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STRUCTURE OF PHLOEM AND WOOD/BARK SHEAR STRENGTH OF THE SESSILE OAK DURING DORMANT AND GROWING PERIOD

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ABSTRACT

Sessile oak (*Quercus petraea* (Matt.) Liebl.) with a 9.3% representation in the Slovak forests is an important wood material for the wood industry and pulp and paper industry. Oak bark with the structure and properties is completely different from the wood. Therefore the processing of logs is often necessary to separate the bark from the wood. The work evaluated shear strength on the interface wood / bark, in the course of year in the longitudinal and tangential direction, on the two moisture levels. The lowest shear strength values were found in the growing season fresh in tangential load. In the dormant period it was recorded almost twice a big the average value of the shear strength compared to the growing season. The values of shear strength in the longitudinal direction were approximately half higher than in the tangential direction. The values of shear strength in the dry state (w = 12%) grew on average compared to the fresh state in the longitudinal direction approximately about 280 %, and in the tangential direction about 360 %. Shear infringement was observed at the beginning of the growing season observed in the zone of early phloem. Based on analysis of shear area, it can be assumed that especially multiseriate phloem rays, significantly affect the adhesion at the interface wood / bark.

Key words: bark, sessile oak, shears strength, cambium, phloem.

INTRODUCTION

Bark as an integral part of every tree presents complex of complicated cell structure, which influences wood processing. Bark cell structure differs from wood cell structure significantly, therefore heterogeneity is increased by the cellular structure, chemical composition and physical and mechanical properties. Resulting from this high heterogeneity between wood and bark and bark itself, the requirement is that the bark should be separated from wood before processing it to pulp as completely as possible.

Oaks are the most-represented tree species in Slovakia after beech, which are processing to pulp. Oaks represents 13.1 % from all tree species, from this 9.3 % is *Quercus petraea*, 1.31 % is *Quercus robur* and 2.5 % is *Quercus cerris* (GREEN REPORT 2015).

Oak bark consists from living phloem and rhytidome (GRIČAR *et al*. 2015, HOWARD 1977). Phloem of *Quercus robur*, 4 to 13 mm thick, forms an important layer of oak bark. Phloem forms conductive part of bark, in which assimilation rate flows. His structure collapses when phloem in oak bark cease to perform the function of vascular tissue. Thin cell walls deform gradually and their arrangement becomes chaotic (HOWARD *et al.* 1977). Phloem forms annual increments. Early and late zone can be observed especially in the last year increment of phloem. Boundaries between increments cannot be clearly distinguished as in wood (GRIČAR 2010; BOWYER *et al.* 2003).

Oak phloem cell structure is possible to study just in a narrow strip near to cambium in last year phloem increment. Transition from early to late phloem was estimated based on creation of strip of fibres with secondary lignified cell walls (GRIČAR 2010). Phloem zone in oak bark is divided to collapsed and non-collapsed phloem (GRIČAR 2010, GRIČAR *et al.* 2015, SEN *et al.* 2011). GRIČAR *et al.* 2015 described structure of non-collapsed phloem on cross and radial cut of *Quercus petraea*. They noticed that early, the youngest annual increment of phloem is composed mostly from conductive sieve tubes (early sieve tubes) and axial parenchyma. Contrarily, late phloem consisted from higher number of groups of phloem fibre and narrower sieve tubes. Conductive function in phloem has sieve tubes and phloem rays. Phloem rays formed by phloem parenchyma secure horizontal flow of assimilate. Thick-walled phloem rays and sclerenchyma secure mechanical strength. A storage function is fulfilled by axial parenchyma cells (HOWARD *et al.* 1977, SEN *et al.* 2011, BOWYER *et al.* 2003).

Morphological structure of oak bark was studied by several researchers (GRIČAR *et al.* (2015), SEN *et al.* (2011), GRIČAR (2010), TROCKENBRODT (1995, 1994, 1991 and 1990), HOWARD (1977), WHITMORE (1962) and HOLDHEIDE (1951). According to WHITMORE 1962, morphology of bark cells of *Quercus petraea* only slightly differs from *Quercus robur* bark cells morphology. On the other hand, GRIČAR *et al.* (2015) state that proportion of early phloem is significantly higher in *Quercus petraea* than in *Quercus robur*. Sieve tubes of early phloem and axial parenchyma in *Quercus robur* collapse a few weeks after the termination of cambium (growing season). Afterwards, sclerenchyma being formed, mainly in multi row phloem rays. In *Quercus robur* is possible to observe radial groups of multi row rays together with sclerenchyma groups, which are formed into so called "palm formation" (GRIČAR *et al.* 2015). HOWARD 1977 also observed that oak bark has a large number of druse crystals in parenchyma cells. Wheeler et al 1989 described druse crystals as crystal more or less sphere shaped, from which some parts protrude and gives whole structure shape of a spherical star.

HOLHEIDE (1951) studied cell morphology of *Quercus petraea* and noticed that annual phloem increment had width about 0.2 mm at the beginning and later it was compressed to approximately half size (0.10–0.13 mm). Extensive early phloem of *Quercus petraea* contains only a few parenchyma cells. Conversely, late phloem is formed by incomplete bundle of phloem fibres followed by bundle of parenchyma cells. A few sieve tubes are situated into the bundle of parenchyma cells. Oval sieve tubes have 30–43 μm wide diameter in early phloem. Phloem rays are the main component of oak bark. They are always situated in late phloem in narrow, ribbed large groups. Diameter of phloem rays is approximately 15 μm wide. Lumen is narrow and ribbed. *Quercus petraea* has stripes of phloem rays 3–7 cells wide.

Similar study was made by SEN *et al.* 2011 on *Quercus cerris*. They found out that sieve tubes and axial parenchyma are formed at the beginning of every phloem increment firstly. Subsequently, tangential stripes fibres and sclerenchyma are formed with a slight delay.

Oak bark structure, especially phloem layer, is closely related to adhesion of bark on wood. Adhesion is expressed as an adhesiveness of two different structures, wood and bark. According to several authors (CHOW AND OBERMAJER 2004, FISCUS *et al.* 1982, HARDER *et al.* 1977, EINSPAHR *et al.* 1971), adhesion on wood/bark interface near phloem differs significantly not only between tree species but also between dormant and growing season.

HARDER *et al.* 1977 and EINSPAHR *et al.* 1971 evaluated wood/bark adhesion based on shear strength measured in dormant and growing season. EINSPAHR *et al.* 1971 focused to determine shear strength of *Quercus macrocarpa*. They noticed that disruption zone depends on season of dormancy and growth by evaluating the shear area on wood/bark interface. Their shear strength measurements showed different values in dormant and growing season.

Shear strength value was 0.5 MPa and 1.0 MPa in growing season in June and dormant season in August, respectively. Shear strength depends on quality and quantity of cambial zone, which varies during the year. Quality and quantity of cambial zone was studied by AKKEMIK *et al.* 2006 and GRIČAR 2013. They observed influences of various environmental factors on cambial activity.

EINSPAHR *et al.* (1971) localised zone of disruption near cambium or in cambium during growing season. On the other hand, zone of rupture moved to the phloem zone, between sclerenchyma fibres, crystal-containing parenchyma and sieve tubes, during dormant season. Increased adhesion is attributed also to phloem rays and fragile structure of oak bark, which unfavourable influence the debarking process.

According to PRISLAN *et al.* 2013, main factors influencing shear strength, except quality and quantity of cambial zone, are size and quantity of phloem rays, age of tree and climatic conditions (temperature, precipitation etc.).

The aim of the study is determination of shear strength on wood/bark interface of *Quercus petraea,* in longitudinal and tangential direction. Shear strength was measured in two moisture levels, fresh state and moisture after conditioning at 20 °C and φ 65 %. Evaluation of phloem cellular elements, analysis shear area on the wood/bark interface was done by scanning electron microscope.

MATERIAL AND METHODS

Material to determine adhesion wood/bark based on shear strength was collected from *Quercus petraea*, living dominant trees, about 100 years old, with 30-35 cm in DBH, grew on research area Včelien of the University Forest Enterprise of the Technical University in Zvolen in altitude about 450 m. a. s. l. in Kremnica Mountains. Research area was characterised as a mixed forest of hornbeam, oak and beech trees. Samples, 50 mm thick, were taken from three trunks from DBH height monthly from July 2015 to June 2016.

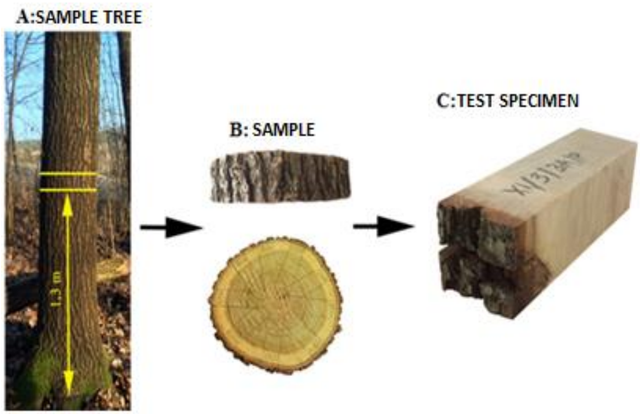

Fig. 4 Sequential steps to get the testing samples.

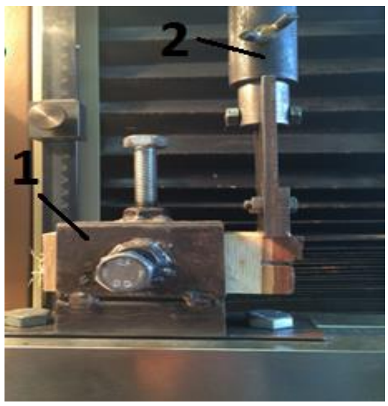

Fig. 5 Depiction test specimen an anchored by screws (1) and the jaw edges on the different interfaces wood/bark (2).

Subsequently, fresh samples were cut to test specimens with dimensions about 30 × 30 × 60 mm (L × T × R). Afterwards, bark surfaces were cut to two identical areas with dimensions about 30 × 13 mm. Then, shear strength was measured in fresh and dry state on thus created areas. Shear strength was measured in tangential and longitudinal direction. Dry state of specimens were reached by conditioning at temperature at 20 °C and relative humidity 65 %, which corresponded with equilibrium moisture content of specimen about 12 %.

Adhesion testing on wood/bark interface was performed on testing device TIRATEST. Specimens were fixed with anchoring screws. The loading arm of testing device was equipped with special thorn, which shape copied wood/bark interface. Tension – deformation diagram was recorded from every specimen, from which maximal loading force, needed to rupture the wood/bark interface, was determined. Value of maximal shear strength for longitudinal and tangential direction in fresh and dry state was calculated using equation:

$$\tau_{t/l} = \frac{F}{S} \text{ [MPa]} \qquad (1)$$

Where: $\boldsymbol{\tau_{t,l}}$ – limit of shear strength in the longitudinal and tangential direction [MPa] **F** – maximum loading force [N], **S** - shear area [mm^2].

According to GÄRTNER, SCHWEINGRUBER (2013), macerates of phloem zones were made for better understanding of adhesion on wood/bark interface. Macerating agent consisted from mixture of hydrogen peroxide and concentrated acetic acid at ratio 1:1.

Wood/bark interface shear area was analysed on wood and bark preparations. Preparations at dimensions about 10 × 10 mm (L × T) were metallised by gold and observed by scanning electron microscope.

RESULTS AND DISCUSSION

Shear strength on the boundary wood/bark

Shear strength was evaluated in two anatomical directions and two moisture levels (fresh and dry state). Results showed that values of shear strength changed during the year considerably (Fig. 6). Significant differences were observed not only between loading directions and moisture levels, but also between growing season and dormancy. These results correspond with several authors (CHOW AND OBERMAJER 2004, FISCUS *et al.* 1982, HARDER *et al.*1977, EINSPAHR *et al.* 1971). Higher values of shear strength were measured in longitudinal direction than in tangential direction in fresh state. Shear strength in tangential direction had about half lower values than in longitudinal direction in each month during the year (0.22 MPa, 0.37 MPa in April and 0.39 MPa 0.80MPa in September).

Growing season (fresh state) lasts from March to August at the sampling area. Similar results were observed by EINSPAHR *et al.* (1971). They defined duration of growing season from April to August. It should be noted that mentioned authors measured values of shear strength on other botanical oak species from other forest vegetation levels; however, trend curves show similar patterns as our results (Fig. 6). AKKEMIK *et al.* (2006) and GRIČAR (2013) reported that factors affecting cambial activity are average temperature, precipitation intensity and intensity of solar radiation. They also reported that thickness of bark can be considered as an important factor affecting the wood/bark adhesion.

Dormancy is characterised with higher values of shear strength. Equally, double increase of average shear strength was observed in longitudinal direction (0.61 MPa) in comparison with tangential direction (0.36 MPa). Similar values were measured by HARDER *et al.* (1977). They tested more oak species (*Quercus rubra, Q. alba,* Q. *falcata* a. o.). Higher values of shear strength were assigned for loading in longitudinal direction to orientation of particular construction elements around cambial zone, which are responsible for structural changes in zone of disruption between wood and bark. This is in accordance with HARDER *et al.* (1977) and EINSPAHR *et al.* (1971). They noticed that zone of disruption has been moving from cambium to phloem during dormancy. Some layers of last year phloem annual ring shows lower shear strength than cambial zone in dormancy.

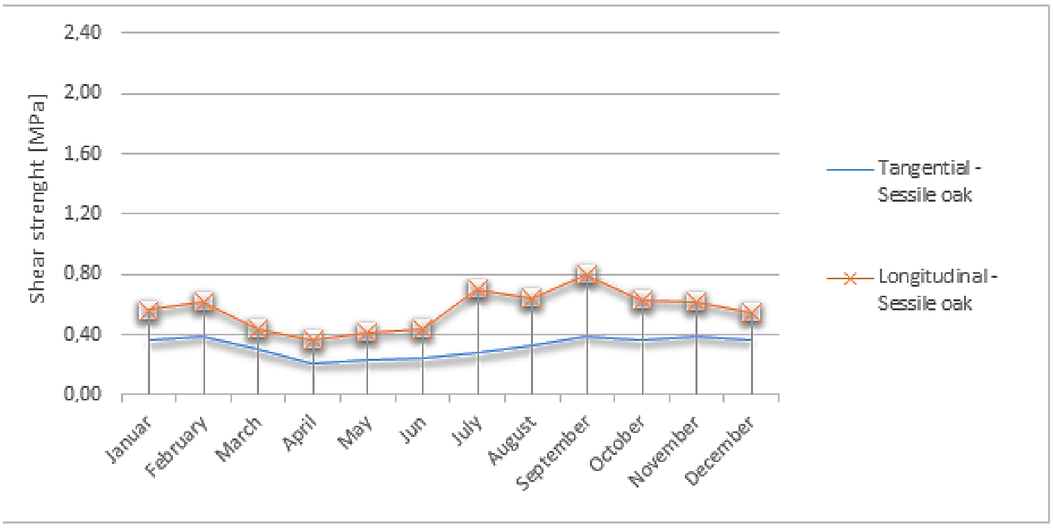

Fig. 6 Development of wood/bark shear strength in the fresh state on the wood species sessile oak.

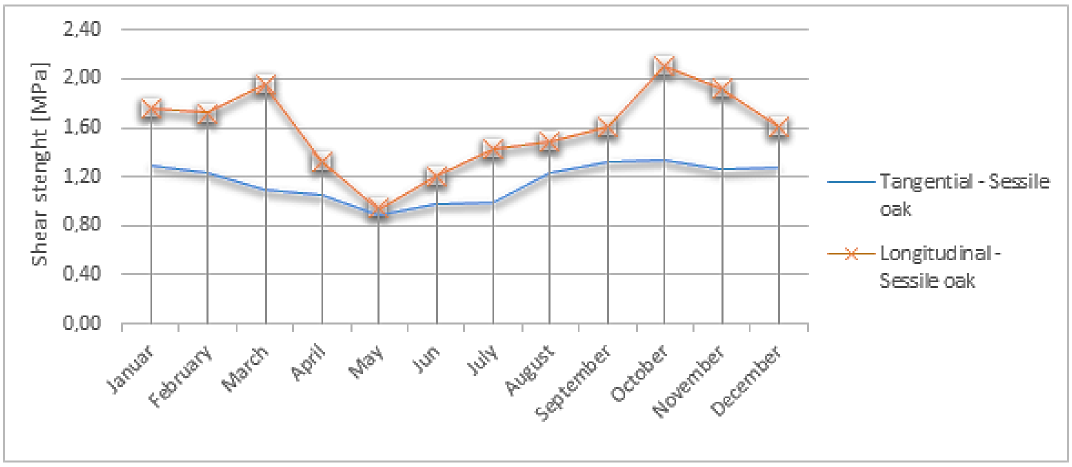

Fig. 7 Development of wood/bark shear strength in the dry state on the wood species sessile oak.

Dry state was achieved by conditioning of test specimens for three months in following conditions: 65 % humidity and temperature at 20 °C. Equilibrium moisture content of test specimens was around 12 %. Values of shear strength in dry state (w = 12 %) increased significantly. Average values increased about 280 % and 360 % in comparison with fresh state in longitudinal and tangential direction, respectively. This fact confirms that moisture content of wood and bark is the main factor affecting shear strength on wood/bark interface, CHOW and OBERMAJER (2004) observed effect of moisture content in inner phloem to shear strength of bark. Their measurements showed that with lowering the moisture content of inner phloem, the shear strength grows exponentially. Part of the vegetation season has shown to have effect on shear strength in dry state (Fig. 7). The lowest values were recorded in May (0.89 MPa) and June, July (0.98 MPa) in tangential direction, the same tendency was observed in longitudinal direction (May – 0.94 MPa), June – 1.21 MPa). The difference between longitudinal (1.59 MPa) and tangential direction (1.17 MPa) was more than 20 %.

Phloem structure of the sessile oak

Phloem of *Quercus petraea* was analysed in two layers. Inner phloem contained mostly non-collapsed increment. The second sample was separated from outer collapsed part of phloem. Inner non-collapsed phloem was composed from phloem fibres, sieve tubes, sclerenchyma cells, axial parenchyma and ray parenchyma. SEN *et al.* (2011) observed same composition of inner phloem of *Quercus cerris* and they claimed that fibres and sclerenchyma are created with slight delay. Similar cellular composition described HOLHEIDE (1951). He divided annual increment to early and late phloem. Phloem ray is characteristically composed from thin parenchyma cells. Thick-walled sclerenchyma creates margin of the ray (Fig 9 B). GRIČAR *et al.* (2015) stated that thin one-row phloem rays are composed only from thin-walled parenchyma cells. Multi row rays have thick-walled sclerenchyma and parenchyma filled with prismatic crystals on their circumference. Prismatic crystals were observed in axial and ray parenchyma (Fig. 9A). HOWARD (1977) observed druse crystals in parenchyma cells. These druse crystals were also observed in thin-walled axial parenchyma in our specimens (Fig. 9A).

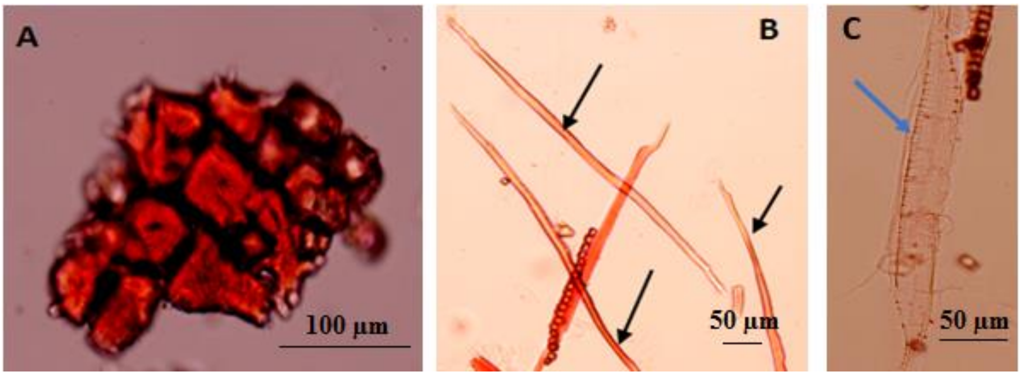

Fig. 8 Maceration of phloem the sessile oak. A – sclereids, B – phloem fibres (black arrow) and C - sieve tube (blue arrow).

The newest increment was not significantly differed from collapsed increment in morphological structure. Difference was noticed only in presence of phloem fibres and sclerenchyma (Fig 8 A, B), which was higher in collapsed phloem. Contrarily, presence of sieve tubes was higher in the youngest phloem increment. Oak bark can be divided to two main zones; non-collapsed (functional) and collapsed (unfunctional) phloem (GRIČAR 2010, GRIČAR *et al.* 2015, SEN *et al.* 2011). Thin-walled sieve tubes collapse and lose their conductive function after one year (HOWARD 1977). Volume rate of sclerenchyma and phloem fibres increases by collapsing the sieve tubes.

Thick cells, phloem rays and sclereids were observed in the oak bark. The thickness of the cell walls can be assumed that cells are participating on the mechanical function of bark. Storage function on the vice versa assumed axial and ray parenchyma (HOWARD *et al.* 1977, SEN *et al.* 2011, BOWYER *et al.* 2003). The conductive functions in phloem provide thin sieve tubes, but only in no collapsed zone of phloem. Sieve tubes cease to perform conductive function, which collapsed in second phloem growth.

Structural evaluation the shear surface

For better understanding of the impact structure of bark on the shear strength, surface of shear was analysed from the point of view bark and wood. There was evaluated in which zone of cambium or phloem the shear strength was occurred after load from the boundary wood/bark. For this review, microscopic preparations were used to analyse the surface of the wood or bark.

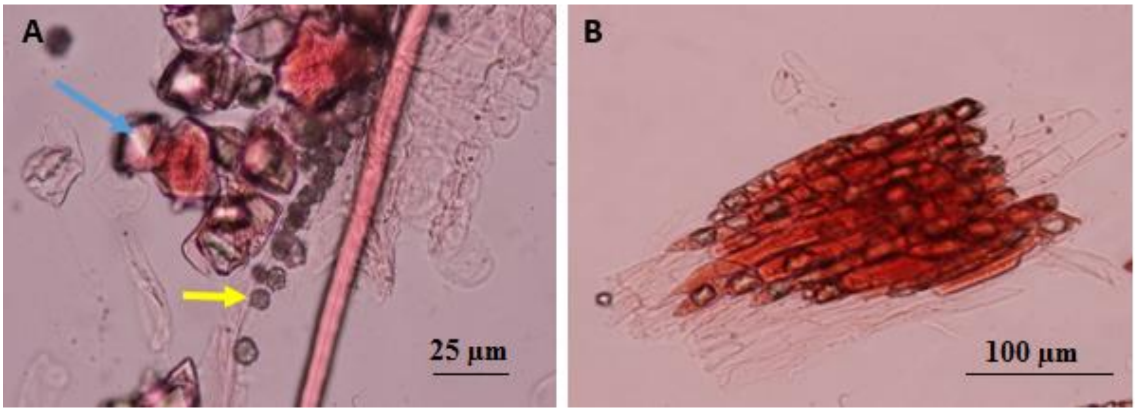

Fig. 9 The look of: A – prismatic (blue arrow) and druses crystals (yellow arrow), B – Phloem ray containing sclereids and prismatic crystals.

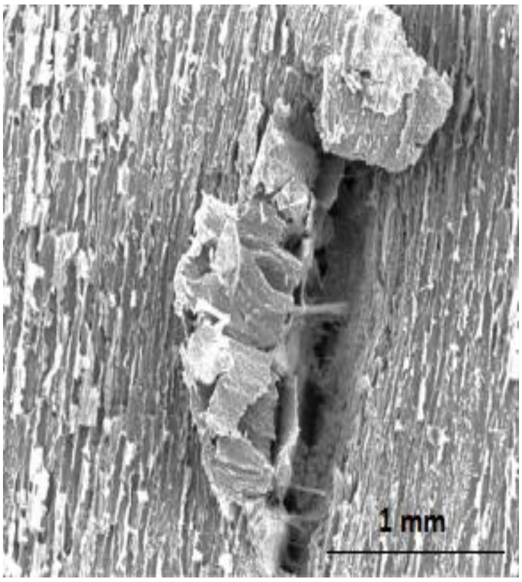

Fig. 10 Shear surface conduct through youngest sieve tubes. From the point of view bark.

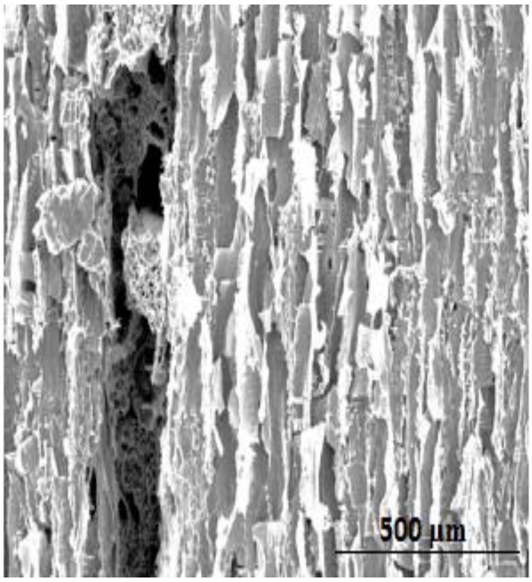

Fig. 11 Shear surface conduct through youngest sieve tubes. From the point of view wood.

On the samples that were selected from the trunk on the beginning of the growing season was observed shear damage in zone of early sieve tubes. Early sieve tubes are thin and relatively wide in compared with thin wall of cambial cells. Sieve tubes are not lignified, which can also reduce their impact on the shear strength. GRIČAR *et al.* (2015) shows for early sieve tube diameter of lumen 49.7 µm. Early phloem is formed mainly by the thin sieve tube cells and small proportion of the axial parenchyma (SEN *et al.* 2011, HOWARD *et al.* 1977). Phloem fibres and sieve tube was not found in early phloem. GRIČAR *et al.* (2015) measured youngest increase of phloem which has 2.1 % percentage of phloem.

Rays have significant effect on shear strength on wood/bark interface. Likewise rays in wood, rays can also be found in phloem. The rays have one to three or multiple rows. Narrow phloem rays composed of non-lignified thin-walled parenchyma cells are sheared off in cambial zone or alternatively in early phloem when loaded. Multi row phloem rays 7 and more row thick are not sheared off on shear plane, but they are being pulled out from wood ray in wedge like form. Thus, phloem rays ascend above shear disruption zone. Contrarily, deep apertures occur in place of multi row phloem rays when analysing shear zone in wood. Different shear disruption of one row and multi row phloem rays can be explained by different structure (GRIČAR *et al.* 2015, SEN *et al.* 2011, HOWARD *et al.* 1977). Whereas narrow phloem rays are composed of thin-walled and non-lignified parenchyma cells, thick, multi row phloem rays may include thick-walled sclerenchyma in the youngest phloem increment, which significantly increase shear strength of ray. The most of prismatic crystals occur in parenchyma cells of multi row phloem ray (GRIČAR *et al.* 2015), which also contribute to increase of ray shear strength (Fig. 14). Druse crystals occur in axial parenchyma in addition to prismatic crystals (Fig. 15).

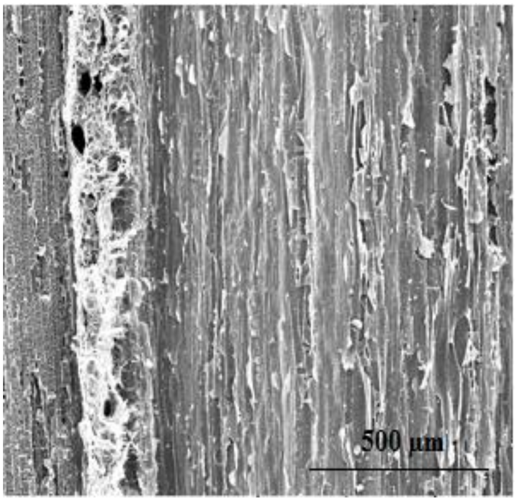

Fig. 12 Shear surface from the bark side. Multi phloem ray pulled off from wood.

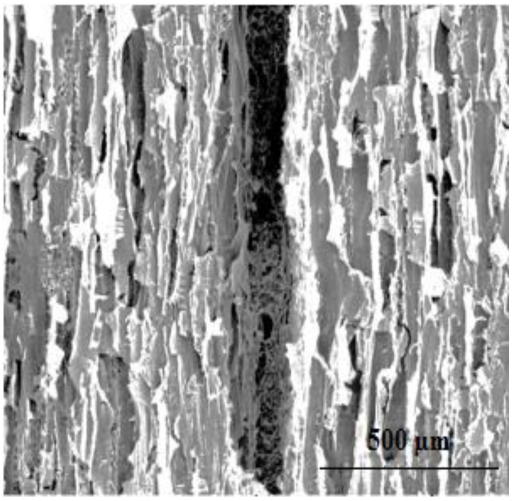

Fig. 13 Shear surface from the wood side. Wedge-shaped crater in the wood after removal phloem ray.

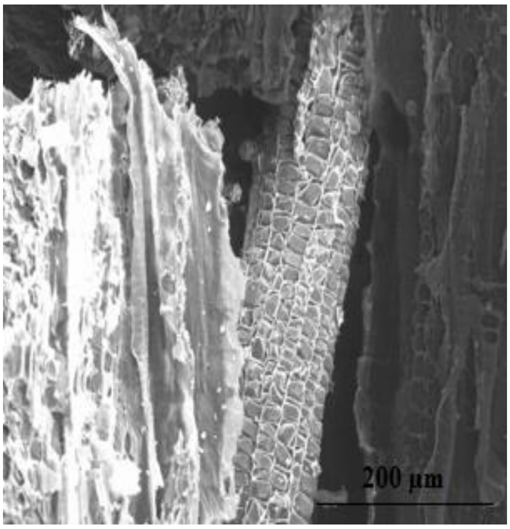

Fig. 14 Prismatic crystals in fragments of phloem rays.

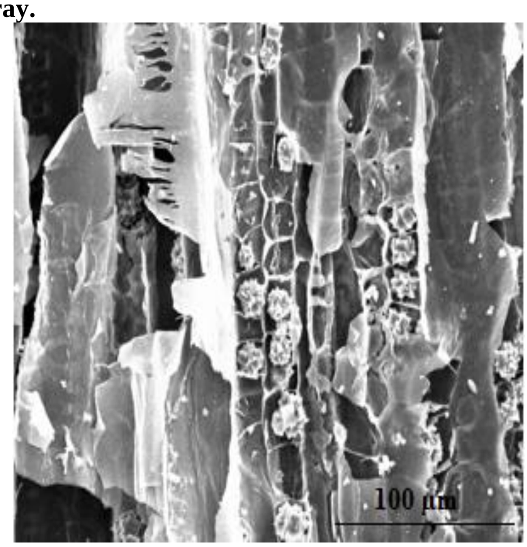

Fig. 15 Druse crystal in filling parenchyma.

CONCLUSION

- The samples of sessile oak (*Quercus petraea* L.) was assessed by shear strength at the interface wood / bark, on a monthly basis from July 2015 to June 2016 in two anatomical directions (longitudinal and tangential) and two moisture levels (fresh and conditioned state about 12 %).

- Shear strength values in the fresh state, in the longitudinal direction was approximately about half higher than in the tangential direction.
- For the dormant period almost twice a big average value of the shear strength was recorded in comparison with the growing season, as well as in the longitudinal and tangential directions.
- The values of shear strength in the dry state (w = 12 %) grew up in compare with fresh state in the longitudinal direction approximately about 280 %, and in the tangential direction by about 360 %.
- Shear fracture was observed in the zone of the first early sieve tubes at samples from the beginning of the growing season.
- Important role in the interface shear strength on the border wood /bark have multiseriate phloem rays which are at a shear cut his load in the plane of the infringement, but be pulled out of the wood ray wedge-shaped.
- Multiseriate phloem rays unlike the single contain a large amount of thick-walled sclereids and parenchymal cells with prismatic crystals.

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