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BONDING OF THE THERMALLY MODIFIED NORWAY SPRUCE WOOD WITH THE PUR AND PVAc ADHESIVES

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ABSTRACT

The quality of the bonded joints in wood structures and furniture products is influenced by the surface characteristics of wood and the physical-chemical characteristics of adhesive. In this study the polyurethane (PUR) and polyvinyl acetate (PVAc) adhesives were used for bonding the Norway spruce (*Picea abies* /L./ Karst.) wood which was firstly dried at 100 °C and subsequently thermally modified for four hours at the temperatures of 160 °C, 180 °C, 200 °C, and 220 °C. The shear strength of the lap joints in bonded wood specimens, determined in accordance with the standard EN 205:2016, was analysed in relation to (a) the type of adhesive, (b) the temperature at thermal modification of wood, and (c) the composition of the bonded wood specimen formed from two slabs which were prepared from unmodified, unmodified and thermally modified, and thermally modified timbers, respectively. The shear strength of the lap joints was lower (a) at using the PVAc adhesive compared to PUR adhesive, (b) at using the wood slabs prepared from timber thermally modified at higher temperatures from 160 °C to 220 °C, and (c) for the specimens formed from slabs both thermally modified. In comparison to the reference specimens, there the highest decrease in the shear strength – by 42.3%, from 9.3 MPa at using PVAc adhesive, or by 56.1%, from 11.2 MPa at using PUR adhesive – was found for specimens formed from two slabs thermally modified both at 220 °C. Only at the highest modification temperatures, a cohesive type of failure occurred directly in the wood adherent.

Key words: polyvinyl acetate, polyurethane, shear strength, spruce, thermal modification.

INTRODUCTION

The use of wood in the construction industry is currently experiencing a renaissance. New joining methods and construction principles, as well as the discovery of the classic and modified wood-based material for modern architectural solutions, have opened up new possibilities for building with wood (ŠTEFKO and REINPRECHT 2004). The advent of different engineered wood products from sawn timber and veneer such as Cross-Laminated Timber, Glued-Laminated Timber, Laminated Veneer Lumber, *etc*. as a versatile material that can be fully combined with other building materials has led to its increased use for building detached houses, especially for multi-storey apartment buildings and in high-tech architecture. On the other hand, the world of the engineered wood products is rapidly changing and dynamic innovation process in this field is continuously expanding (SANDBERG *et al.* 2018).

The bonding quality of engineered wood products from sawn timber and veneers is affected by various factors to which mainly belong (1) the structural characteristics and physical properties of the wood used in a function of adherent such as its: - chemical composition which can be changed at its chemical and thermal modifications, as well as at its physical surface modifications with electrical discharge-corona, cold plasma treatments, laser treatment and mercerization (KAMKE and LEE 2007, PETRIČ 2013, NOVÁK *et al* 2015 KONNERTH *et al.* 2016, AICHER *et al.* 2018, BEKHTA *et al.* 2018, JAKES *et al.* 2018, REINPRECHT *et al.* 2020), - porosity, - surface free energy, - moisture content, - roughness which is influenced by wood morphology and its surface machining by sawing, sanding or planing (HASS *et al.* 2014, KNORZ *et al.* 2015), (2) the properties of the adhesive such as its: - chemical composition, - modification with additives, - solid content, - viscosity and surface free energy, - pH value, - buffering capacity, - hardening time, - rate of its curing or solidification (AYDIN 2004, HUNT *et al.* 2018, TRAN *et al.* 2020), and (3) the processing parameters of bonding such as: - spread rate, - open and closed assembly time, - temperature, - pressure (FOLLRICH *et al.* 2007, ŠMIDRIAKOVÁ and KOLLÁR 2010, BEKHTA *et al.* 2014).

Bonding of thermally modified wood can pose some issues. Changes in the chemical composition, anatomy, physical and mechanical properties of wood after thermal modification can affect the ability of adhesives at jointing the wood surfaces (SERNEK *et al.* 2008). The improved dimensional stability of thermally modified wood commonly improves the bonding performance, because the stresses due to shrinking or swelling on the cured adhesive bond of wood are reduced (REINPRECHT and VIDHOLDOVÁ 2008). However, heat treatment of wood can be expected to cause significant changes related to its adhesion with adhesives, which makes it necessary to adapt the bonding process (KRYSTOFIAK *et al.* 2013). Strong adhesion between the adhesive and the wood is achieved by appropriate liquid flow of the adhesive, its penetration into wood and following curing.

Thermally modified wood is less hygroscopic (HILL 2006, REINPRECHT and VIDHOLDOVÁ 2008, VIDHOLDOVÁ *et al.* 2019, KUČEROVÁ *et al.* 2019), which can alter the distribution of the adhesive on the wood surface and the penetration of the adhesive into porous of wood (FOLLRICH *et al.* 2006). The intensity of water absorption from the water-borne adhesive could affect its hardening process and subsequently the quality of the adhesive bond. Several studies have shown that the wettability of wood with water decreases after heat treatment (WANG *et al.* 2015, HUANG *et al.* 2012, BUDHE *et al.* 2020, KÚDELA *et al.* 2020), mainly because the surface of the heat-treated wood is more hydrophobic, less polar and significantly repellent to water (REINPRECHT and REPÁK 2019, BAAR *et al.* 2020). BASTANI *et al.* (2016a) found that the processing time needed for the adhesive to be absorbed into the thermally modified wood is higher due to slower penetration rate. Changes of the pH value of the thermally modified wood surface might retard or accelerate the curing of adhesives, depending on their type (CAI *et al.* 2018). Adhesives penetrates relatively easily into the voids and porous structure of wood tissue (KAMKE and LEE 2007, HUNT *et al.* 2018), also after its initial thermal modification (BASTANI *et al.* 2016b). Due to thermal modification of wood at higher temperatures, there in its anatomical structure are created other free spaces like cracks in the cell walls (TIRALOVÁ and MAMOŇOVÁ 2005, BOONSTRA *et al.* 2006). Additionally, there are created smaller substances due to depolymerisation reactions in its lignin-polysaccharide components, and changes occur also in the chemical reactivity of some chemical components of wood cell walls (INARI *et al.* 2007).

Both glue-laminated wood and thermally modified wood offers the interesting opportunities in area of the engineered wood products. In some studies (SERNEK *et al.* 2008, KRYSTOFIAK *et al.* 2013, MIRZAEI *et al.* 2017, and PULNGERN *et al.* 2020) the bonding performance of the thermally modified wood and also the glulam made only from thermally treated timbers were evaluated. However, there is no information about quality of the joints in bonded timbers when thermally modified and unmodified wood is bonded together.

The aim of this experiment was to determine the bonding performance of the thermally modified wood prepared at various modification temperatures – through the adhesive bond strength and the type of delamination.

MATERIALS AND METHODS

Wood materials

The sound Norway spruce (*Picea abies* /L./ Karst.) timber with the moisture content of 10% ± 2% were machine-milled to a thickness of 10 mm and dried at a temperature of 100 °C for four hours. The dry timbers were then exposed to thermal modification processes at the temperatures of 160 °C, 180 °C, 200 °C, and 220 °C, lasting four hours under atmospheric pressure in the laboratory heating oven Memmert UFE 500 (Schwabach, Germany). Finally, the timbers were cooled down and 14 day-long conditioned at a temperature of 23 ± 2 °C and a relative air humidity of 50 ± 5 %. Equilibrium moisture content of the unmodified timber was w = 9.1% ± 0.1%, while of the thermally modified timber was lower: 160 °C: w = 6.73% ± 0.10%, 180 °C: w = 6.23% ± 0.03%, 200 °C: w = 5.34% ± 0.15%, and 220 °C: w = 4.52% ± 0.18%.

Adhesives

Two different adhesives were used in the experiment: (a) one-component polyurethane (PUR) Kestopur 1030 (Kiilto Oy, Tampere, Finland), and (b) one-component polyvinyl acetate (PVAc) Rakoll® 4330 (H.B. Fuller Europe, Zürich, Switzerland). The specific characteristics of adhesives as well as the recommended processing conditions of these adhesive systems are summarized in Table 1.

Tab 1. Adhesive systems and processing conditions.

Adhesive	Kestopur 1030	Rakoll® 4330
Type	Polyurethane (PUR)	Polyvinyl acetate (PVAc)
Viscosity at 20 °C [mPa·s]	7000	13000
Density [kg/m^3]	1200	1100
Colour	Transparent, light after drying	White, transparent after drying
pH value	-	3
Recommended spread rate [g/m^2]	160-200	160-180
Open time [min]	30	8-12
Pressing time [min]	90-120	10-15

Lap joint shear "laminated" specimens

The spruce timbers dried at the temperature of 100 °C, as well as those following thermally modified at the temperatures from 160 °C to 220 °C, were machine-planed to a thickness of 5 mm and subsequently machine-grinded with sandpaper number of 120. The lap joint shear specimens were prepared according to the standard EN 205:2016 with these requirements: (a) only straight cut wood - parallel with the fiber orientation, and (b) the growth ring angle of the wood adherents only between 30° and 90°. The single-lap joints with the overlap of 10 mm were prepared from two wood slabs with dimensions of 80 mm × 20 mm × 5 mm.

The lap joint shear specimens were made from unmodified and thermally modified wood slabs as follows:

I. Slabs from unmodified timber (timber dried at 100 °C) – variant I (reference);

II. The combination of slabs of unmodified timber and thermally modified timber – variant II;

III. Slabs from thermally modified timber – variant III.

The schematic of single-lap joints for shear strength test – laminated spruce specimens – are shown in Figure 1. Due to the facts, that the curing reaction of PUR adhesives requires water, the thermally modified slabs were additionally moisturized by spring with water to rise the moisture content of their surface on 12 ± 2%. The spread rate of adhesives slabs surfaces was 180 g/m^2.

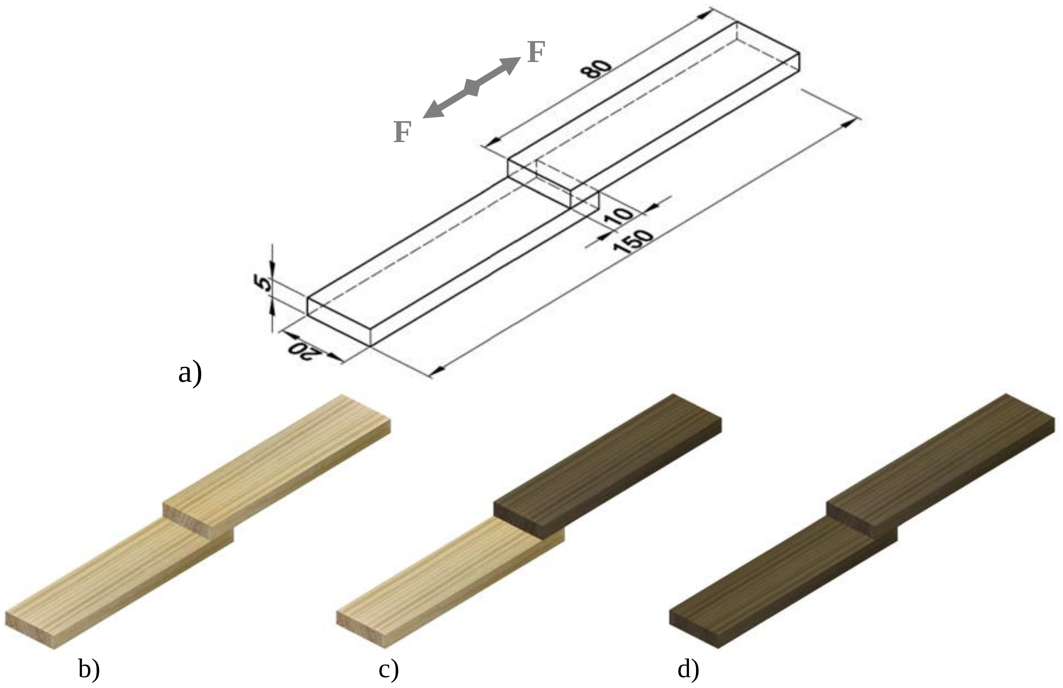

Fig. 1 The single-lap joint shear – laminated spruce specimens used in the experiment.

a) Schematic configuration of specimen according to the standard EN 205:2016. b–d) Single lap-joint of three tested variants: b - slabs from timber dried at 100 °C, c - the combination of slabs of timber dried at 100 °C and thermally modified timber, and d - slabs from thermally modified timber.

Shear strength test

The shear strength of the laminated single-lap joint shear specimens was tested in the machine LabTech 4.050 (LaborTech s.r.o., Opava, Czech Republic) with 5 kN head. Specimens were placed into the testing machine directly after being removed from the standard climate (20 °C, 65% RH, 7 days after bonding), and loaded with a speed of 50 mm·min^{-1} until breakage occurred according to EN 205:2016.

The shear strength was computed as the ratio between the maximal force and the bonded area (10 × 20 = 200 mm^2). Delaminating failures in the specimens were estimated visually.

RESULTS AND DISCUSSION

The highest shear strength of the bonded wood specimens was recorded for the reference ones – composed of two slabs prepared from unmodified timbers dried at 100 °C (variant I.)

– bonded with the PUR adhesive. At using the PVAc adhesive, the shear strength capacity of the reference specimens achieved only 83% comparing to using the PUR adhesive (Table 2).

The shear strength of the bonded specimens from unmodified and thermally modified timber (variant II) and only from thermally modified timber (variant III.) continuously decreased with increasing the temperatures 160-220 °C used at thermal modification. A higher reduction in the shear strength was determined with application of the PUR adhesive than with the PVAc adhesive (Table 2 - see average values, Duncan test, and p-level of significance, Figures 2 a 3). This result can be explained by the more significant effect of the increased hydrophobicity of thermally modified wood surfaces on the deteriorating bond quality of the PUR adhesive.

Tab. 2. The shear strength of the bonded specimens formed from the spruce slabs and the PVAc or PUR adhesives.

Slab's combination in bonded specimen	Shear strength									
	PVAc					PUR				
	Average	SD	Duncan test		Wood failure	Average	SD	Duncan test		Wood failure
	[MPa]		(*p*-level)		[%]	[MPa]		(*p*-level)		[%]
I. Slabs from unmodified timber (Reference)										
100°C/100°C	9.3	0.8	-		< 10	11.2	1.6	-		< 10
II. Slabs from unmodified timber and thermally modified timber										
100°C/160°C	9.2	1.1	d	(0.811)	10-20	9.5	1.5	d	(0.056)	10-20
100°C/180°C	8.3	1.0	d	(0.080)	20-30	9.4	2.1	d	(0.056)	10-30
100°C/200°C	7.9	1.2	c	(0.011)	40-50	7.9	1.7	a	(0.000)	90-100
100°C/220°C	7.0	0.8	c	(0.011)	90-100	6.2	1.3	a	(0.000)	90-100
III. Slabs from thermally modified timber										
160°C/160°C	8.0	1.2	c	(0.022)	10-20	8.2	1.3	b	(0.001)	10-20
180°C/180°C	7.8	1.2	c	(0.016)	40-50	8.0	1.4	b	(0.001)	90-100
200°C/200°C	6.6	1.3	a	(0.000)	90-100	7.5	1.3	a	(0.000)	90-100
220°C/220°C	5.4	0.6	a	(0.000)	90-100	4.9	1.1	a	(0.000)	90-100

Notes: Average - mean values from 10 replicates of tested single-lap joint of laminated specimens; SD - standard deviations; a, b, c, d - indexes of the Duncan test characterizing the significance level of shear strength in relation to the reference laminated specimens 100/100 (a - very significant decrease > 99.9%, b - significant decrease > 99%, c - less significant decrease > 95%, d -insignificant decrease < 95%).

The lowest shear strength of bonded specimens was determined in the case of using spruce slabs thermally modified with the highest temperature of 220 °C – i.e., drop in comparison to the reference specimens by 42.3%, from 9.30 MPa at using PVAc adhesive, or by 56.1%, from 11.20 MPa when using PUR adhesive. Results of the shear strength valued by the Duncan test (Table 2) show statistically significant differences in relation to the shear strength of reference specimens – from "c" (*p*-level lower than 0.05, at 95% level of confidence) to "a" (*p*-level lower than 0.001, at 99.9% level of confidence). The statistically lower shear strength was determined mainly for bonded specimens formed from slabs prepared only from the thermally modified timber (variant III. – significant and continuous decrease of strength from 160°C/160°C to 220°C/220°C). On the contrary, for the bonded specimens formed from the reference slabs "i.e., from timber dried at 100 °C" and the thermally modified slabs "i.e., from thermally modified timber", there at using slabs modified at lower temperatures (variant II. - combinations 100°C/160°C and 100°C/180°C) were not determined statistically significant decreases (*p*-level higher than 0.05)

By the linear correlations was analysed the decrease in the shear strength of the bonded specimens in dependence of the increased temperature during the thermal modification of spruce timbers (Figure 2). A significantly negative effect of the increased modification temperature (t) was confirmed by the coefficient of determination r^2 and the *p*-level = 0.000

of the linear correlation "σ = a + b · *t*". The r^2 was 0.390 for PVAc adhesive and 0.469 for PUR adhesive at the combination of slabs from timber dried at 100 °C and from thermally modified timber (variant II.), respectively, 0.648 and 0.654 at the combination of slabs only from thermally modified timber (variant III.).

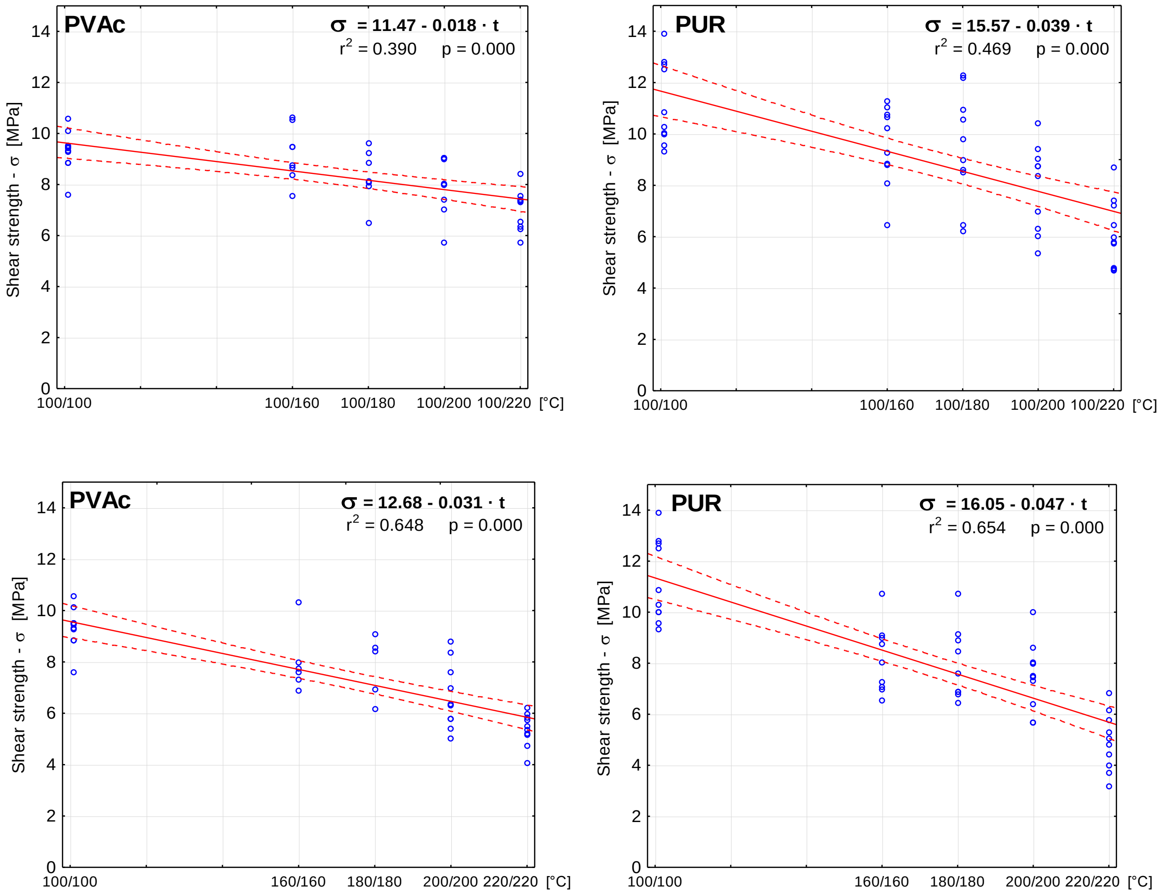

Fig. 2 Linear correlations between the shear strength (σ) of the bonded spruce specimens and the modification temperatures – including the drying temperature of 100 °C (*t*).

Generally, the shear strength of the bonded spruce specimens decreased apparently less for those ones made by combination of spruce timber dried at 100 °C and thermally modified timber, in comparison to the bonded spruce specimens made only from thermally modified timber (Figure 3).

The lower shear strength values of the bonded thermally modified timber determined some other researchers, as well. For example, UZUN *et al.* (2016) found out that the reduction of density and changes in surface properties of heat-treated wood, as well as the physical-chemical characteristics of adhesive, could potentially affect the bonding performance of thermally modified wood. ANDROMACHI and EKATERINI (2018) also mentioned that the shear strength reduction of bonded wood can be due its degradation during its previous thermal treatment in connection with its density reduction and not due to the reduction of the adhesive bond capacity.

The summarised view for the reduction of the shear strength of the bonded thermally modified timber were offered in study of TAGHIYARI *et al.* (2020). The shear strength reduction of bonds created from thermally modified timber can be attributed to: (a) a reduction of polar groups in the cell walls of wood due to the degradation of amorphous polysaccharides by the heat treatment, resulting in less sites available for bonding, (b) an increased stiffness of the cell walls after heat treatment, which results in a reduction of internal surfaces for chemical bonding or mechanical interlocking of adhesives, and (c) a reduction in wettability that may retard the proper penetration and curing of water-based adhesives such as PVAc adhesive. The formation of micro-cracks and checks due to the heat treatment at temperatures above 180 °C might also contribute to a declined shear strength of heat-treated wood.

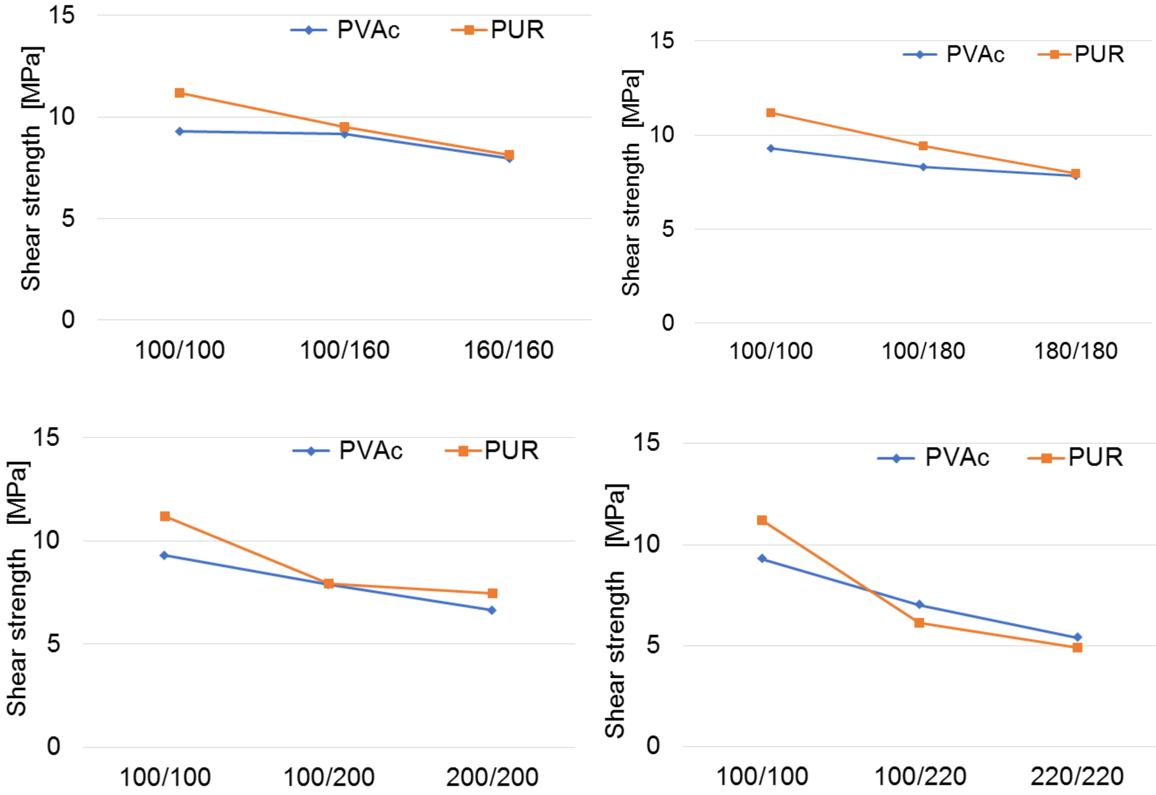

Fig. 3 The shear strength of the bonded spruce specimens formed from spruce slabs exposed firstly to temperatures from 100 °C to 220 °C.

The failures in the bonded specimens created during the shear tests were located mainly in the wood adherent thermally modified at the highest temperatures of 200 °C and 220 °C – cohesive type of failures (Table 2, Figure 4). It means that the adhesion of the used adhesive to the thermally weakened wood was higher than the internal cohesion strength of the thermally damaged wood.

Fig. 4. Failure modes of bonded spruce specimens at the shear test by EN 205:2016

CONCLUSIONS

- The shear strength of the single-lap joints, valued in the dry state of the reference bonded specimens prepared from the unmodified spruce slabs, was 11.2 MPa at using the polyurethane (PUR) adhesive or 9.3 MPa at using the polyvinyl acetate (PVAc) adhesive.
- Applying the thermally modified timber, the shear strength decreased more apparently if the bonded specimens were formed only from the thermally modified

timber and less apparently if they were formed both from the reference and the thermally modified timber – at which the shear strength continuously reduced with increased modification temperature of timber from 160 °C to 220 °C, comparable at using both adhesives.

- At the shear strength test the cohesive type of failure in the wood adherent occurred mainly if the spruce slabs were prepared from timber thermally modified at the highest temperatures of 200 °C and 220 °C.

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