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(editors)

# **Processing Technologies for the Forest and biobased Products Industries**

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## Wood densification processing for newly engineered materials

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### ABSTRACT

Wood is a renewable, bio-based material with a mixture of different properties and qualities, used in numerous applications. Beside many species with high wood qualities several species suffer due to a number of disadvantages, where low hardness and abrasive resistance are characteristic for low-density species. This paper presents examples of on-going European research projects and industrial processes mostly related to wood densification methods. Wood densification is a classical thermo-hydro-mechanical (THM) wood treatment process, through which density is increased by mechanical compression of wood perpendicular to the grain, by impregnation of cell lumens or cell walls with solutions or melted substances (resins, waxes), or by a combination of both. The purpose is to produce newly designed and engineered materials and products with new property profiles, which would potentially find new markets. In general, the THM processes consist of three stages: plasticization of the wood cells, followed by the actual compression, and finally solidification of the compressed wood in order to prevent elastic spring-back and the moisture-induced set-recovery. The wood densification

*process refers but is not limited to solid wood and might apply to whole wood pieces, or to local areas within given pieces only. Another THM method is the mechanical compression of wood parallel to the grain, which leads to a product with high flexibility. A European wood research network, represented by the authors of this contribution, has extended experience in many wood modification processes, as demonstrated through ongoing researches and case studies in this paper.*

## 1 INTRODUCTION

Regarding the difficulty of the gap between the amount of growing wood resources and the amount of wood usage (Mantau et al. 2010), it is of interest to increase its lifespan, mechanical properties, and the amount of usable wood assortments. Modification can offer more possibilities for both outdoor and indoor applications. Modification methods are heat treatment (including heat treatments under different atmospheres, thermo-hydro-mechanical (THM) treatments such as densification of the surface layer or throughout the entire cross-section, longitudinal compression, wood welding, etc.), chemical modification (acetylation, Belmadur technology with DMDHEU, etc.), impregnation modification (furfurylation, resins, waxes, etc.) and surface modification (e.g. plasma treatment) of wood.

One important mechanical modification process is the densification, used to achieve a permanent deformation of wood cells and thereby an increase in density of a piece of wood or of a part of it – transverse compression or densification – or to increase the flexibility of the wood piece in e.g. bending – longitudinal compression (Sandberg et al. 2013). The main goal of densification is to increase its hardness and surface abrasion resistance, but also to increase its strength. The degree of densification of a workpiece of wood can vary depending on what is desired to be achieved. To increase the density in the entire thickness and thereby improve the overall mechanical properties of the wood material, all the cells throughout the thickness must be compressed and deformed (through-thickness densification). The density of wood can theoretically be increased to a value close to that of the cell wall, about 1500 kg/m<sup>3</sup>, and thereby achieve considerable improvements in mechanical properties. The energy consumption for this densification is very high, especially in the final phase of compression when the denser cells in the annual rings are to be deformed. If the purpose is just to achieve a hard and abrasive-resistant surface, only the wood cells close to the surface need to be compressed and deformed (surface densification). From a structural perspective, surface-densified wood has higher material usage efficiency. For some products, it has better dampening characteristics. The treatment needs to densify the cells close to the surface. This may allow a faster, less energy-consuming, and thereby a less costly treatment process with a positive influence on the retention of the volume and the lightness of the material.

THM processes basically consist of three main phases: (1) softening of the wood material (plasticization), (2) mechanical modification of the cells (densification), and (3) elimination/reduction of the spring-back and set-recovery by different techniques. These phases interact with each other, but are here for simplicity described as separate processes. This paper focuses mostly on densification. Other methods under development such as a modified wood welding technique (Vaziri et al. 2015) or self-bonding compression techniques (Cristescu et al. 2015) can also be used for surface densification, but are not discussed here.

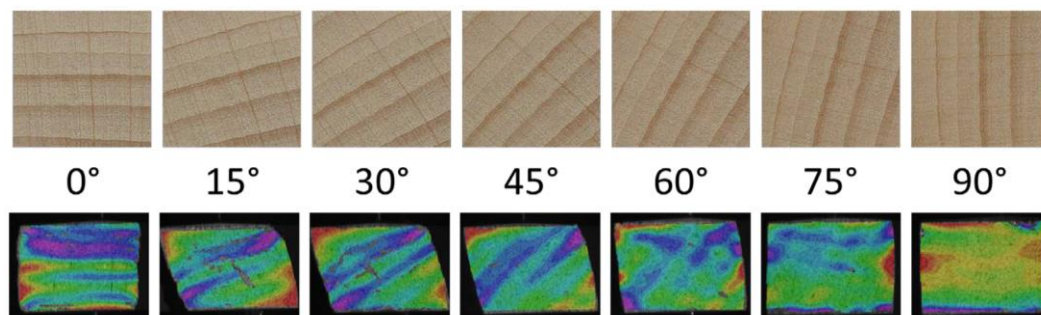
### 1.1 Plasticization

Plasticization treatment is an extremely important part of the process where mechanical wood modification is desired, by which wood can withstand the compressive deformation without fractures. Partial plasticization can be used to precisely determine the location of the densification within a material. Wood can be softened and shaped so that it keeps its new shape after the plasticization is finished. The observed glass transition of wood constituents occurs over a temperature range of 50 °C, and it is dependent on the moisture content (MC) and the heating time (Kúdela et al. 2018). Nilsson et al. (2011) described a densification technique based on compressing Scots pine sawn timber in the radial direction, which shows no or little cell-wall fracture even if the densification is performed at 20 °C at a MC of about 8%, i.e. un-plasticized. The densified wood was used as the surface layer of a multi-layer composite with a light-weight core. A combination of moisture and heat decreases the modulus of elasticity of wood, therefore it is an effective way of softening, but chemical methods can also be used with excellent results. In a former study (Rousek et al. 2015), plasticization by vacuum impregnation (0.2 MPa) with ammonia gas at room temperature was compared to plasticization treatment with saturated steam at atmospheric pressure and a temperature of 100 °C. Ammonia plasticization reduced the compression forces significantly and was sufficient for densification, but was slightly less efficient than saturated steam. The best plasticization effect was reached by combining these two methods.

Microwave (*MW*) heating is a time-reducing method of heating that can in combination with moisture be used for plasticization and has been studied by e.g. Norimoto and Gril (1989) and Dömény et al. (2017). When *MW* radiation is applied to the wood with certain *MC*, the water in the cell cavity absorbs the energy and is vaporized. Plasticization by *MW* heating has several advantages compared to the conventional water vapor method, such as lower energy consumption, rapid heating of wood over the whole volume and application in continuous processes. In contrast, structural changes can occur during *MW* treatment and this may lead to changes in wood strength (Oloyede and Groombridge 2000). The high internal steam pressure can cause the cell to rupture and generate micro cracks in the wood structure.

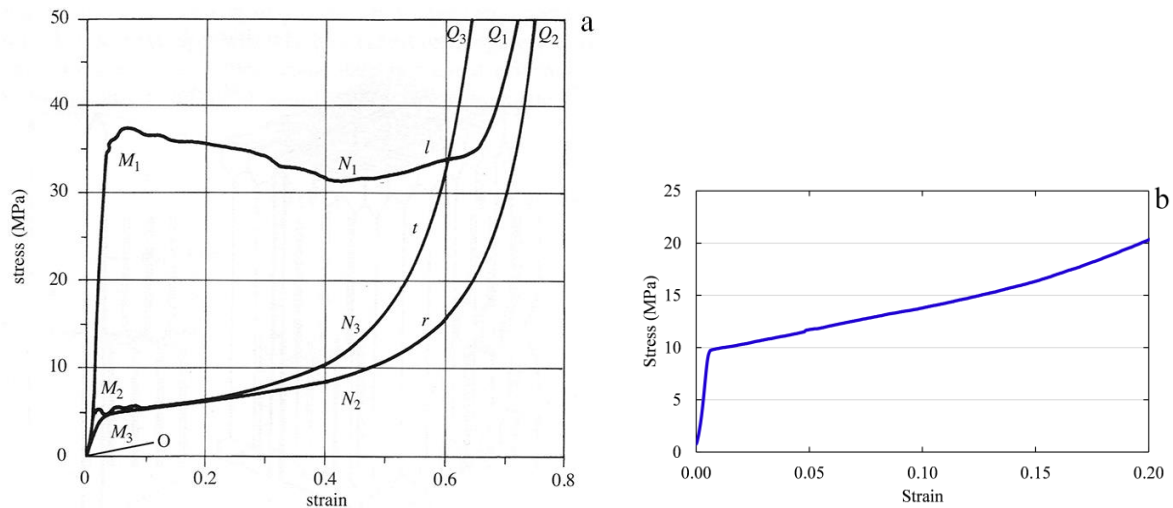
## 1.2 Densification

Densification can be achieved in one or more directions, but the process is mostly performed along one of the orthotropic axes, and the efforts are more successful for diffuse porous hardwoods than for softwoods. Densification can optimally be achieved mainly in the radial direction for softwoods and in the tangential direction for hardwoods with large aggregated rays. If e.g. Scots pine is densified along the tangential direction, the latewood of the annual rings spreads into the earlywood and forms a zigzag pattern and the whole piece of wood may be crushed (Sandberg 1998). Densification in the radial direction, on the other hand, flattens the cells without any noticeable damage at the micro- or macroscopic level. In hardwoods with large aggregated rays, the same type of crushing phenomenon happens with the rays (Rousek et al. 2015). During transverse compressive loading, the typical stress-strain curve of wood has three distinct regions corresponding to three different types of cell deformation: 1) a linear elastic part, 2) a 'collapse' region where the stress is almost constant even at high strain, 3) a sharp increase in stresses due to contact between the inner cell walls. Cellular collapse occurs by elastic buckling, plastic yielding or brittle crushing, depending on the test conditions and on the nature of the cell wall. It is also known that wood responds differently to radial and tangential compression. Rousek (2014) has described how the strain varies within beech under densification according to the annual ring orientation in its cross-section (Figure 1).



**Figure 1.** Densification of beech with different annual ring orientations in the cross section and Digital Image Correlation (DIC) showing the strain at 26% compression (blue colour: high strain; red colour: low strain) (Rousek 2014)

In radial compression, earlywood primarily controls the elastic and plastic parts of the stress-strain response, while the final compression stage is dominated by the elastic deformation of latewood (Figure 2a). In the tangential direction, the final compression stage begins after buckling of the latewood layers. The compressibility of different wood tissues affects the distribution of void areas, and thus also the density distribution and mechanical properties of compressed wood (Kamke and Casey 1988). The location of the buckling of both the cell walls and the annual rings during densification cannot be predicted precisely, as it depends on the morphology of the microstructure and the degree of plasticization.



**Figure 2.** Stress-strain curves of samples of poplar (a) of dimensions 25x25x5 mm<sup>3</sup> subjected to compression in the l, r, t directions under controlled displacement at a rate of 1 mm/min (Navi and Sandberg 2012), and oak (b) subjected to controlled compression in the longitudinal direction (based on Báder and Németh 2018)

In longitudinal (axial) compression, the mechanism of compressive deformation is fundamentally different. The sample illustrates almost 3 segments in the stress-strain curve: a quasi-linear segment followed by the second segment non-linear curve with a negative slope. This segment can be interpreted as being due to localized longitudinal buckling of the cell walls and/or local fracture. The third segment shows an increase in the modulus of the sample. In the case of high density hardwoods the stress-strain curve highly differs (Figure 2b). A more angular stress-strain curve can be seen, compared to the poplar samples: a quasi-linear segment followed by the second segment almost linear curve with a positive slope. The third segment shows a slight increase in the modulus of the sample. The strain is smaller and at the final point, a local large deformation of wood tissue appears due to a local Euler type buckling in the walls of the cells, which leads to the formation of a shear band on the wood macro-level (Sandberg et al. 2013).

### 1.3 Spring-Back and Set Recovery

The major obstacle preventing the widespread commercialization of surface-densified wood products is perhaps the elastic spring-back and in particular the set-recovery of the compressed wood cells. The elastic spring-back – which occurs immediately when the compression force ceases – is greatly reduced by introducing a cooling stage to reach a temperature of the densified wood below 80 °C before the pressure is ceased at the end of the densification process (Neyses 2016). The moisture-induced set-recovery can be eliminated by chemical modification, by impregnation with resin, or by a thermo-hydro-mechanical post-treatment. Five general approaches to achieve a long-term fixation of the densified wood cells have been identified: (1) mechanical fixation by gluing or impregnation with adhesives, such as epoxy resin, or nailing, screwing, etc., (2) the formation of cross-links between molecules of the wood matrix by chemical modification: deactivation of the OH-sites, (3) formaldehydation (fixing of H<sub>2</sub>CO between two hydroxyls to obtain a strong chemical bond), (4) relaxation of internal stresses within the wood matrix during densification, or (5) reducing the accessibility of the cell wall to water.

The main objective of the present paper is to give a concise state-of-the-art presentation of the most recent developments in the field of mechanical wood modification. Therefore, the following paragraphs will introduce the actually used or developing mechanical wood modification methods.

## 2 CASE STUDIES FOR MECHANICAL WOOD MODIFICATION PROCESSES

### 2.1 Lignamon

The production process of Lignamon<sup>®</sup> combines vacuum-pressure impregnation of beech with ammonia vapour at a temperature of 90°C and densification. The process continues with drying, stabilization (180 °C) and acclimatization (Stojčev 1979). The treatment of beech by gasification with ammonia and simultaneous steaming plasticizes the wood and allows the densification of the material up to a density of about 1100 kg/m<sup>3</sup> with an appropriate increase in the mechanical properties, as shown in Figure 3 (Pařil et al. 2014).

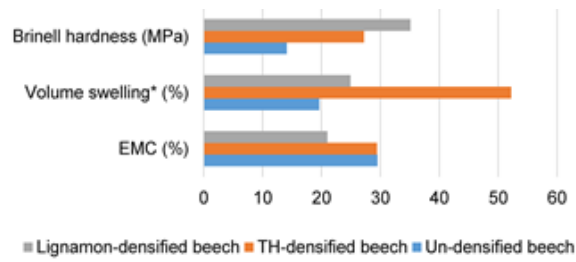


Figure 3. The Lignamon process compared to control and steamed-densified beech (based on Pařil et al. 2014)

The heating increases the dimensional stability of the wood under moist conditions and can improve the durability compared with that of untreated beech (durability class DC5) up to durability class DC1. Lignamon was presented as a material with considerably improved properties, but despite this the factory closed and Lignamon is no longer being produced.

### 2.2 Viscoelastic Thermal Compression (VTC)

Densification has the potential to improve the properties of widely available low-density wood species, opening up new fields of application, and fostering the use of wood products in general. Figure 4 shows an example of how low-density poplar from the Central Czech Republic (clone Max 4) was used to improve the strength properties by densification. The density was increased up to three times compared to undensified poplar with the result that the modulus of elasticity (MOE) also increased considerably (Hornicek et al. 2015; Rademacher et al. 2014).

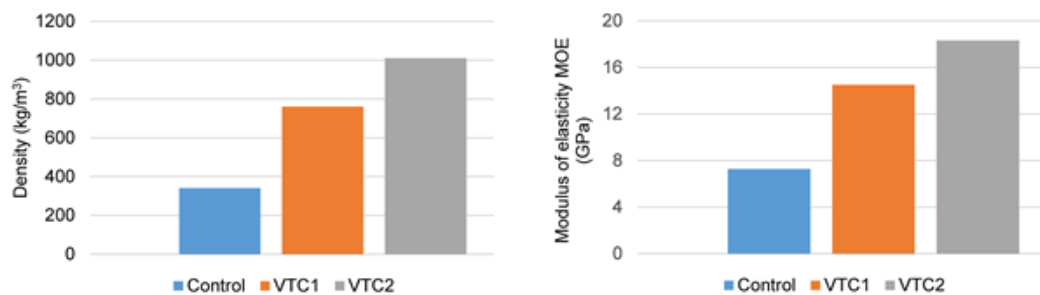


Figure 4. Densification of light-weight poplar wood by the VTC-process, i.e. heating and steaming in a closed-system at 170-200°C for about 7 min and compression. The compression ratio was 1:2.3 for control to VTC1 samples and 1:2.9 for the control to VTC2 samples (based on Rademacher et al. 2014)

### 2.3 Continuous Compression Process

Densification throughout the cross section may be a drawback in situations where, for example, it is desirable to maintain the low bulk density of wood, e.g. flooring applications. In such situations, an alternative approach is to densify only the first few millimetres beneath the surface of the wood, which places a great demand on the plasticization and compression process stages to achieve the desired density profile within the wood. In a former study, a high-speed continuous surface densification process was introduced by Neyses et al. 2015, where the surface of solid Scots pine boards could be densified at speeds of up to 80 m/min by a roller pressing technique (Figure 5). The present focus is to make the process more industrially adapted by integrating the roller pressing technique with various pre- and post-treatment methods to reduce negative effects such as set-recovery, colouring, or embossment of the surface. A continuous process with steel belts instead of rollers has been studied by Sadatnezhad et al. (2017).

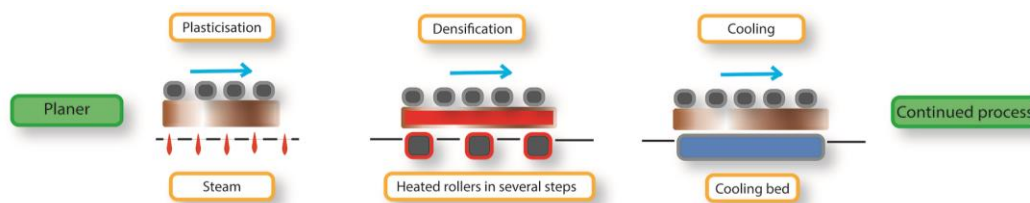
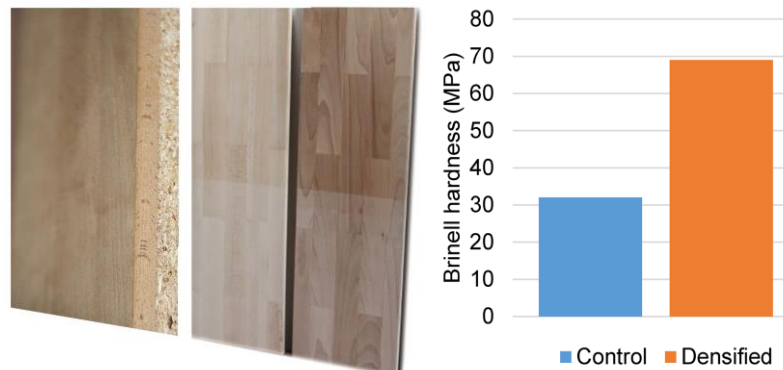


Figure 5. Schematic illustration of a continuous surface densification process (from Sandberg et al. 2017)



## 2.4 Densified Beech Wood of Multi-layer Consistence

Dömény et al. (2017) used microwaves for plasticization before and stabilization after the densification of beech. The densified beech was used as surface layer for a two-layered flooring panel where the other layer was particleboard (Figure 6). The Brinell hardness value of the densified beech is more than double that of the undensified beech.



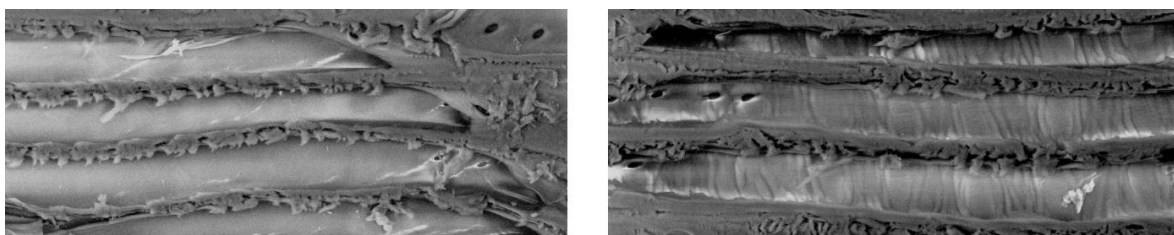
**Figure 6.** A layered flooring panel (left) with densified beech as top surface (on the right of the center image), an undensified beech (on the left of the center image) and Brinell hardness values (right) (based on Dömény et al. 2014)

## 2.5 Densification of lignin/hemicellulose content reduced wood

In this new process described by Song et al. (2018), lignin and hemicellulose are removed from the natural wood using a boiling process in an aqueous mixture of NaOH and Na<sub>2</sub>SO<sub>3</sub>. Afterwards, due to the densification at 100 °C temperature, the cell walls collapse. The result is a specific strength of this highly modified wood, higher than that of most structural metals and alloys, while the modified wood remains light-weight compared to these materials.

## 2.6 Longitudinal Compression (Pleating)

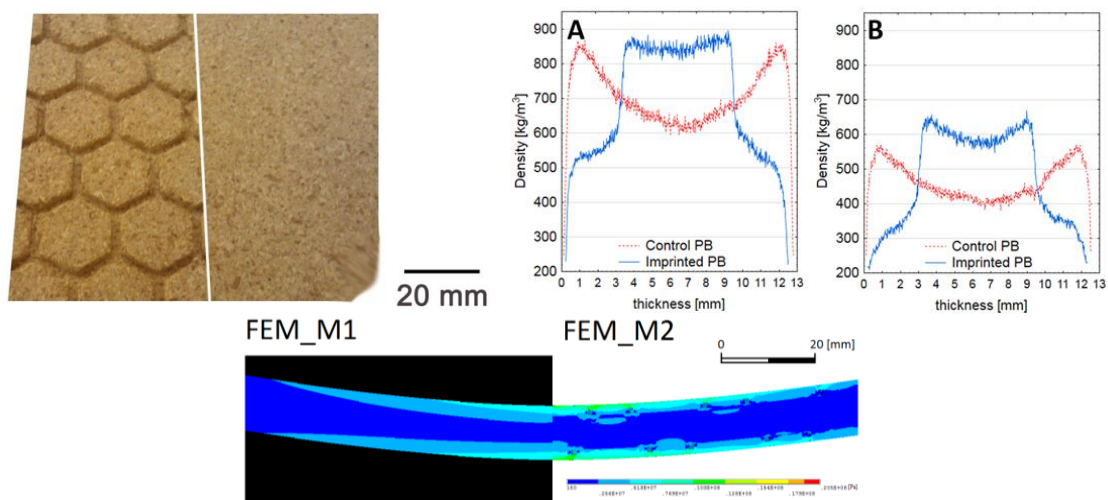
While densification increases the density and other properties of wood by mechanical compression perpendicular to the grain, compression along the grain makes wood easier to bend to a smaller radius by the distortion of the fibres. This method requires a high-quality hardwood raw material with at least middle density. After plasticization, the compression ratio is 15-25% of the original length. After compression, the degree of compression should be maintained for a while for relaxation of internal stress to further increase the effects of the compression. 1 minute relaxation time results in an increase in maximum deflection during 4 point bending tests to 353%, and in a decrease to 37% in *MoE* and to 44% in bending stress at 5 mm crosshead displacement, compared to the control samples (Báder and Németh 2018). During the modification process the normally smooth cell walls deform (crinkle, Figure 7). Therefore this method may be practically called "pleating". The indentation modulus of the secondary cell wall layer S2 significantly decreases as a result of pleating, whereas the hardness of the S2 cell wall is only slightly affected. AFM images show microfibril disorientation in the S2 cell wall layer due to the treatment. The main advantage of the pleated wood is that it can be stored for a long time (the *MC* must be kept) and bent when it is needed in a cold state. While the moisture content is high, it is more bendable than in a dry condition, but always easier than uncompressed wood. This material is primarily used in interior design and for furniture components.



**Figure 7.** SEM images on radial sections of oak: before (left) and after (right) longitudinal compression (magnification 1000x) (images from Báder M.)

## 2.7 Surface Pattern Imprinted Wood-based Panel with Improved Internal Bonding

This research is on a new development towards three-dimensional surfaces for wood-based panels, i.e. particleboards (*PB*), by imprinting a self-designed stainless steel grid on both sides of *PB*. Following a factorial experimental design, 12 mm thick *PB* was produced in the laboratory and cut samples were tested according to standards. As results, the 3D-pattern imprintment resulted in a much higher internal bonding (*IB*): for the 500 kg/m<sup>3</sup> *PB*, the *IB* increase was 35%, whereas for the 700 kg/m<sup>3</sup>, the increase was even 67%. In the vertical density profile a roof-like shape was observed, with a high density level in the core layer (Figure 8). The modulus of rupture and stiffness turned out to be much lower than those of the control sample. A successful compensation for the reduced bending performance is demonstrated through a two-side veneer sheet addition. Finite element modeling (*FEM*) is also shown to be instrumental for the property optimization of the 3D-pattern imprinted *PB*. Since *IB* has significantly increased, the overall *PB* density could be reduced for the same product performance. The new 3D-pattern imprinted *PB* also constitutes a decorative design feature. Since the imprintment process is easy to execute, this new *PB* product option might be easily adoptable by the industry (Klímek et al. 2017).



**Figure 8.** Surface of imprinted and control particleboards (top left), density profile of imprinted and control particleboards (top right) and calculated stress distribution in control (FEM\_M1) and imprinted particleboard with veneered surface (FEM\_M2) (Klímek et al. 2017)

## 3 CONCLUSIONS

Research on wood densification is intensive in several research groups in Europe trying to understand the plasticization and densification of wood, and to develop processes and products that are environment-friendly. The focus has changed from the densification of the entire thickness towards the densification of specific regions, where the increase in density is needed in a specific product, e.g. flooring. The main challenges for the future are finding a fast and environment-friendly method for the elimination of the set-recovery and scaling up to profitable industrial applications. These are also true for other mechanical modification processes e.g. surface pattern imprintment and longitudinal compression (pleating).

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