

## MICROSCOPIC INVESTIGATION OF DEFECTS IN THERMALLY COMPRESSED POPLAR WOOD PANELS

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### ABSTRACT

The combined effects of temperature and compression on the microstructure of solid-wood panels produced by Aspen (*Populus tremula*) wood were evaluated. Thermal compression was applied on aspen wood to increase the density for improving its physical and mechanical properties. The solid wood panels with dimensions of 100 mm by 500 mm by 25 mm were hot-pressed by using a laboratory hot press at a temperature of either 150 °C or 170 °C and pressure of either 1MPa or 2 MPa, respectively, for 45 min. Changes in the microstructure were detected by using a light microscope. The microscopic investigations revealed that the wood exhibited much defects in the process conditions of 170 °C / 2 MPa, and the distribution of defects were not uniform in the growth rings of the wood specimens in the two treatment groups. All defects in cell structure were quite distinct in the beginning and the last parts of the growth rings and the largest damages occurred in the fibers and vessels for the two process conditions. The results indicate that growth ring structure, vessel porosity, and cell wall thickness have a strong effect on wood behavior in various process conditions.

**Keywords:** Cellular failure, poplar wood, thermally compressed wood, wood densification, wood microstructure.

### INTRODUCTION

In comparison to many other materials, wood is unique. It has been used throughout the history mostly because of its low cost, renewability, strength properties, and low processing energy requirements. Recently, there has been an increasing demand for industrial wood due to population and economic growth in the world. It is thought that timber production may not be able to meet the needs of forest products industry in the future. To overcome this phenomenon, the plantation forests and the fast growing tree species have received great attention during the last years (Hill 2006). However, wood formation with low density restricts the industrial usage of timber from fast-grown tree species. As known, most of the mechanical and physical properties of wood are clearly correlated with its density (Blomberg 2006).

Wood density can be increased by using some densification techniques (Blomberg 2006). Thermal compression of wood has been in use since World War II (Seborg *et al.* 1945), and compressed wood products using with low density wood species have been also produced in 1980s especially for utilization of some fast growing tree species (Norimoto 1993, Norimoto 1994, Wang *et al.* 2000).

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Thermal compression is a wood modification method that combines thermal and mechanical processes and results in densification of wood. The main purpose of combining compression and heat on wood is to improve physical and mechanical properties as well as overcome weakness regarding low wood density and low surface hardness of fast growing trees (Gong and Lamason 2007, Tu *et al.* 2014, Guo *et al.* 2015). On the other hand, deformation by compression produced during densification process is not stable and the wood reverts totally or partially to its original shape after re-moistening and heating (Blomberg *et al.* 2006, Guo *et al.* 2015).

Improvements in the quality of timber are considerably related to changes in the chemical components in cell wall structures during thermal treatments, especially the degradation of amorphous carbohydrates (Fengel and Wegener 1984, Tjeerdsmas *et al.* 1998, Boonstra and Tjeerdsmas 2006, Boonstra *et al.* 2007, Kamdem *et al.* 2002, Awoyemi and Jones 2011). Although desired changes in wood properties start to appear at about 150 °C (ThermoWood Handbook 2003), damages in wood microstructure become frequent at 150 °C and above (Fengel and Wegener 1984, Terziev *et al.* 2002, Boonstra *et al.* 2006a, Boonstra *et al.* 2006b, Awoyemi and Jones 2011, Altgen *et al.* 2015, Batista *et al.* 2015).

Even though changes in the anatomical structure of wood at various temperatures have been studied in detail, there is limited information about the microstructure of different wood species subjected to thermal compressing process.

Dogu *et al.* (2010) found that the distribution of deformation was not uniform in the growth rings and the highest deformation occurred in earlywood regions for all process conditions (temperature of either 120°C or 150°C and pressure of either 5 or 7 MPa for 1 h) on thermally compressed Scots pine wood. Buckling and ruptures were detected particularly cell walls of the earlywood tracheids, also separations between compound middle lamella were detected. Cell-wall thickness was evaluated as an important factor in wood behavior during thermal compressing processes. The study showed that a homogenous structure of growth rings with the uniform earlywood and latewood widths plays a major role in prevention of cell collapse.

Tirak Hizal *et al.* (2012) noticed that the largest damages were particularly in the axial parenchyma and vessel cell walls of thermally compressed river red gum wood. Cell collapse occurred in almost all axial parenchyma for all process conditions (temperature of either 150°C or 180°C and pressure of 2 MPa for 45 min.) and severe damages were observed in the cell walls of vessels and ray parenchyma at the highest temperature.

Examination of compressive deformation of hybrid poplar (*Populus deltoids* x *Populus trichocarpa*) wood in saturated steam, transient condition and in superheated steam at 150°C, 160°C, and 170°C revealed that the wood was densified without cell wall fracture in all treatments. It was assumed that the wood samples contained tension wood in high degree, and therefore the storage modulus and softening temperature were lower than normal wood (Kutnar *et al.* 2012).

Microscopic observation revealed that the deformations present in thermo-mechanically densified *Populus tomentosa* wood arose from the viscous buckling of cell walls without fracture. Collapses in the vessels and flattening in the direction of the compression were determined (Tu *et al.* 2014).

Dogu *et al.* (2015) examined the microstructure of thermally compressed paulownia wood at a temperature of either 150°C or 170°C and pressure of 2 MPa for 45 min. and reported that deformations in the cell walls were promoted by increased temperature. Microscopic investigation showed that deformations were especially on axial parenchyma, fiber and vessel cell walls.

Physical and mechanical properties of the aspen wood were examined by Candan *et al.* (2013b). It was stated that the moisture content (MC) of the aspen wood panels decreased with increasing press temperature or press pressure. The average MC value of the unmodified aspen wood panels was around 15% while the MC value of the panels modified with 170°C was around 10%. The average density values of the aspen wood panels increased by press pressure. As for mechanical performance, janka hardness values were improved by the thermal compression technique. The maximum increase in the

janka hardness property was around 32%.

Depending on process conditions, thermal compression process could also affect physical (Wang *et al.* 2000, Epmeier *et al.* 2003, Wang and Cooper 2005b, Gong and Lamason 2007, Johansson 2008, Candan *et al.* 2010, Candan *et al.* 2013b), mechanical (Wang and Cooper 2005a, Unsal and Candan 2007, Unsal and Candan 2008, Abraham *et al.* 2010, Unsal *et al.* 2011a, Unsal *et al.* 2011b, Kutnar *et al.* 2012, Candan *et al.* 2013a) and durability (Kamdern *et al.* 2002, Edlund and Jermer 2004, Hakkou *et al.* 2006, Unsal *et al.* 2009, Boonstra *et al.* 2007) properties of wood.

Wood properties in nature vary among tree species, between trees of the same species, between pieces from the same tree as well. The behavior of wood in different process conditions will be most likely different. Therefore, it is important to know the effects of modification methods in anatomical structure to evaluate the success of the methods. Aspen (*Populus tremula*) is one of the fast growing tree species in Turkey. Wide availability and the low cost are some of the advantages of that wood species. However, it has also some undesired properties such as low surface hardness, low density, low dimensional stability and some drying problems (Candan *et al.* 2013b). In the recent study, thermal compression was applied on aspen wood to improve those undesired properties (Candan *et al.* 2013b). The aim of this study was to evaluate the effects of varying temperatures and press pressures on the microstructure of solid-wood panels produced by using aspen wood.

## MATERIALS AND METHODS

This study was performed on wood samples, which exposed to two different thermal compression process, were taken from the materials examined by Candan *et al.* (2013b). Aspen solid wood panels having no defects with dimensions of 100 mm by 500 mm by 25 mm were hot pressed using a laboratory hot press at a temperature of either 150 °C or 170 °C and pressure of either 1 MPa or 2 MPa, respectively, for 45 min. A total of four panels (two compressed panels from each treatment group) were tested. The panels were pre-dried to a moisture content of 15% before hot pressing (Candan *et al.* 2013b).

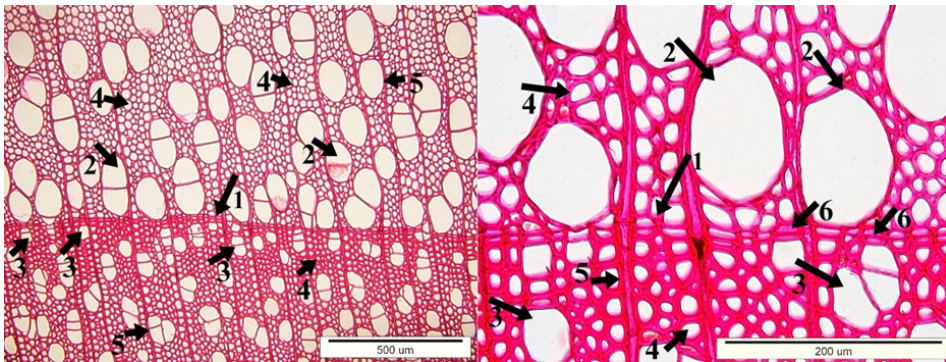
Light Microscopy (LM) was employed to reveal deformations in the anatomical structure of the panels subjected to varying thermal compression conditions. Small wood samples were obtained from the outer region of cross sections of the solid wood panels in order to eliminate possible effects of juvenile wood. The samples with dimensions of 10 mm (R) x 10 mm (T) x 20 mm (L) were cut for the LM evaluations. These samples were kept under vacuum in the presence of alcohol : glycerin : water (1:1:1) at room temperature in order to become softened and were then cut into thin sections (about 20 µm - 30 µm) by using a Leica SM 2010R sliding microtome. The microtome knife was often changed to minimize surface roughness and any possible artificial damage on the surfaces of the samples. The sections were then stained with safranin and fast green according to standard techniques to supply good contrast between cell walls (Wilcox 1964, Ruzin 1999). A microscopic slide of aspen wood from the Xylarium of Faculty of Forestry, Istanbul University (ISTUFw) served as control. The microscopic sections were already stained by safranin.

The microscopic sections were observed under an Olympus BX51 Light Microscope. Images were taken by using analySIS FIVE Software and a DP71 Digital Camera installed and adapted on the microscope.

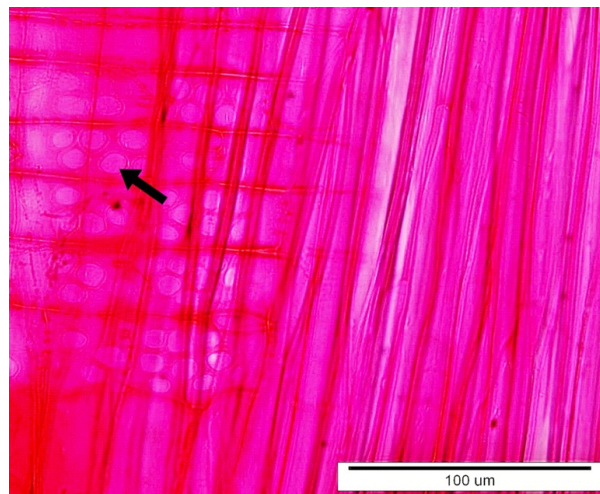
All microscopic studies were only realized visually on the microscopic sections.

## RESULTS AND DISCUSSION

Changes in anatomical structure were investigated to understand the effects of temperature and compression and the results were evaluated separately for each treatment. In this study, repeated micro-damages in the thermally compressed wood structure were taken into account to avoid wrong interpretation. Images of untreated wood samples are shown in Figure 1 and Figure 2 for comparison. Aspen wood is diffuse- to semi-ring-porous, growth ring boundaries are distinct. Vessels are partly solitary, partly in radial multiples of 2 to 4. Axial parenchyma are apotracheal diffuse and in marginal bands. Rays are exclusively uniseriate and homogeneous with procumbent cells. Inter-vessel pits are alternate. Perforation plate is simple. Vessel-ray pits are with much reduced borders to apparently simple: pits rounded or angular, or restricted to marginal rows are extremely large and simple. Fibres are with simple to minutely bordered pits (IAWA Committee 1989, Schoch *et al.* 2015).



**Figure 1.** Light micrographs of untreated wood in different magnifications. Arrows: 1. Growth ring boundary, 2. Earlywood vessels, 3. Latewood vessels, 4. Fibers, 5. Rays, 6. Axial parenchyma cells in marginal band (cross sections).

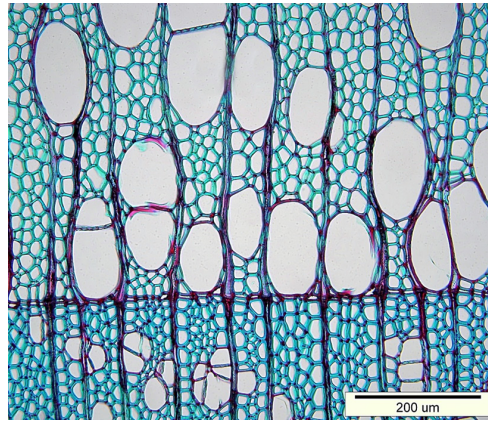


**Figure 2.** Light micrographs of untreated wood (radial section) Vessel-ray pits (arrow).

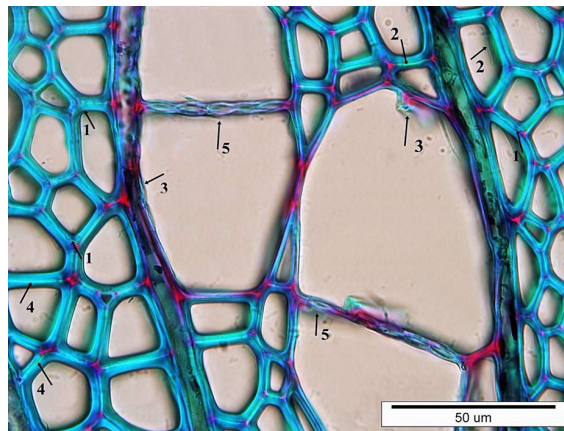
### Process Condition of 150 °C /1 MPa

Minimum structural damage and no visible distortion in the plane of growth rings were observed in cross sections at 150 °C / 1 MPa (Figure 3). The vessels and the fiber cells were also in their natural shapes considerably (Figure 3). Minute cracks and low degree damage were observed particularly in the cell wall of the fibers and vessels located in the beginning and the last part of the growth rings (Figure

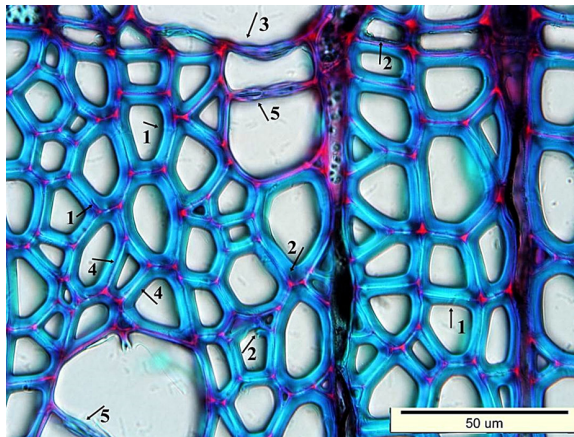
4 and Figure 5). Slight separations between compound middle lamella in fiber cells were noticed. Both damaged and undamaged bordered pits were also found in the cell walls of vessels arranged in the radial rows of 2 to 4 multiples. On the radial section, slight separations between compound middle lamella in ray parenchyma cells were noticed and no visible damages were seen on cross-field pits (Figure 6).



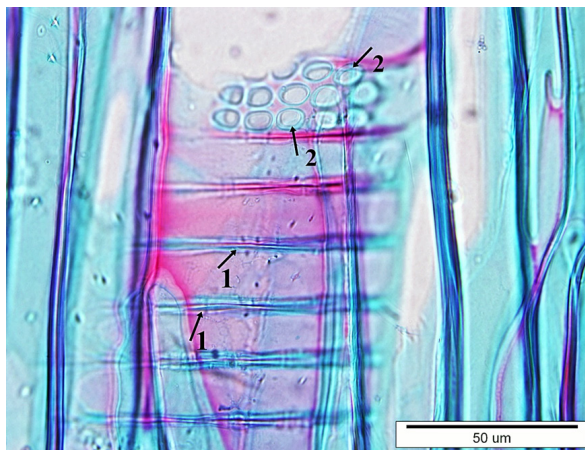
**Figure 3.** No visible distortion in the plane of growth rings at 150 °C / 1 MPa (cross sections).



**Figure 4.** Minute cracks in the fiber cell walls (arrow 1) and low degree cell wall damages in the fibers (arrow 2) and the vessels (arrow 3) in the beginning part of the growth ring, slight separations between compound middle lamella in fiber cells (arrow 4) damaged bordered pits in the vessel cell walls (arrow 5) at 150 °C / 1 MPa (cross section).



**Figure 5.** Minute cracks in the fiber cell walls (arrow 1) and low degree cell wall damages in the fibers (arrow 2) and the vessels (arrow 3) in the last part of the growth ring, slight separations between compound middle lamella in fiber cells (arrow 4) damaged bordered pits in the vessel cell walls (arrow 5) at 150 °C / 1 MPa (cross section).

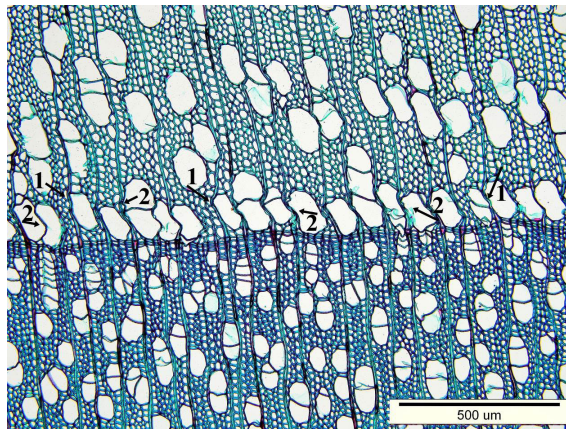


**Figure 6.** Slight separations between compound middle lamella in ray parenchyma cells (arrow 1), no visible damages on cross-field pits (arrow 2) at 150 °C / 1 MPa (radial section).

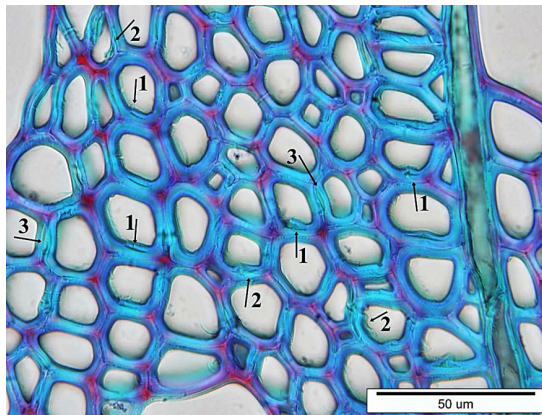
### Process Condition of 170 °C / 2 MPa

Depending on the process condition, much more damages were observed in the wood microstructure at 170 °C / 2 MPa. Buckling on the radial cell walls of the vessels were found clearly in the initial part of the growth rings. These structural alterations on vessels resulted in a slight wavy pattern in the relevant regions and in the rays (Figure 7) On the other hand, no collapse was observed in the cells. It was thought that the buckling in the radial cell walls of vessels was an indication that abrupt fractures would occur. Ruptures were also seen in the vessel cell walls. Distinct cracks and severe degradation were observed in the fiber cell walls located in the beginning and the last part of the growth rings. It was also seen distinct separations between compound middle lamella in the fiber cells (Figure 8 and Figure 9). A great number of ruptured bordered pits were detected in the tangential cell walls of vessels arranged in the radial rows of 2 to 4 multiples. On the radial section, slight separations between compound middle lamella in horizontal ray parenchyma cells were noticed and the less damage was seen on cross-field pits. Besides, the low degree damage in the horizontal cell walls of rays was also

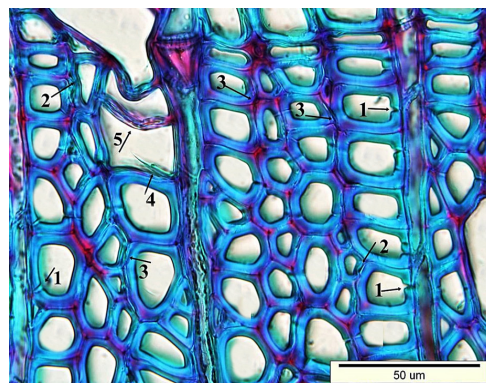
observed in radial section (Figure 10).



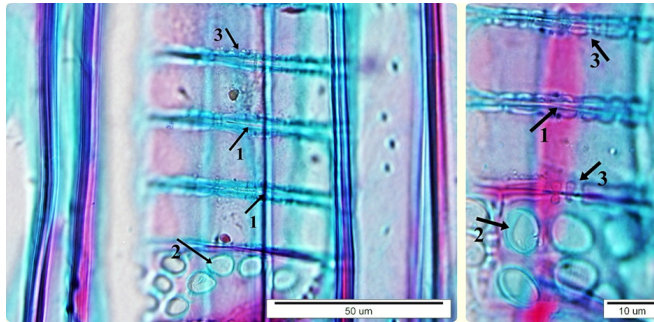
**Figure 7.** Slight wavy pattern at the initial part of earlywood region and in the rays (arrow 1) caused by buckling on the radial cell walls of the vessels (arrow 2) at 170 °C / 2 MPa (cross section).



**Figure 8.** Distinct cracks (arrow 1) and severe damage (arrow 2) in the fiber cell walls, distinct separations between compound middle lamella in fiber cells (arrow 3) in the beginning part of growth ring at 170 °C / 2 MPa (cross section).



**Figure 9.** Distinct cracks (arrow 1) and severe degradations (arrow 2) in the fiber cell walls, distinct separations between compound middle lamella in fiber cells (arrow 3), ruptures in the vessel cell walls (arrow 4), damaged bordered pits in the vessel cell walls (arrow 5) in the last part of growth ring at 170 °C / 2 MPa (cross section).



**Figure 10.** Slight separations between compound middle lamella in horizontal ray parenchyma cells (arrow 1), less damage on cross-field pits (arrow 2) and the low degree defects in the horizontal walls of ray parenchyma cells (arrow 3) at 170 °C / 2 MPa (radial section).

This study showed that much more damages in thermally compressed samples at 170 °C / 2 MPa were seen when compared to compressed samples at 150 °C / 1 MPa and the distribution of defects was not uniform in the growth rings of two treatment groups. All deformation in cell structure was quite distinct at the beginning and the last part of the growth rings and the largest damage occurred in the fibers and vessels. The similar findings were obtained in densified white ash (Tabarsa and Chui 2001) and *Populus ussuriensis* (Ou *et al.* 2014) and paulownia (Dogu *et al.* 2015) woods. On the other hand, some authors reported that morphological changes between the earlywood and the latewood regions of *Populus* spp. were not different under densification process (Tabarsa and Chui 2001, Kutnar *et al.* 2009, Tu *et al.* 2014). The contradiction among the results of studies may be based on the process conditions, usage of different wood species and the wood properties such as different growth ring structure and different amount of latewood and earlywood. Tu *et al.* (2014) mentioned that a high densification ratio will lead to a higher rate of deformation in the wood cells, as well as uniform distribution throughout the growth ring in *Populus tomentosa*. Candan *et al.* (2013b) indicated that the percentage of maximum increase in density was 18%, at 170 °C / 2 MPa. Kutnar and Šernek (2007) indicated that the increase in density was in the range of about 25% to about 500%, though preferably in the range of about 100% to about 200% in the densified wood. In this context, even the maximum density did not reach to value of 25% in the current study. On the other hand, the vessel porosity of wood is diffuse- to semi-ring-porous in the study and the vessels in the earlywood are larger than those in the latewood of the previous and of the same growth ring. Cell wall thickness of fibers is also visually clear different in the same regions of the growth rings. Therefore, it was considered that distribution of stresses was not homogenous throughout the growth rings resulted in differentiation in the severity of micro-defects.

## CONCLUSIONS

Microscopic investigations on thermally compressed aspen wood revealed that the highest damages occurred in fibers and the vessels and changes in microstructure were not uniform in the growth rings for the two treatment groups.

The contradiction among the results of different studies performed on poplar wood indicated that growth ring structure and vessel porosity have a strong effect on wood behavior in various process conditions. In this context, further studies on the microstructure of wood subjected to different modification methods are needed.

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