3rd International Conference on Bio-Based Building Materials

June 26th - 28th 2019
Belfast, UK

THERMO-STRUCTURAL BEHAVIOR OF SOFTWOOD UNDER HIGH X-RAY TOMOGRAPHY RESOLUTION

K. Abahri*, D. Kosiachevskyi, A. Rima, C. El Hachem, M. Chaouche
LMT-Cachan, ENS Cachan, CNRS, Université Paris Saclay, 61 Avenue du Président Wilson, 94230 Cachan, France.
*Corresponding author; e-mail: kamilia.abahri@lmt.ens-cachan.fr

Abstract

This study consists in better understanding of the influence of the temperature variations on softwood morphological and structural properties. In this context, the X-ray tomography protocol has been used to follow the property variations, a specific tomographic platinum was used to generate thermal flows and to solicit the sample during scans. The study was carried out for temperatures between -5°C and 40°C. The numerical treatments allowed us to investigate the evolution of three microstructural properties of the wood: porosity, specific surface and the pores diameter. In addition, the material exhibits hysteresis behavior along a heating and cooling cycle caused by the anisotropic changes expressed by the swelling / shrinkage of the wooden cell walls. To better quantify these morphological variations, a Digital Image Correlation (DIC) were investigated and the deformation field were calculated function of the wood anatomical structure for both earlywood and latewood. In the tangential direction, the deformation presents a maximum of 1.5 times larger than the radial one. In addition, the deformations caused by heating are larger than those of the cooling cycle. This study provides the literature and future research with promising results especially on the evolution of the wood morphology for better prediction of its macroscopic hygro-thermal behavior.

Keywords:
Thermal solicitations; X-ray tomography; microstructure; anisotropy; Digital Image Correlation

1 INTRODUCTION

The use of building materials based on plant fibers, such as wood, can reduce energy consumption and environmental impacts in contrast to conventional materials.

In this work, the studied material is softwood, which is particularly known for its thermal, acoustic, structural and even hygric performances. Despite the many functions it provides, wood is a hygroscopic material that is very sensitive to hygrothermal stresses and consequently undergoes dimensional variations (swelling, shrinkage) when the relative humidity increases or decreases [Ranfsanjani et al, 2013]. These dimensional variations are likely to modify the properties of wood and create degradation. For better understanding of the wood behavior towards the thermal and hydrous stresses, it is necessary to describe its microstructure and then its interaction with these stresses.

In fact, this material presents a complex, anisotropic and heterogeneous aspects. Its structure can be described at different levels: macroscopic scale of the tree or microscopic cells. Softwood is a porous material formed of tracheid's, parenchyma and pores called lumens [El Hachem et al, 2019]. These pores contain variable proportions of water, vapor and dry air. We distinguish two types of wood: earlywood and latewood.

Earlywood tracheids have thin walls with a large inner diameter allowing greater sap flow and hence low density. On the contrary, latewood tracheids have thick walls with a small inner diameter and a higher density [EL Hachem et al, 2018]. We distinguish three orthogonal planes: transverse plane (RT) perpendicular to the axis of the stem and on which are observed the annual rings, radial plane (RL) passing to the center of the stem and the tangential plane (TL) in an eccentric plane and parallel to the axis of the stem [Mäkinen et al, 2007 and Stamm, 1964]. These three longitudinal, radial and tangential directions are the directions of anisotropy of the wood whose hygrothermal behavior differs according to the plan.

Against this anisotropy, few works in literature studied the impact of the hydrothermal solicitations on the wooden materials.

[EL Hachem et al, 2019] presented an experimental and numerical approach for prediction of the microscopic hygro-mechanical behavior of spruce wood. Their 3D study allows the estimation of estimation of the Poisson coefficient and swelling coefficient of the cell walls function of the relative humidity. These authors evaluated in another paper the Fiber's hygromorphic effect on thermal conductivity of wooden fibrous insulation using X-ray tomography [EL Hachem et al, 2017]. [Bennai et al, 2017] investigated the deformation and displacement fields of biobased aggregates in mortar subjected to hydric stress. The displacement and deformation fields were obtained by 2D Digital Image Correlation technique (DIC). In other work, finite element simulations approach were elaborated to analyze the influence of material structure on the effective thermal conductivity of theoretical porous materials [Carson et al, 2003].

Most of these researchers focus on the effect of relative humidity on dimensional variations. In fact, the impact of temperature on the microstructure of bio-sourced materials and their durability is a problem that is poorly addressed in the literature. That's why this paper proposes a scientific approach to characterize the morphological variations of softwood towards thermal stresses. To attend this goal, different approaches based on non-destructive imaging techniques have been developed. An experimental investigation was conducted to estimate the percentage of swelling / shrinkage generated by these solicitations.

2 EXPERIMENTAL PROCEDURE

The scientific interest of this work consists in a better evaluation of the morphological behavior of softwood during heating and cooling cycles. In fact, the microscopic characterization at the cell walls scale is a necessary step that permits to apprehend the material's macroscopic behavior. Hence, we will first describe in this part the adopted experimental procedure using X-ray-tomography with thermal flux generation. Then, the DIC experimentation for will be presented. Finally, the DIC uncertainty assessment and the X-ray tomography reconstruction will be presented. Both, DIC and X-Ray tomography procedures are complementary for the measurement of the dimensional variation under thermal stress.

2.1 Characterization by X-ray tomography

X-ray tomography is a non-destructive technique that allows to obtain the three-dimensional reconstruction of the studied object and subsequently the actual microstructure of the sample. After preparation of the sample, it is placed between an X-ray source and a camera. This last records the attenuation spectrum after the passage of radiation through the object. These recordings are repeated at the same time as the X-ray source and the camera rotate around the sample to obtain a series of 2D images that allows us to do 3D reconstruction of the sample. The used X-ray micro-tomography device is X-50, at the Laboratoire de Mécanique et Technologie (LMT) [Roux 2008].

To take into account the impact of thermal stress, a tomographic plate (Figure 1) is used. It generates heat flows and solicit the studied sample, which is of 1 cm diameter of a maximum height of 5 cm. The sample is covered by a carbon cylinder which allows X-rays to pass through. Temperature conditioning is done using a probe that heats at a rate from 2 to 10°C/min. The temperature of the probe varies between -10°C and 160°C. In the present study, X-ray tomography scans were performed at temperatures between -5°C and 40°C and at intervals of 15°C

Figure 1. Tomographic stage for the generation of thermal flows

2.2 Characterization by heat DIC

2D digital image correlation is a non-destructive method that allows determination of displacement and strain fields between two instants of acquisition. After the conditioning of sample, it is placed in a desiccator, which is equipped with a camera. PCO-EDGE camera with a pixel size of 6.5 x 6.5 µm was used. The HUBER Bath Circulator connected to PT100 in the desiccator was used to control the temperature. A fan lets to accelerate the heat exchange. Silica gel was used to limit the relative humidity at 0% (Figure 2).

The softwood sample size is 22,39(±0,0468) x 19,81(±0,03) x 9,5(±0,226) mm^3 (measured as the average of 6 values with the calipers «Facom 1320» with the precision 0,01mm). In order to study the hysteresis two situations were studied: passages from 30°C to 60°C (Figure 3) and from 60°C to 30°C. Time to reach the thermal balance was 2 hours after the change of the temperature.

2.3 DIC uncertainty assessment

There are several causes of measurement errors, such as off-plane movement, imperfect positioning of the pixel coordinates due to geometric defects in optical devices, or the intrinsic noise of the images, etc [Akkaoui et al, 2017]. The determination of correlation resolution is essential to receive reliable results with the smallest possible calculation uncertainty. In order to determine the zone of interest (ZOI) resolution that can give the smallest uncertainty, the uncertainty study was made. Thus, the values of the standard deviation of the deformation and displacement fields were determined in the case of ten correlations between successive images of the sample in the thermal equilibrium for each type of wood (transverse or radial, latewood or earlywood).

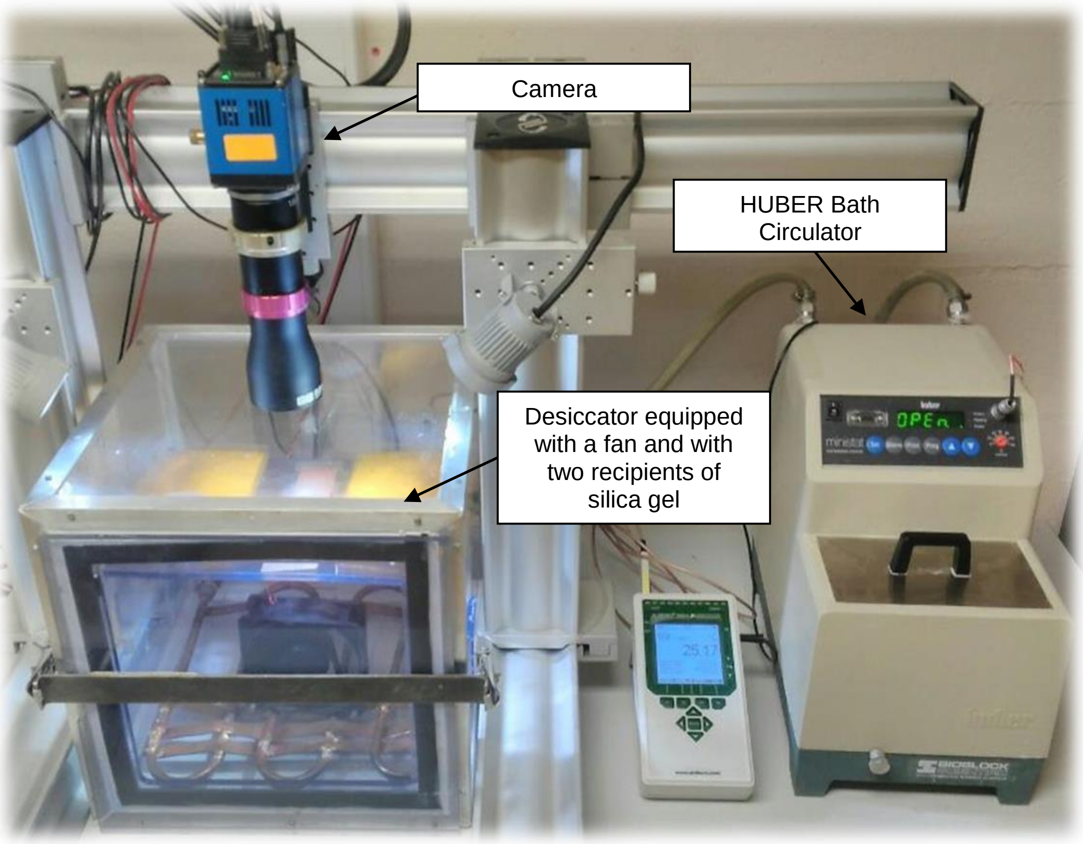

Figure 2. Experimental device for digital image correlation

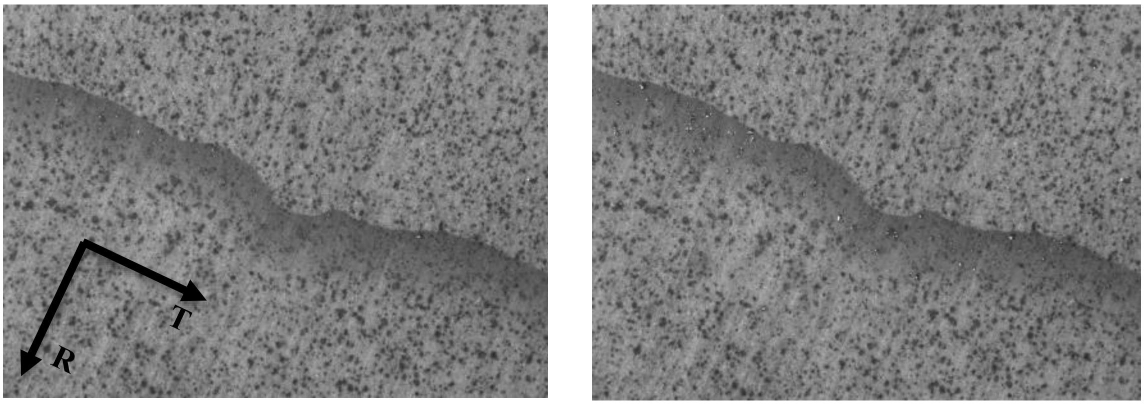

Figure 3. Reference image of the sample at 30°C, 0% RH (left) and last image of the sample at 60°C, 0% RH (right) with the signification of microstructural directions: radial (R) and transverse (T)

3 RESULTS AND DISCUSSIONS

The thermal stresses exert a great influence on the properties of wood and its morphology. In particular, when the temperature is below the freezing point of the water, there will be gel formation and consequently a change in the properties of the wood. That’s why, we tested a large temperature domain [-5°C, 40°C].

Concerning the micro-tomographic tests, a cylindrical wood sample of 5.5 mm diameter and 8.89 mm height were cute and placed on the platen and covered by a cylinder to maintain the temperature and reduce the transfer with the outside. The sample was taken inside the tomograph. For each temperature level and when the sample reaches its equilibrium, a scan will be performed to obtain a series of images and make the 3D reconstruction of the sample. The sample was heated for 2 hours at each temperature. The initial position of the sample must be retained and the same zone of interest (ZOI) selected each time in order to compare and analyze the variations of the wood's morphology with respect to the thermal stresses. The processing of images from the scan is done using the software ImageJ and iMorph. First, the sample is placed at 40°C and is scanned using tomography. Once the scan is complete, the sample is placed at 25°C until the new equilibrium state is reached and the scan is resumed. This step is repeated for two additional temperatures 10°C and -5°C.The same zone of interest (ZOI) is carefully selected (Figure 4) for each image series. The dimensions of ZOI were 400x400x400 pixels^3 where each 1 voxel corresponds to 2.7 µm. For each temperature level, the porosity, the specific surface and the particle size are calculated.

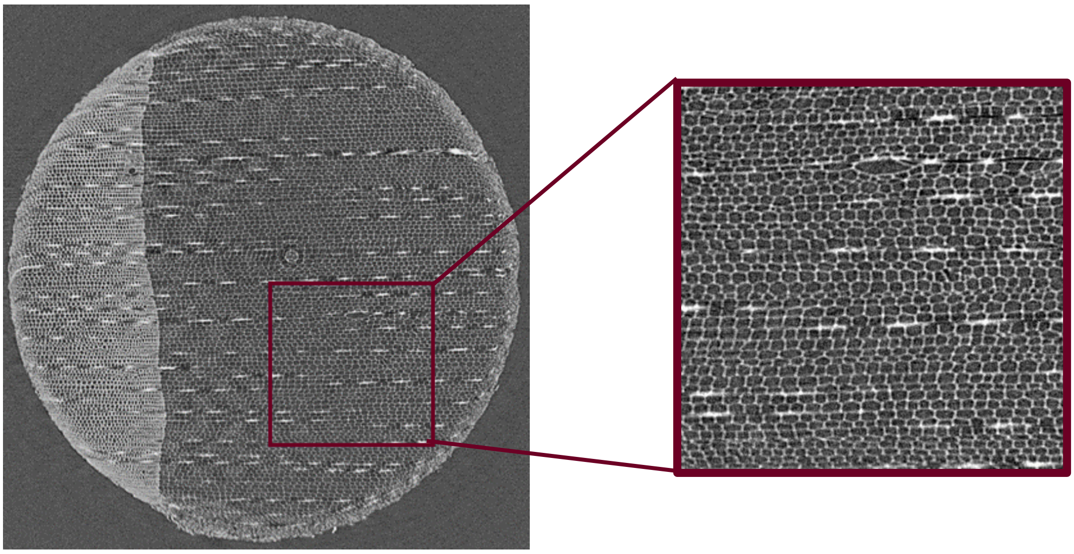
(a) (b)

Figure 4. 2D image of wood at 40°C from tomography scan: (a) Initial image (b) zone of interest (ZOI)

3.1 X-ray tomography

First, comparison between the different characteristics of the wood for different temperatures has been made. Then we studied the variation in pore size distributions, porosity and pore surface area, by decreasing the temperature from 40°C to -5°C incrementally with an interval of 15°C. Results are shown, respectively in Figure 5, 6 and 7.

Figure 5 shows changes in lumen diameters calculated for the following temperature: -5°C, 10°C, 25°C and 40°C. For the 4 temperatures, the average value of the pore diameters varies between 21 and 25 µm (Figure 5). The diagram presents two distinguished peaks, the first corresponding to small cells representing latewood phase, and the second one to big cells representing earlywood phase. The latewood/earlywood specimen contains the most of small lumens, and less of the biggest lumens. This distribution is affected by the temperature variation and no particular tandem can be shown. In order to understand these deviations on the pore diameters, the study of the evolution of the porosity and the specific surface area seems necessary.

Figure 7-a and 7-b show the variation of porosity and specific surface area for cooling and heating cycle of the fourth selected temperatures. Initially, at 40°C, the wood sample contains constitutional water. Once the temperature decreases to 25°C, the porosity decreases from 69% to 60% with an increase in pore surface area (Figure 7-b). This is due to the increase in water content in the wood sample. In this case, the fibers absorb this quantity of water which causes the swelling of the wood and the bound water takes volume, as indicated in figure 6. In addition and after swelling, the pore sizes decrease and the connected pores become unconnected (closure of the porosity) as shown in Figure 7-a. This configuration verifies the increase of the specific surface (figure 7-b). When the temperature reaches 10°C, the porosity of the medium increases and the pore surface area decreases with increasing pore sizes. The porosity increase can be explained by the interaction of the hemicellulose which is sensitive to the variation of the temperature. In fact, at 10°C, hemicellulose will react and change morphology. This causes the modification of the structure and opens the porosity again.

Once the temperature becomes -5°C, below the freezing temperature of the water, the bound water and constitutional water (gel) in the wood sample expands and then the volume of the water content increases. This causes the increase in porosity and the specific surface area of the pores (creation of small pores due to the dilation of the water).

In the inverse scheme (during the heating cycle) and increasing the temperature gradually with an interval of 15°C, the same behavior is noticed with the appearance of the hysteresis. When the temperature decreases from -5°C to 10°C, the bound water will melt resulting in reduced porosity.

When the wood is subjected to cycles of heating and cooling, the maximum variation of the porosity is equal to 15%. It is the results of the anatomical structure of the softwood for such small size of the sample. It is also the results of the interaction between water content and the wood constitution water function of the temperature.

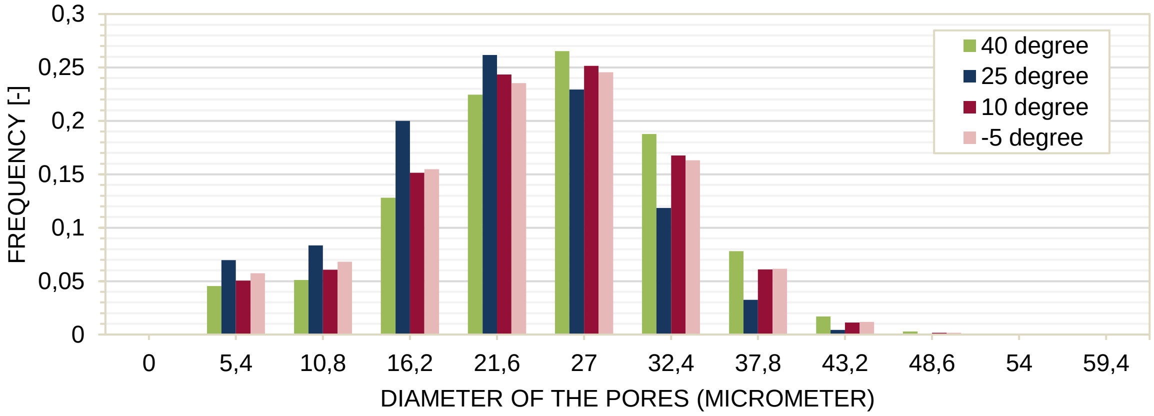

Figure 5. The pore diameter distribution (μm) for different temperatures for wood 400x400x400pi3

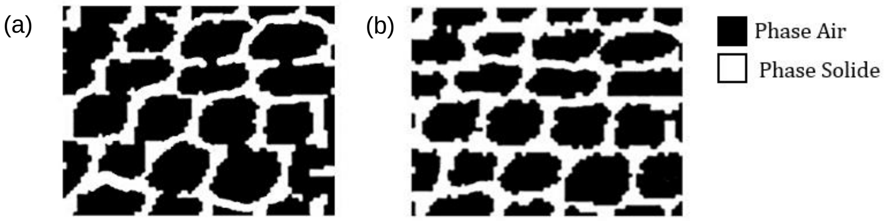

Figure 6. Evolution of porosity by decreasing the temperature from (a) 40°C to (b) 25°C

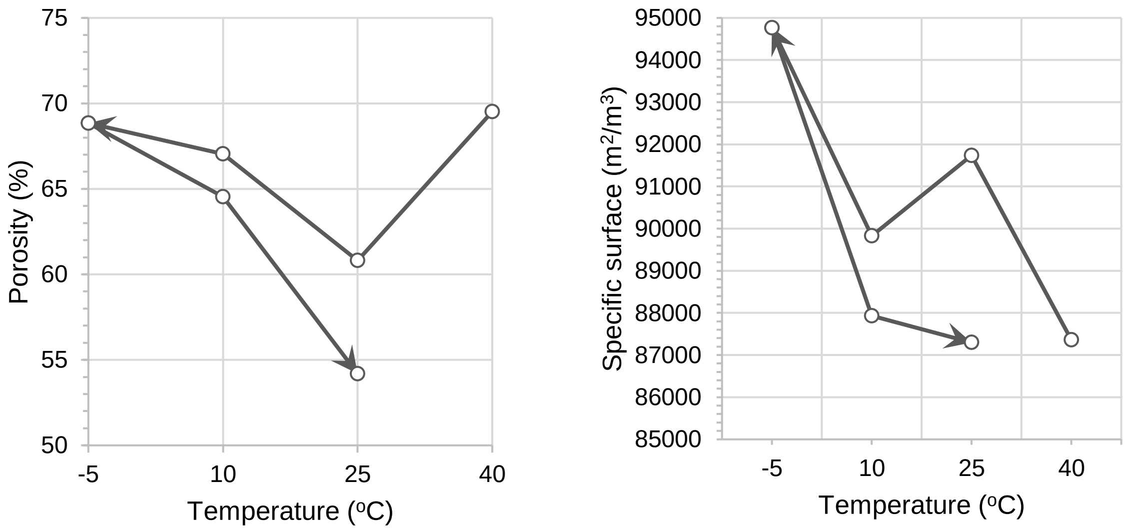

Figure 7. Variation of (a) porosity (%) and (b) specific surface area (m^2 / m^3) as a function of temperature (°C)

3.2 Digital Image correlation

In order to better characterize the thermo-morphological behavior of the softwood and complete the X-ray tomography results, anatomical comparisons of the thermal deformations been performed using DIC.

Figure 8 show comparisons for both cases of heating (from 30°C to 60°C) and cooling (from 60°C to 30°C). It can be observed that the deformations are anisotropic and dependent on the microstructure (on the type of wood and the direction).

For the heating case, we can see that deformations of latewood are larger than those of earlywood in two directions. This is related to the difference in the density of cellular walls of latewood and earlywood. For both, earlywood and latewood, transverse deformations are larger than radial ones.

In the case of cooling, it can be observed that the same dependence on the type of wood and the directions remains.

At the same time, we can see that the deformations caused by heating are larger than those of the cooling. Therefore, the materiel shows highly anisotropic behavior towards thermal solicitations.

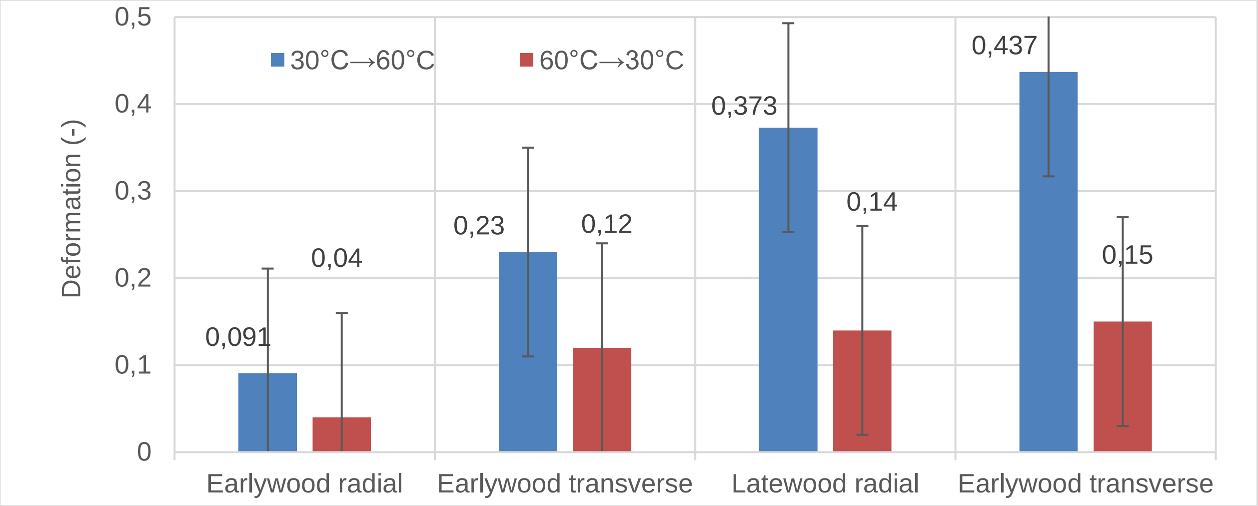

Figure 91. Deformation values for different types of wood (earlywood and latewood) in fonction of the direction (radial and transverse) dor two cases: of heating (from 30°C to 60°C) and cooling (from 60°C to 30°C)

4 CONCLUSIONS

In this work, softwood's thermal/morphological characterization was studied, using two no-destructive experimental methods: micro-tomography and digital image correlation.

Based on the micro-tomographic scans, the evolution of the porosity and the specific surface area were calculation for the heat and cooling cycle. These evolutions were calculated at resolution of 2.7 µm/pixel sufficient to provide refined microstructural information concerning cell walls/pores interface properties. Post-processing of the reconstructed volumes on iMorph served as well to pores' diameters calculation. In fact, although this high resolution leads to have access to very small details like pits and cells' roughness, it presents as well some limitations because of the small scanned volume, which might not be a representative one, of course depending on the purpose of the considered study.

When subjected to heating and cooling cycle, wood presents a hysteresis as to the evolution of the porosity and the specific surface area. The maximum hysteresis deviation presented for these two parameters was obtained for the 25 °C and 40°C. This is expressed by the evolution of the bound water content in the wood during drying, which modify the wood cell thickness and therefore the material porosity.

Further, the DIC results show different structural dimensions for earlywood and latewood, as well as high standard deviations for each parameter in both wood types. Obviously, both wood types' differences, which cause the anisotropy of this material, presents a considerable influence on the thermal deformations as well as on the softwood microstructural properties

This study provides future researches with sufficient refine structural properties concerning spruce wood's microscopic characterization. These results are primordial for either transfers or mechanical wood studies, and serve as well as input parameters for physical simulations.

ACKNOWLEDGMENTS

The technical support of this work has been financially supported by the French "Agence Nationale de la Recherche" through the ''Investissements d'Avenir" program (ANR-10-EQPX-37 MATMECA Grant). The authors would like to thank Fares Bennai and Benjamin Smaniotto for their support and useful discussions.

5 REFERENCES

[Ranfsanjani et al, 2013] Ranfsanjani, A.; Lanvermann, C.; Niemz, P.; Carmeliet, J.; Derome D.; *Multiscale analysis of free swelling of Norway spruce*, Composites Part A 54, 70–78.

[EL Hachem et al, 2019] El Hachem, C.; Abahri, K.; Bennacer, R.; Original experimental and numerical approach for prediction of the microscopic hygro-mechanical behavior of spruce wood, Construction and building materials 203, 258-266.

[EL Hachem et al, 2018] El Hachem, C.; Abahri,K.; Vicente, J.; Bennacer, R.; Belarbi, R.; *Hygromorphic characterization of softwood under high resolution X-ray tomography for hygrothermal simulation*, Heat and Mass Transfer, 54, Issue 9, 2761-2769.

[Mäkinen et al, 2007] Mäkinen, H.; Jaakkola, T.; Piispanen, R.; Saranpää, P.; Predicting wood and tracheid properties of Norway spruce, For. Ecol. Manage. 241, 175–188.

[Stamm 1964] Stamm, A.J.; *Wood and Cellulose Science*, Book, 1964

[EL Hachem et al, 2017] El Hachem, C.; Ye, P.; Abahri, K.; Bennacer, R.; *Fiber's hygromorphic effect on thermal conductivity of wooden fibrous insulation characterized by X-ray tomography*, Construction and Building Materials 150, 758–765.

[Akkaoui et al, 2017] Akkaoui, A.; Caré, S.; Vandamme, M.; Experimental and micromechanical analysis of the elastic properties of wood-aggregate concrete. Construction and Building Materials 134, 346-357.

[Bennai et al, 2018] Bennai, F.; El Hachem, C., Abahri, K. ; Belarbi, R.; Microscopic hydric characterization of hemp concrete by X-ray microtomography and digital volume correlation. Construction and Building Materials, 188, 983–994.

[Carson et al, 2003] Carson, J.K.; Lovatt, S.J.; Tanner, D.J.; Cleland, A.C.; *An analysis of the influence of* *material structure on the effective thermal conductivity of theoretical porous materials using finite element simulations.* International Journal of Refrigeration, 26, 873-880.

[Roux 2008] Roux, S.; Hild, F.; Viot, P.; Bernard, D.; Three-dimensional image correlation from X-ray computed tomography of solid foam, Composites Part A 39, 1253–1265.

.