.4 %	85.3 %			
							16'	87.5 %

4 Discussion and Conclusion

The density is approximately tripled by compression because of the lumens closing during this process. It is important to note that the compression is not maximal; as a matter of fact the spruce porosity is 70 % (mean porosity of 12 typical samples). Consequently about 3 % residual porosity remains.

The densification might also be done in tangential direction, but in this case the wood would keep its anisotropic behaviour between tangential and radial directions, since the wood rays would not be destroyed. The increase of weight of densified wood shown in Tab. 1 may be imputed to the water kept in the pores.

Just after post-treatment many observations have been done. The densified wood colour changed during post-treatment. Treatment degrades the wood constituents at high temperature: the longer the post-treatment period, the darker the colour of the sample. Burned wood smell was also detected with 180°C (16') and 200°C (4') post-treated samples; for lower temperature densified wood exhaled a smell of caramel. In fact hemicelluloses and cellulose are hydrolysed to monosaccharides and glucose respectively, dissolved in water and finally can pyrolyse if temperature is too high (higher than 270°C).

Visible cracks to the naked eye were observed on the 180°C (24') and 200°C (4', 8' and 16') THM post-treated samples. These cracks are attributed to the abrupt depression when blowing off: the pressurised water kept in the sample cannot get out of the sample and cracks the places, which are degraded by the hydrolyse.

Tab. 2 shows that equivalence exists between time and temperature during post-treatment for set-recovery. For instance, 160°C and 90 minutes post-treated sample has the same R_1 as 200°C and 2 minutes post-treated one. Moreover the set-recovery decreases when post-treatment temperature and length of time increase. Therefore long post-treatments at low temperature have the same effect than short ones at high temperature on the set-recovery of wood. This does not mean that if two samples have the same set-recovery they have the same mechanical behaviour and have gone the

same chemical degradation. In fact high temperature post-treated samples got a darker colour and cracked during post-treatment comparing to the low temperature post-treated ones. This fact can be explained by the low thermo-chemical resistance of lignin at temperature above 140°C. Tanahashi et al. [5] worked on chemical changes under steam-explosion condition. Their results are interesting, as the conditions of the steam-explosion (just before explosion) are not far from ours. Because lignin is degraded at high temperature through a homolytic cleavage of β -O-4 ether linkage, it could be removed from its original inter-microfibril location by elution. On the other hand, hemicelluloses could be hydrolysed and their bondings with cellulose and lignin cleaved. In both cases the global result is a lower cohesion between the different components of wood, which can explain the cracking of our samples at 200°C along the middle lamella. The middle lamella contains a high percentage of lignin, higher than in the cells wall and so is exposed to faster chemical degradations than cellulose, which has a higher chemical resistance against hydrolysis. The cellulose has also a much higher thermal softening temperature than lignin. If the lignin is considered as a matrix in which cellulose and hemicelluloses are embedded, the cohesion between wood cells can be decrease in case of lignin cleavage, especially at high temperature (200°C). In any case, this could explain the brittleness of the 200°C post-treated samples. Then the 200°C (4', 8' and 16') THM post-treated samples cannot be used as furniture wood or as structural wood, especially because of its brittleness (cracks).

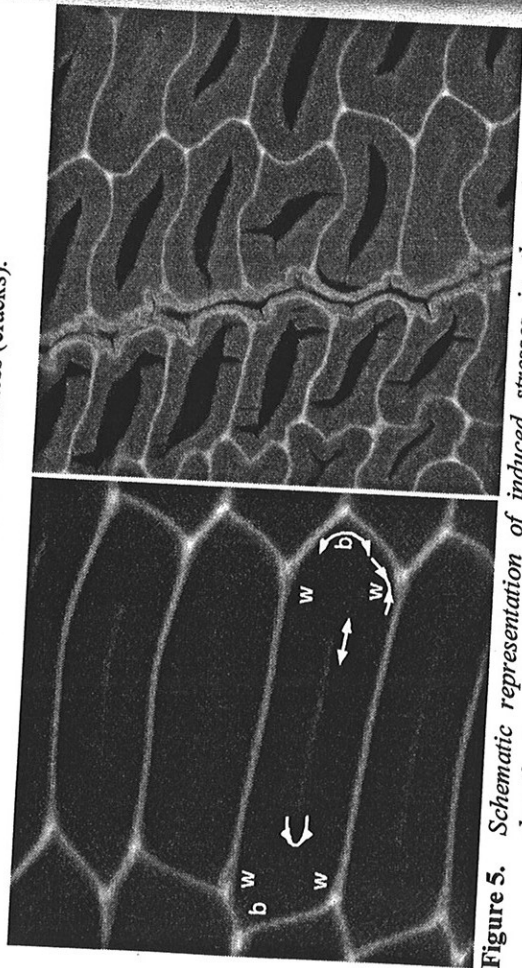


Figure 5. Schematic representation of induced stresses in the cell wall during densification. On the left : external surface of blunt angles (b) and internal surface of wide angles (w) are under traction, when opposite surfaces are under compression. On the right : cracked produced in latewood cells

Concerning the permanent fixation of densified wood an explanation connected with lignin and hemicelluloses degradation is given by Tanahashi et al. The mobility of these

components caused by steaming make cellulose free from other constituents in wood. Therefore the free cellulose is fused together under high pressure to make large microfibrils or crystallites before explosion, suggesting that it could happens during post-treatment. This phenomenon causes an increase of cellulose crystallinity. The clear result of our investigation is that steam post-treatment has increased the crystallinity index. Moreover, the longer the time and the higher the temperature, the higher the Cr Tanahashi et al. [5] proposed three stages in the reaction of cellulose. In the first stage the inner stress in the crystalline region of cellulose is loosened by partial hemicelluloses and paracrystalline cellulose hydrolysis. Then the paracrystalline cellulose can be relocated to the crystalline regions and the width of cellulose microfibrils increases. In the second stage the length of the microfibrils decreases to 1000-2000 Å by cutting at some node of the cellulose crystalline. Finally, the surface of cellulose crystalline is gradually hydrolysed if the treatment is too long at high temperature and the microfibrils width and crystallinity of the cellulose decrease.

During the densification process, the hexagonal cells are subject to a radial compression stress, which forces them to close their lumen and the different angles of the cell to blunt or to widen. This leads to cracks opening of the wide angles during the set-recovery test (see Fig. 5). The residual porosity mainly consists in opened wood rays and slightly opened lumens.

When micrographs are compared before and after the set-recovery test one can clearly observe that the opening of the earlywood cells is the first mechanism which takes place in the shape memory of wood as they have undergone bigger deformation and stored higher inner stress than latewood cells during densification. This opening seems to begin at the earlywood-latewood cells interface and to progress as the recovery increases (Fig. 6).

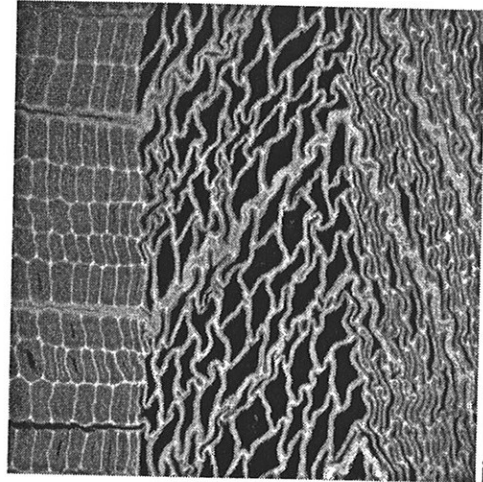


Figure 6. Earlywood cells close to the early-latewood interface, opened after set-recovery test

It is not clear when latewood cells start to open. We can only say that it happens after the first earlywood cells open; obviously it depends on the distribution of the inner stress between the cells.

Finally one can observe a difference between the behaviour of the central and the boundary cells of the samples especially 140°C post-treated densified (Fig.8). The result is a bigger opening of the latewood lumen at central part and a curved form at macroscopic level (Fig. 7). On the other hand in pre-treated samples the lumens are open during the pre-treatment, which makes densified wood more homogeneously treated, and so water can easily reach the center of the sample. Fig. 9 clearly shows colour differences between central part and boundary part of the post-treated sample when pre-treated one is homogeneous.

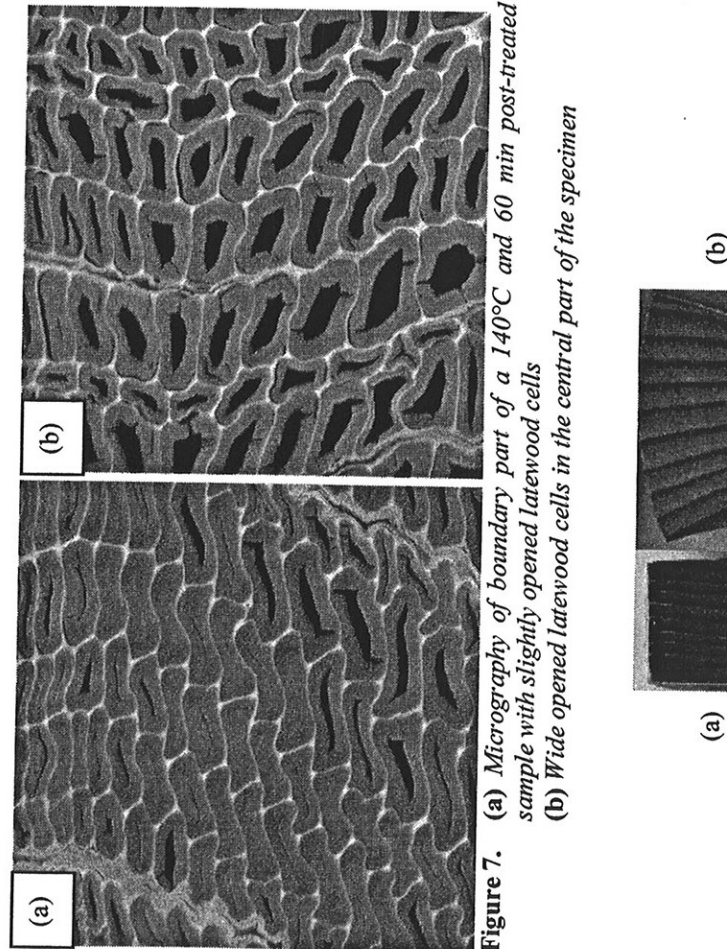


Figure 7. (a) Micrography of boundary part of a 140°C and 60 min post-treated sample with slightly opened latewood cells
(b) Wide opened latewood cells in the central part of the specimen



Figure 8. (a) Photo of the Fig. 7 sample before set-recovery test
(b) During the set-recovery test the thickness of the central part increased more than this of the boundary part

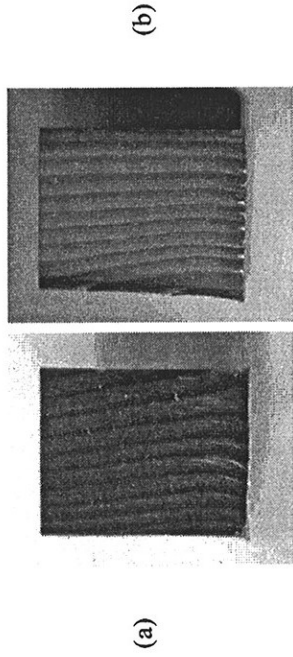


Figure 9. (a) Pre-treated densified wood
(b) Post-treated densified wood

4 Acknowledgement

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