WOOD RESEARCH
58 (1): 2013
101-112

STRUCTURE EVALUATION OF COMPRESSING OF SPRUCE AND BEECH PLYWOODS PART 1: MICROSCOPIC STRUCTURE

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(Received July 2011)

ABSTRACT

The work deals with research and description of five-layer Norway spruce (*Picea abies* [L.] Karst.) and European beech (*Fagus sylvatica* L.) plywoods. It seeks to make an integrated view of structure of plywoods and focuses on glued joint and deformation of wood elements caused by the manufacturing technology. The objective of the work was to establish and verify a methodology of taking of microscopy specimens (slices) of hard plied materials and following taking of micro images. Another objective was macroscopic and microscopic research of plywoods using methods of image analysis and comparing with the results of density profile measuring. The results include various ways of adhesive distribution through elements of spruce and beech wood. At the same time, influence of technology on resulting shape of glued joint is evaluated.

KEYWORDS: Plywood, European beech, microscopy, anatomical structure, glued joint, density profile, deformation of wood elements.

INTRODUCTION

Plywood is defined as board with layers glued together, whereas orientation of fibres of neighbouring layers is perpendicular to each other. In general, the outside and inside layers on both sides are placed symmetrically towards the middle (or centre) layer.

Ever wider use of layered composite materials for construction purposes have led to research of their properties which change depending on their structure and the degree of thickening. Better properties of the final product can be achieved by pressing and compacting the composite. Bekhta et al. (2012) dealt with the effect of rewriting factors on the properties of veneers. In their work they demonstrated dependence of compression ratio on the technological parameters of veneer pre-pressing (e.g. pressure, temperature, and time). They proved that the pre-pressing time had the least effect of the monitored parameters on compression ratio. The largest growth in compression ratio in dependence on the pre-pressing time was observed in the first 30 seconds. A far more significant effect was proven in the pre-pressing pressure, but temperature appeared to be the main factor affecting compression ratio. They observed that compression ratio increased with increasing temperature and pressure. They also state that the results in their publication were rather different from their previous studies (Bekhta et al. 2009, Bekhta et Marutzky 2007). Bekhta et al. (2012) further proved that the surface roughness decreased with increasing pressure and temperature of veneer pre-pressing. They confirmed that at the amount of coating 90 $g.m^{-2}$ the shear strength of the final composite (plywood) increased with increasing temperature of veneer pre-pressing. Also, they confirmed the hypothesis that by the pre-pressing of veneers the adhesive penetration depth decreased.

It is known that the amount of compression ratio has an effect on the mechanical properties of veneers and composites made of them, but a clear correlation has not been found yet between compression ratio and the tensile strength of veneers, across the grain (Bekhta et al., 2012). The mechanical and elastic properties of wood-based laminated composite materials depend to large extent on the composition and arrangement of sets, the number of layers, their thickness, and the quality of veneers used. The current research deals with the possibility of using modified veneers in plywood production with the aim to increase dimensional stability. Dieste et al. (2008) confirmed that by modifying veneers with 1.3-dimethylol-4.5-dihydroxyethyleneurea (DMDHEU), increased endurance and dimensional stability were achieved, as well as maintaining or a favourable effect on the modulus of elasticity (MOE), but the maximum bending force value (WMLB) decreased.

Strength of glued connection depends on density of used veneers, and with increasing density of the same kind of wood strength properties of glued connection increase as well (Eisner et al. 1966). Research of pine boards (Hse 1971) showed that quality of gluing depends on whether it is a connection of summer wood – summer wood (L-L), spring wood – spring wood (J-J) or spring wood – summer wood (J-L). The highest strength was achieved with the J-J version.

When the plied material is compressed, the adhesive is distributed into the glued material. Suchsland (1987) found out that depth of penetration of the adhesive into the wood depends on length of fibres and angle they form with the surface. Depth of penetration of the glue can be calculated theoretically. In spruce plywood, penetration increases with increasing spread of glue and increasing angle of fibres.

Šteller and Pleško (1972) determined the effect of quality of peeled veneer on shear strength of beech plywood. The quality criteria of veneer – tension strength across fibres, average depth of cracks and frequency of cracks. In high-quality veneers, thinner spreads of adhesive were achieved with higher strength of gluing. Pizzi and Mittal (2003) state that the smaller is the contact angle or surface roughness of veneers, the higher is the strength of the glued joint and that the strength of the glued joint decreases with increasing thickness of the glued joint. Phenol-formaldehyde adhesives are extremely sensitive to the content of moisture in veneer.

It has been proven before that density has an effect on most of the physical and mechanical properties of composite materials, particularly on bending properties (Maloney 1993, Suchsland and Woodson 1986). Chen et al. (2010) have recently dealt with an analysis of structure and density distribution. They examined structure and density distribution on four common types of wood-based materials (plywood, OSB, MDF, and PB) using a digital X-ray analysis. The same system of X-ray scanning is used by Wang et al. (2007) in their research dealing with density distribution.

The objectives of this research were: a) to establish and verify a methodology of taking of microscopy specimens (slices) of hard plied materials and following taking of micro images;

b) macroscopic and microscopic research of plywoods using methods of image analysis and comparing with the results of density profile measuring.

MATERIAL AND METHODS

Manufacturing of plywoods intended for testing

The work used standardly produced Norway spruce (*Picea abies* L. Karst.) and European beech (*Fagus sylvatica* L.) veneers 2.6 and 1.5 mm thick. They were produced via peeling, dried to 7 % of moisture, cut to the size of 550 x 550 mm and air-conditioned for 14 days. Thus prepared veneer sheets were classified by quality. For gluing of plywoods, phenol-formaldehyde (PF) and urea-formaldehyde (UF) adhesive were used. In addition, the urea-formaldehyde adhesive was dyed with red colour filler which enables observing it in microscopic and macroscopic slices. Spreading of adhesive was followed by compressing. The total of 21 boards was compressed. Compressing was performed in a testing press at a compressing temperature of 126°C. Compressing time was set depending on the total thickness of the board being compressed and the kind of wood – 10 min for spruce and 8 min for beech. Compressing pressure was set at 1.6 and 3.3 $N.mm^{-2}$ and 1.9 and 4.1 $N.mm^{-2}$ for spruce and beech veneers, respectively.

Preparation and selection of samples

Samples were selected, cut and inspected according to the standard "ČSN EN 326-1 1995 Sampling, Cutting of Testing Bodies and Interpretation of Test Results".

Side surfaces of the samples were worked using a special shank cutter with replaceable cutting tips. It ensured high precision and quality of working of surface while the wood elements were not damaged, especially in the direction of cross section. Further grinding only worsened quality of surfaces. Thus worked blocks with a size of 50 x 50 mm x material thickness enabled us to observe surface of cross section of samples without presence of softeners under a binocular magnifying glass and enabled us to make macro images of the surface.

Measuring of density profile

Density profile was measured with 96 samples of 24 sample plywoods. Precise sizes of samples (width, length and thickness) were found using a digital slide gauge with 0.01 mm precision. Samples were then weighed on laboratory scales with a precision of 0.001 g and moisture of them was measured. Thus prepared samples were put in the instrument X-RAY DENSE-LAB and density profile in the required direction was measured. Density profile was measured in steps by 0.01 mm.

Methodology of acquiring of microscopic slices

Acquiring of microscopic slices of layered wood-based materials on is especially difficult and it ranges among special method of wood-microtechnique (Wolf 1954). In the optimum case, material should be conveniently processed as sections of mineralogical specimens. However, good results were also achieved using the following methodology.

Softening – In order to obtain a true image of all structural changes manifested by the sample, it was necessary to ensure that it will not get in contact with water or to minimize contact with water to a maximum possible degree. Ideally, the particular microscopic slices should be worked in dry conditions. However, thus obtained samples are quite thick and they break and crumble. In our case, a modified procedure of softening with a mixture of glycerol-ethanol (glycerol and 30 %

ethanol in the ratio of 1:1) turned out to be the most appropriate one. The advantage is that blocks can be left in the mixture for an unlimitedly long time (the samples were fully applicable) and the rate of swelling of wood elements is low. In order to detect changes that could occur during softening of material, macroscopic photographs of intact material were taken before softening.

Fixing and cutting – the particular microscopic slices were made in microtoms (R. Jung, Heidelberg) modified for cutting of hard massive wood. Cutting of samples used successfully both a solid microtomy knife and razor knives fitted in a special clamping head that can be easily replaced when they get blunt. Making a good slice sometimes posed a big problem. Slices twisted easily and disintegrated when being flattened back. If this was the case, the cutting surface of the objects was spread with Schweitzer medium (8 g of chloral hydrate, 20 g of gum Arabic, 7 ml of glycerol, 65 ml of distilled water). Schweitzer medium has brightening effect (chloral hydrate) and softening effect (glycerol). Transparent flexible film that is formed holds the slice together (Sárkány and Stieber 1952, Němec et al. 1962).

Making of temporary specimens – microscopic slices were placed, using a thin hair-pencil on a microscope slide in a drop of glycerol and covered with a cover glass. The margin of the microscope slide with specimen was identified with a code of the respective sample using a permanent marker and examined under microscope fitted with a digital camera.

Methodology of taking of microscopic pictures

Selected parts of microscopic specimens were photographed with a digital camera (Olympus E330) fixed onto a microscope (Olympus BX37) in a mode of image analysis (QuickPHOTO MICRO 2.3). A calibration constant was inserted in the scanned image; it enabled measuring of lengths of the chosen direction in true distances.

RESULTS AND DISCUSSION

Figs (Fig. 1 and Fig. 2) illustrate a macroscopic image of the worked side surface of a plywood, provided with a scale in the left bottom corner and with a unique code identification of the examined sample in the right top corner. The density profile measured in the device X - RAY DENSE - LAB and a microscopic image of the microscopic specimen taken of the respective sample in the respective point were also inserted into the figures and so were microscopic images of selected parts of samples taken in different magnifications, so that the procedure of investigation of changes in anatomical structure and glued joints of the tested samples is clear. The figures enable watching of a glued joint (distribution of adhesive), macroscopic and microscopic wood composition, quality of peeling, scope of deformation and rate of compressing of wood elements caused by the effect of manufacturing technology of plywood.

Description of distribution of adhesive through material depending on the microscopic structure of wood

Penetration of adhesive to material occurs for several reasons. Firstly, it is due to microscopic structure of wood given by the kind of the wood plant. Adhesive rises through elements in both longitudinal and transversal section of wood. In the longitudinal direction, adhesive is distributed through vessels (tracheae), tracheids and libriform fibres. In the transversal direction, it is distributed through vascular rays (parenchyma cells), and among individual elements it is distributed through pits (single thinning) and bordered pits (double thinning). High pressure causes disintegration (total rupture in some points) of bordered pits which increases penetration of adhesive through tracheids. Adhesive also penetrates through resin ducts. Penetration depth is affected also by other factors such as adhesive viscosity and type, coating amount, wood species, the length of fibres and the angle that they form with the glued joint (Suchsland, 1957), and surface roughness. Micro images (Figs. 3a,b,c) of spruce plywood illustrate both longitudinal distribution of adhesive through tracheids and transversal distribution through vascular rays. Singh et al. (2008) came to very similar conclusions in their work on the research of pine plywood.

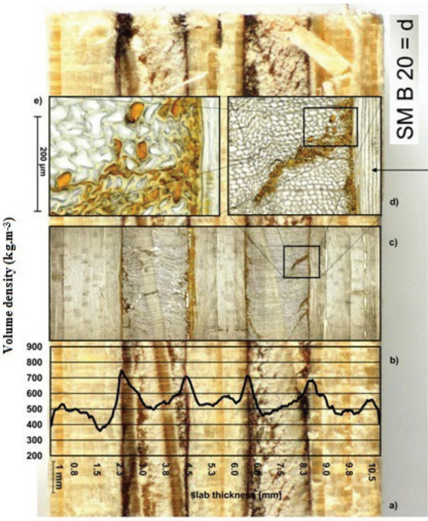


Fig. 1: Macro image inset with a micro image and a graph of the course of density profile of five-layer spruce plied board (veneer quality B) produced at compressing pressure 1.6 N.mm⁻².
a) macro image, b) density profile, c) micro image at 40 fold magnification, d) and e) micro images of selected parts of the cut at various magnifications. The image shows macro and micro structure of the board being examined. In particular, the board shows apparent distribution of adhesive, depth of penetration of adhesive and the rate of deformation of wood elements.

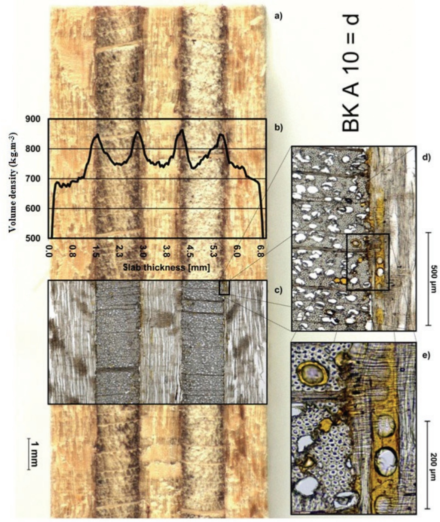


Fig. 2: Macro image inset with a micro image and a graph of the course of density profile of five-layer spruce plied board (veneer quality A) produced at compressing pressure 1.9 N.mm⁻².
a) macro image, b) density profile, c) micro image at 40 fold magnification, d) and e) micro images of selected parts of the cut at various magnifications.

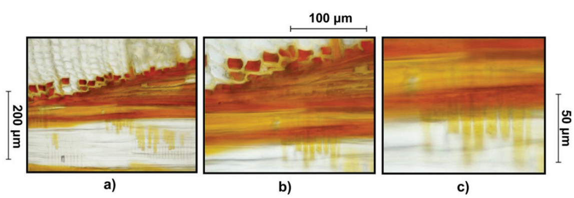
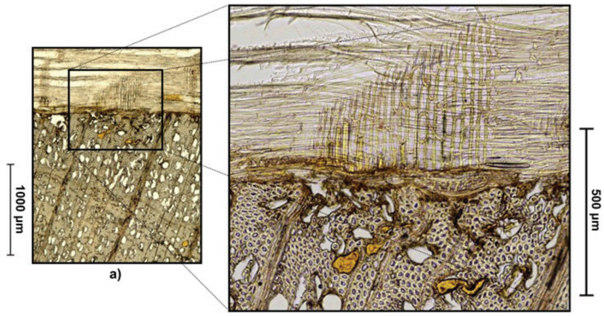


Fig. 3: Micro images of connection of two layers of a spruce plied board products at compressing pressure 1.3 N.mm⁻², a), b) and c) different magnifications of the same part of the sample. The macro images show distinct longitudinal distribution of adhesive through tracheids and transversal distribution of adhesive through pith rays.



a) b)

Fig. 4: Micro image of connection of the second and third layer of a beech plied board produced at compressing pressure 1.9 N.mm^{-2}, a) hundred-fold magnification, b) two-hundred-fold magnification. The image shows apparent shape of the glued joint and deformation of wood elements. It also illustrates penetration of adhesive through parenchyma cells of a pith ray and compression in the point of the pith ray.

Micro images of beech plywood (Figs. 4a, b) show both longitudinal distribution of adhesive mostly through tracheae (and slightly through libriform fibres) and transversal distribution through vascular rays.

Influence of technology of plywood production on adhesive distribution (forming of glued joint)

The rate of adhesive penetration is largely influenced by technology. In particular, it is quality of peeling (quality of surface and fissures, particularly in the case of spruce and pine veneers), roughness of surface, amount of adhesive spread, compressing temperature and high compressing pressure. Even the pressure of 1.6 N.mm^{-2} causes local compression in spruce veneers, leading at points to deformation of cell walls, in particular in spring wood, in which the cells have larger lumen (tracheids) and thinner (thin-wall) cell walls. Thus deformed part soaks with adhesive and changes significantly its mechanical and physical properties. (Fig. 5 b). The penetration of adhesive into cell walls takes place in the place of their disturbance – both in the place of disturbance by machining and in the place of the disturbance of cell walls by distortion (collapse) of cells. The amount of adhesive penetration into the material may be affected also before the surface treatment, for example by pre-pressing of veneers, as stated by Bekhta et al. (2012). They proved that the surface roughness of veneers decreased by pre-pressing of veneers and the pores partly closed. The consequence is a smaller penetration depth of the adhesive mixture and the possibility to apply a smaller coat of adhesive.

Quality of peeling (forming of fissures) influences perpendicular or sideward (Fig. 5a) penetration of adhesive against the plane of the glued joint. Side ward penetration is largely caused by deformation (shear-off) of cell walls as a consequence of compressing and adhesive penetrating to them. The normal penetration of adhesive is mostly caused by cracks in vascular rays caused by the production of rotary-cut veneers. Also Singh et al. (2008) in their work point out to the normal penetration of adhesive through vascular rays.

In beech plywood, wood elements are also compressed but due to its anatomical structure compressing is even and cell wall deform only rarely, in vessels. Libriform fibres have small lumen of wall and thick cell walls, therefore they deform only partially. A smaller rupture of wood structure and cell walls in beech causes a far smaller depth of adhesive penetration.

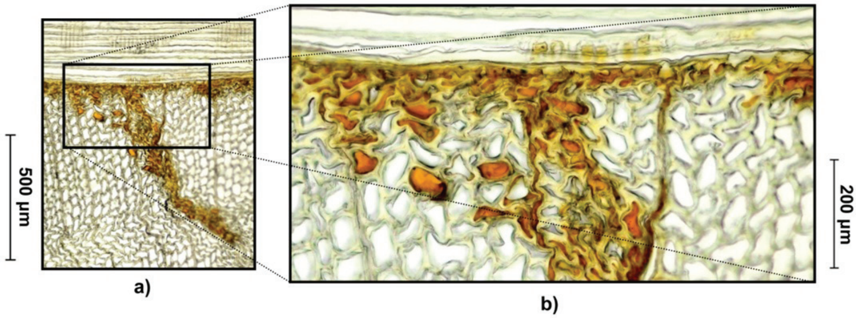


Fig. 5: Micro image of connection of the second and third layer of a spruce plied board produced at compressing pressure 1.6 N.mm^{-2}, a) hundred-fold magnification; the image shows deep sideward penetration of adhesive caused by deformation of cell walls in a spring annual ring due to high compressing pressure, b) two-hundred-fold magnification, the image shows the glued joint and deformation of cell walls.

The adhesive can only penetrate into larger depths through the lumens of large vessels (Fig. 6a). Also vascular rays prevent significant deformations as they function as transverse reinforcing beams and bear most of load. At a compressing pressure above 1.9 N.mm^{-2}, they are twisted (bent at ends, see Fig. 6b) or sigmoid-curved. Curving of vascular rays occurs only at higher compressing pressures.

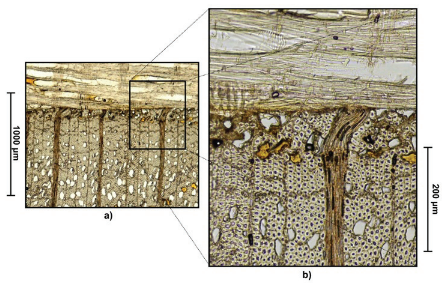


Fig. 6: Micro image of connection of the second and third layer of a beech plied board produced at compressing pressure 4.1 N.mm^{-2}, a) hundred-fold magnification, b) two-hundred-fold magnification.

General description of glued joint and determination of thickness of glued joint and points with significantly higher density

The term glued joint means a sum of the depth of adhesive penetration to both interconnected materials. Penetration depth can be measured using an optical analysis of an image of calibrated macro and micro photos or using an analysis of density profile. The optical analysis of image is more precise for measuring of adhesive penetration in selected parts but we recommend using the analysis of density profile to make a general picture. Ideally, both options should be combined.

Description of glued joint depending on wood plant and compressing pressure

In beech plywoods produced at compressing pressure 1.9 N.mm^{-2} glued joint is straight and adhesive layer is even. An average value of its thickness, determined by an analysis of density profile, was approximately 0.26 mm. In beech plywoods produced at compressing pressure

4.1 N.mm^{-2}, glued joint was slightly corrugated yet still even. An average value of its thickness was approximately 0.34 mm.

A glued joint in spruce plywoods produced at compressing pressure 1.6 N.mm^{-2} is corrugated and forms perpendicular or sideward penetrations reaching up to one third of the layer thickness. An average valued of a glued joint thickness determined by an analysis of density profile was approximately 0.44 mm. A glued joint in spruce plywoods produced at compressing pressure 3.3 N.mm^{-2} was corrugated too and formed perpendicular or sideward penetrations even passing through the whole layer. An average value of its thickness was approximately 0.63 mm.

To examine the glued joint (particularly the penetration of adhesive into the cell wall) we recommend using also the fluorescence method confocal laser scanning microscopy (CLSM) as stated by Singh and Dawson (2004) and Singh et al. (2008). Using this method, the adhesive can be clearly distinguished from the glued wood, as well as the extent of saturation of cell walls with the adhesive. The difference of outputs can be compared in figures (Fig. 7 and Fig. 8). An indisputable advantage of the CLSM method is the possibility of clear recognition of the adhesive from the glued material even if the adhesive used is transparent and is not visible in the glued joint. The adhesive or sample had to be dyed up to that point.

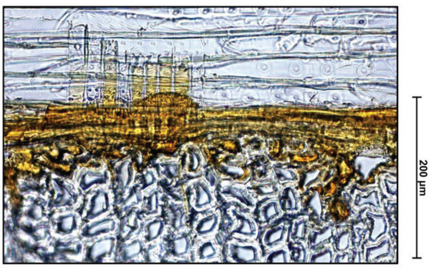


Fig. 7: Micro image of connection of the second and third layer of a spruce plied board produced at compressing pressure 1.6 N.mm^{-2}. The glued joint is clearly visible in the figure – PF adhesive in yellow to brown colour.

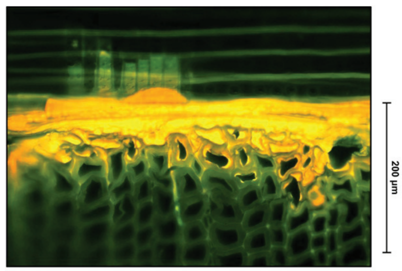


Fig. 8: Composite microscopic image of jointing the second and third layers of a spruce plywood sheet made at the pressing pressure of 1.6 N.mm^{-2} acquired using the CLSM method at parameters EX 450–490, 510–560, and 340–380 nm; DM 505, 575, and 400 nm; BA 520, 590, and 435–485 nm. The yellow to orange colour represents the adhesive and its penetration into the glued material. The green colour represents the glued material (wood) without the presence of adhesive.

CONCLUSIONS

Taking of microscopic samples of plied glued materials based on wood is relatively difficult. The methodology of sampling of individual microscopic slices requires maintaining of precise procedures and skills. However, telling properties of microscopic slices are high and along with density profile and macro images they provide coherent image of the material structure. The methodology of taking of microscopic slices is one of the options of how to make a coherent image of the internal structure of materials.

Using the methodology of taking of microscopic slices and evaluating of them using a visual analysis the material structure – both systematic of glued joint formation and quality of individual material layers – can be observed with high precision. A literary review and the results obtained imply that the resulting quality of plied materials is affected particularly by the raw material (a kind of woody plant) and quality of peeling (a system of steaming and peeling technology), pre-pressing, compressing pressure and compressing diagram, compressing temperature and the type and properties of the adhesive used. The results imply a significant effect of technology on the resulting quality of the composite.

Phenol-formaldehyde adhesive (PF) proved to be significantly better for production of microscopic slices, measuring and observing of their glued joint. Microscopic slices of materials glued with PF adhesive were compact and after softening they did not disintegrate compared to samples glued with urea-formaldehyde (UF) adhesive. PF adhesive was clearly visible in joint and unambiguously distinguishable from material without adhesive. Depth of penetration of adhesive was also clearer.

Penetration of adhesive to material occurs for several reasons. Firstly, it is due to microscopic structure of wood given by the kind of the wood plant. Adhesive rises through elements in both longitudinal and transversal section of wood. In the longitudinal direction, adhesive is distributed through vessels (tracheae), tracheids and libriform (woody) fibres. In the transversal direction, it is distributed through vascular rays (parenchyma cells), and among individual elements it is distributed through dots (single thinning) and pit pairs (double thinning). High pressure causes disintegration (total rupture in some points) of pit pairs which increases penetration of adhesive through tracheids. Adhesive also penetrates through resin ducts. However, this fact is insignificant. Depth of penetration is also influenced by other factors, such as viscosity and type of adhesive, depth of spread and kind of wood plant. The rate of adhesive penetration is largely influenced by technology. In particular, it is quality of peeling (quality of surface and fissures, particularly in the case of spruce and pine veneers), roughness of surface, amount of adhesive spread, compressing temperature and high compressing pressure. Even the pressure of 1.6 $N.mm^{-2}$ causes local compression in spruce veneers, leading at points to deformation of cell walls. Thus deformed part soaks with adhesive and changes significantly its mechanical and physical properties. In beech plywood, wood elements are also compressed but due to its anatomical structure compressing is even and cell wall deform only rarely, in vessels. Libriform fibres have small lumen of wall and thick cell walls, therefore they deform only partially. Also vascular rays prevent significant deformations as they function as transverse reinforcing beams and bear most of load.

The survey of plied materials structure is the first step of assessment of technology (steps of wood compressing and distribution of adhesive into individual layers) influence on physical and mechanical properties of composites that form. This work will followed by another article the content of which will be testing in terms of characteristics.

ACKNOWLEDGMENT

This work was elaborated with the support of MŠMT ČR (Ministry of Education Youth and Sports) within the project Forest and Wood (Les a dřevo) MSM6215648902, in phase III. Characteristics of composite materials, 05/03/01 Compressibility of plied composite materials.

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