

## **Mechanical qualities of adhesive bonds reinforced with biological fabric treated by plasma**

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**Abstract.** The paper deals with the utilization of a biological reinforcement in the area of an adhesive layer at structural adhesive bonds. A significant disadvantage of adhesive bonds is uneven layer of an adhesive, which can be eliminated by various technological procedures. One possibility is to use a reinforcing even layer. The primary aim of this paper was to experimentally investigate an influence of the surface plasma treatment of natural fabrics (flax, cotton) at different intervals of plasma affecting (0 to 90 seconds and power 350 W) on mechanical properties of the adhesive bond. There were positive results from reinforcing the adhesive bond by a layer of linen and cotton. Strength characteristics of reinforced adhesive bond were increased compared to non-reinforced adhesive bonds. When the linen was used, the strength was increased by 43.2% and when the cotton then 15.5% strength increase could be seen. When modifying the surface by plasma, next adhesive bond's strength increase was seen. Using the linen there was approx. 47% strength increase, using the cotton the strength increase was approx. 38% compared to non-reinforced adhesive bonds (without reinforcing phase). It is obvious from the results that plasma modifying showed better results when the cotton was used as the reinforcing material. SEM analysis proved that adhesion was improved with plasma surface modification of biological fibres. In other words the distance between the warp and the resin was significantly decreased for 87.1% when using the cotton and by 46.5% when the linen was used.

**Key words:** Cotton, flax, plasma treatment, SEM.

### **INTRODUCTION**

Adhesive bonding technology is a common method of linking in the industry. Various researches deal with different possibilities of how to increase adhesive bond's strength, which would contribute to better results at the practical application. A significant disadvantage of adhesive bonds is an uneven layer of an adhesive, which can be eliminated by various technological procedures. One possibility is to use a reinforced even layer. Main goal in the adhesive bonding technology research is the quality increase of the adhesive bond, especially the strength increase and the increase of the resistance to external factors.

The aim of the adhesive bonding process is to create a bond, which can provide maximum strength and quality for each adhesive and adherent combinations at minimum costs. According to Messler (2004) following conditions have to be met in order to reach that goal:

- Appropriate modification and surface cleanness of adherents before the application of the adhesive. This will cause proper wetting of the adherent by the adhesive.
- Proper choose of the adhesive for each single adherent and prevailing conditions of use.
- To assure constant layer of the adhesive.
- To take into consideration external surrounding factors that can affect adhesive bonds

Use of reinforcing fabrics in the adhesive layer means better distribution of the adhesive. This distribution has to be even. There are different deformations of adhesive layers in adhesive bonds, where even distribution of the adhesive is not guaranteed. This causes less strength of the adhesive bond.

Due to uneven deformation there is different adhesive deformation through a thickness of the adhesive layer. The deformation is biggest at the end of lapping. So called 'stress peaks' are emerged at the edge of the adhesive layer which causes a hyperbolic course of the stress in the whole length of the lapping.

The concentration of the stress at the edge of the adhesive bond is increased by the impact of bending moment. This causes a breach of the adhesive bond. The reason is uneven distribution in the adhesive layer caused by a flexibility and deformations of adhesive materials. This creates pulling stress at the edge of the bond which is a reason of peeling off with related decrease of the strength. A consequence is a spread of leaks and following destruction of the adhesive bond. A significant contribution for adhesive bonds is a reinforcing layer with fabrics. (Researches with fabrics from glass fibres showed positive influence on strength increase of the adhesive bond). Possibilities and limits of fibre-composite materials are subjected to long-term research in different research fields (Fu et al., 2002; Pothan, 2005; Wong et al., 2010; Hong et al., 2012; Zavrtalek & Müller, 2016b).

Composites made from natural (Idicula et al., 2005; Pothan et al., 2007; Zavrtalek & Müller, 2016b) and biologically flawless matrixes play big part in recent researches Schorr et al., 2014; Thakur et al., 2014). Composites made from these materials decrease the impact on nature surroundings during the production process and also after a lifespan of the fibre composites (Shi et al., 2012; Patlolla & Asmatulu, 2013).

The use of the synergic effect which leads to improved adhesive bond's mechanical qualities is well-known technology and nanoparticles are used for this in particular (Park et al., 2009; Dorigato et al., 2010; Ahmad et al., 2012). But the production of nanoparticles is often very costly. Next problem is their even layout in the matrix, caused for example by a sedimentation. This is technologically very difficult process. This fact leads to the research of the appropriate alternatives which use the synergic effect in adhesive bonds. For example the use of synthetic and natural reinforcing fabrics.

Basic characteristics of the fabric are very important in order to use natural and synthetic fabrics in the field of composite materials (material, fibre orientation, specific weight), their wetting with matrix etc. (Fowler et al., 2006; Karbhari & Abanilla, 2007; Lee et al., 2010; Maheri, 2010; Mizera et al., 2016a; Mizera et al., 2016b).

Already published studies showed that natural fibres like linen, jute, hemp, sisal and pineapple have serious advantage compared to the conventional fibres due to their low density (Sankari, 2000; Munawar et al. 2006; Rao & Rao, 2007; Silva et al., 2008;

Alves et al., 2010; Faruk et al., 2012). Thanks to the low density they have high measured strength and toughness.

The most used fibres are from the following plants: sisal, linen, hemp, cotton, jute. Fibres of natural origin are used as cheaper alternative to the glass fibres.

The main disadvantages of natural fillers utilized in the polymeric composites are their low wettability and non-homogeneity (Herrera-Franko & Valadez-González, 2005; Mizera et al., 2016a). The fibres are mostly hydrophilic natural fibres. Especially these factors are a reason for their low tensile strength (Sharifah & Martin, 2004; Alkbir et al., 2016).

These problems can be alleviated through the use of suitable compatibilisers and plasma surface treatment (Hrabě et al., 2016).

The main disadvantage of plant fibres lies in a combination of a non-polar polymer matrix (hydrophobic) and polar plant fibres (hydrophilic). This combination creates a poor interface with a low adhesion of both components. That implies a poor wettability of fibres by the polymer matrix and low mechanical properties of composites (Boruvka et al., 2016).

The good wettability of the matrix and the reinforcement improves an efficiency of a stress transmission from the matrix to the fibre (Müller et al., 2013; Hrabě et al., 2016). The plasma surface treatment is a suitable method. The plasma can influence the surface energy and clean the surface at the same time (Hrabě et al., 2016).

Generally, plasma treatments have the ability to change the surface properties through the formation of free radicals, ions and electrons in the plasma stream (Boruvka et al., 2016; Hrabě et al., 2016). During the plasma treatment, the surface of the substrate is bombarded with high-energy particles. Surface properties such as the wettability, the surface chemistry and the surface roughness of the substrate can be altered as a result, without the need for any hazardous chemicals or solvents (Boruvka et al., 2016).

A major advantage of employing low pressure plasma treatments is that such plasma can be generated at the low power output. The use of the plasma technology is among the most efficient and economical methods of modifying the surface properties of polymers and fibres without affecting the internal structure. The plasma modifies the surface of microfibres by removing weakly attached surface layers and forming new functional groups on the surface (Boruvka et al., 2016).

The paper deals with the utilization of a biological reinforcement in the area of the adhesive layer. An advantage of the biological reinforcement application is a simplification of following recycling of adhesive bonds compared to used carbon and glass fibre based reinforcements. A good wettability of the reinforcing layer improves an efficiency of a stress transfer in the layer of the adhesive. Plasma can influence a surface energy and clean the surface at the same time.

A chemical cleaning is another possibility of a natural fibre treatment. A rise of waste chemical substances is a disadvantage of the chemical cleaning.

The primary aim of this paper was to experimentally investigate an influence of the surface plasma treatment of natural fabrics (flax, cotton) at different intervals of plasma affecting (0 to 90 s and power 350 W) on mechanical properties of the adhesive bond.

## MATERIALS AND METHODS

Waste fabrics from the manufacturing industry was used for the research. Cuttings in a form of strips were used. This fabric cannot be used in the textile industry due to its size.

Samples were cut from flax and cotton into strips of 30 x 200 mm. The samples were modified by the plasma treatment (Fig. 1).

The strengthening reinforcement was the fabric of cotton and flax. Flax: the mass of the fabric ca. 300 g m<sup>-2</sup>, cotton: ca. 150 g m<sup>-2</sup>. Plasma was generated from a plasma generator (Plasma Reactor KPR 200 mm RM 54) while supplying the reaction gas (oxygen) and maintaining the reactor's pressure at 0.1 Torr with the use of a vacuum pump.

To determine the properties that depend on the discharge power and the treatment time, the plasma treatment was conducted in the power range 350 W for 15, 30, 60 and 90 s.

Adhesive bonds were prepared in accordance with requirements of the Czech standard ČSN EN 1465. The length of adhesive bonds lapping was 12.5 ± 0.25 mm. Adherents from the structural carbon steel S325J0 were used for the research. Those adherents are in accordance with the Czech standard ČSN EN 1465 as far as the size and the shape are concerned (100 x 25 x 1.5 mm). Samples were lit up by the plasma. There was 48 hours delay before the plasma treatment and the adhesive procedure. A construction of the composite reinforced adhesive bond can be seen in Fig. 2, A.

The arrangement of the reinforcing fabrics, i.e. a weft and a warp in the layer of the adhesive is visible in Fig. 2, A. The warp is a bearing element of the fabric, the weft serves for connecting of fabric.

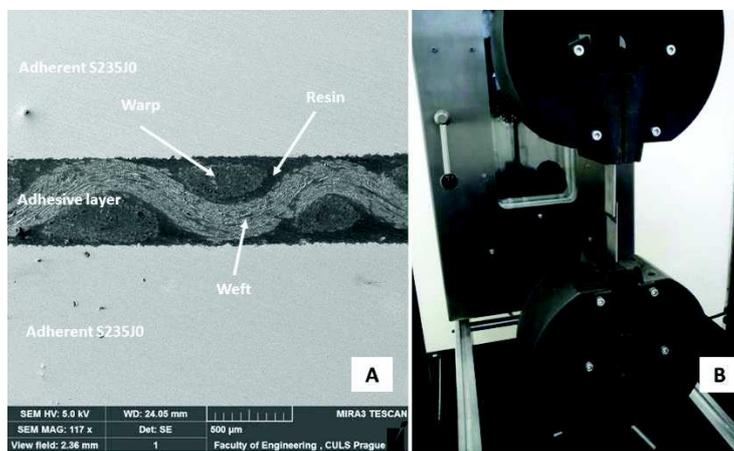
The surface of adherents was mechanically and chemically treated before the adhesive bonding. The mechanical treatment consisted in grit blasting by the garnet MESH 80 under the angle 90 °. The chemical treatment consisted in the cleaning in a bath of acetone. Using the profilograph SurfTest 301 the following values were determined: Ra 1.18 ± 0.08 µm, Rz 6.4 ± 0.96 µm.

A structural two-component epoxy adhesive Glue Epox Rapid (next only Resin) was used in the adhesive bonding process. The reinforcing fabric was put into the layer of the adhesive whose surface was treated for various times in the plasma. The principle consisted in putting the adhesive layer on the first adhesive bonded part, subsequently the fabric was applied and the second adhesive bonded part was attached on which the layer of the adhesive was also deposited. A resulting bond was fixed by a pressure 0.02 MPa. Adhesive bonds were hardened for 48 h under the temperature 22 ± 2 °C and the moisture 65 ± 8%.

The tensile strength test (according to CSN EN 1465) was performed using the universal tensile strength testing machine LABTest 5.50ST (a sensing unit AST type KAF 50 kN, an evaluating software Test&Motion). The course of the test is visible in Fig. 2, B. A speed of the deformation was 5 mm min<sup>-1</sup>. The deformation speed influences the adhesive bond strength. The research results proved that the adhesive bond strength increased with increasing deformation speed. The research results proved that the time of the adhesive bond destruction according to the requirements of the standard CSN EN 1465 was reached at this speed (Muller et al., 2016). Ten test samples were always tested in single series. The failure type was determined at the adhesive bonds according to ISO 10365.



**Figure 1.** Reinforcing phases. A: SEM images of cotton MAG 84 x, B: SEM images of flax MAG 155 x, C: surface treatment of flax using plasma discharge at 350 W.



**Figure 2.** Adhesive bond testing: A: SEM images of cotton – construction of adhesive bond using biological reinforcing fabric (MAG 117 x, secondary electrons), B: tensile strength testing of adhesive bond in universal testing machine LABTest 5.50ST (according to CSN EN 1465).

Fracture surfaces and an adhesive bond cut was examined with SEM (scanning electron microscopy) using a microscope MIRA 3 TESCAN (the fracture surfaces were dusted with gold) at the accelerating voltage of the pack (HV) 5.0 kV and a stereoscopic microscope Arsenal. The difference of the saturation of the various types of fabrics with the epoxy adhesive was observed with SEM.

An evaluation of the shape and the dimensions was performed using the program Gwiddion. The results of measuring were statistically analysed.

Statistical hypotheses were also tested at measured sets of data by means of the program STATISTICA (*F-test*). A validity of the zero hypothesis ( $H_0$ ) shows that there is no statistically significant difference ( $p > 0.05$ ) among tested sets of data. On the contrary, the hypothesis  $H_1$  denies the zero hypothesis and it says that there is a statistically significant difference among tested sets of data or a dependence among variables ( $p < 0.05$ ).

## RESULTS AND DISCUSSION

This experiment brings new pieces of knowledge of how to use biological fabrics in the field of adhesive bonds in order to increase the strength of the adhesive bonds. In the Table 1 there are basic parameters of the adhesive bond. A support frame for the fabric reinforcement is the warp, the weft is used for the fabric connection. The adhesive width showed difference of 9.5%. When the reinforcing fabric was used the difference was decreased to approx. 4.5%. The adhesive layout showed more even distribution when the reinforcing fabric from the flax was used. This finding is very important for the construction of adhesive bonds.

**Table 1.** Basic characteristics of tested adhesive bonds

Adhesive bond characteristic	Adhesive bond thickness ( $\mu\text{m}$ )	Warp – Fabric thickness ( $\mu\text{m}$ )
Resin	$238.93 \pm 22.48$	–
Resin and reinforcement in form of linen fabrics	$266.05 \pm 11.92$	$9.55 \pm 3.36$
Resin and reinforcement in form of cotton fabrics	$496.66 \pm 24.42$	$14.99 \pm 4.04$

A graphic presentation of the results of the adhesive bond strength can be seen from Fig. 3. There were positive results from the reinforcing adhesive bond by the layer of the flax and cotton. Strength characteristics of the reinforced adhesive bond were increased compared to non-reinforced adhesive bonds. When the flax was used, the strength was increased of 43.2% and when the cotton then 15.5% strength increase could be seen. When modifying the surface by the plasma, next adhesive bond's strength increase was seen. Using the linen there was approx. 47% strength increase, using the cotton the strength increase was approx. 38% compared to non-reinforced adhesive bonds (without reinforcing phase). It is obvious from the results that the plasma modifying showed better results when the cotton was used as the reinforcing material.

Adhesive bonds without the reinforcing fabric showed an adhesive type of the failure. When the reinforcing material was added, the adhesive failure type changed to adhesive-cohesive type, where the adhesive type was dominating. This result was caused by even distribution of the adhesive.

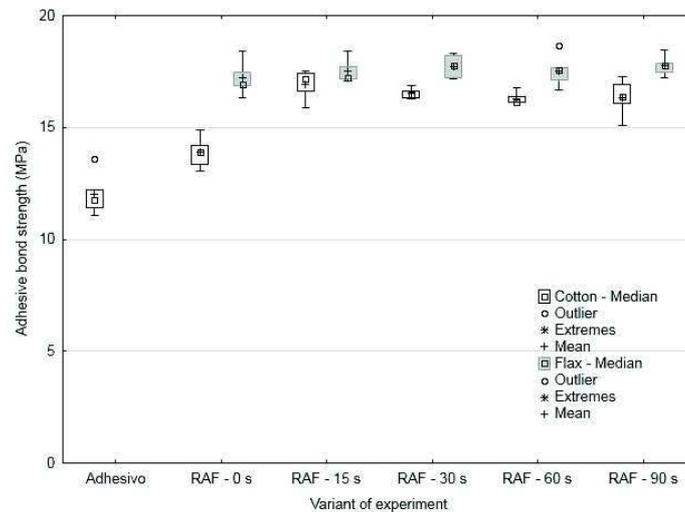
It is obvious from the results that the reinforcing fabric influences the strength of the adhesive bond, so there is the difference between the adhesive bonds with and without the reinforcement ( $p = 0.000$ ).

The hypothesis  $H_0$  was not certified so there is the difference among tested variants in relation to the adhesive bond strength on the reliability level 0.05. Use of the biological reinforcement from the flax and the cotton can be considered as the significant factor which has the impact on final strength of the adhesive bond.

If we check statistics of testing of the plasma surface modification, we can conclude following findings: When the flax was used as the reinforcing material, the plasma modification did not have any proved effect on the strength of the adhesive bond ( $p = 0.641$ ), when the cotton was used as the reinforcing material, the plasma modification statistically proved the impact on the strength of the adhesive bond ( $p = 0.000$ ).

It is also obvious from the results that different time of the plasma surface modification in interval between 15 up to 90 seconds does not have any impact on the final strength of the adhesive bond (flax  $p = 0.872$ , cotton  $p = 0.343$ ) on 0.05 reliability level.

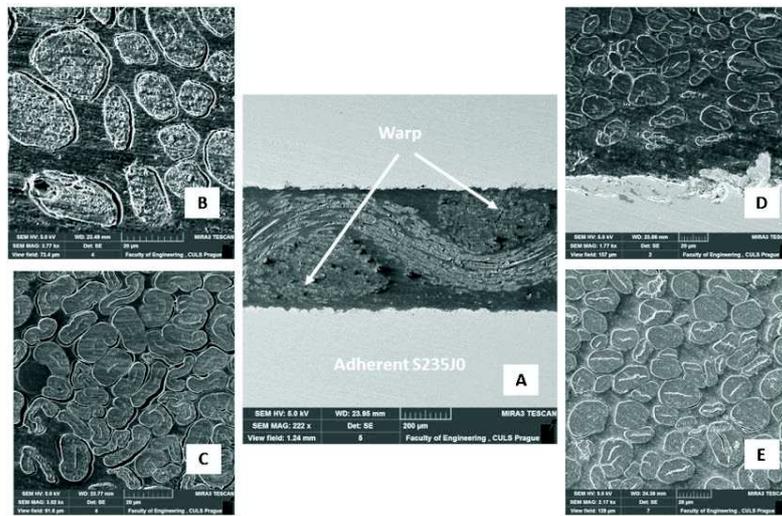
The time used for the plasma surface modification of tested biological fabrics cannot be considered as the significant factor in the adhesive bond application.



**Figure 3.** Dependence of adhesive bond strength on plasma surface modification of reinforcing fabric. (adhesive – without reinforcing phase, RAF – adhesive reinforced with fabric, number shows duration for how long plasma was affecting reinforcing fabric in seconds).

SEM (scanning electron microscopy) analysis was used for a study of the fracture surfaces and cuts of the adhesive bonds. SEM analysis enabled to display the quality of the interaction of the reinforcing biological fabric and the resin. It proved better wetting of the structural epoxy adhesive and the reinforcing phase. SEM analysis proved a good wettability of the adhesive bonded material with the adhesive, see e.g. Fig. 4, A and 4, D. This conclusion is essential because the wettability of the adhesive bonded surfaces is crucial for good adhesive strength (Müller, 2011; Rudawska, 2012; Müller & Valášek, 2013; Müller & Valášek, 2014; Müller, 2015; Müller, 2016; Rudawska et al., 2016).

On the Fig. 4, A the layout of the reinforcing fabric is visible, i.e. the weft and the warp in the adhesive layer. The warp is the bearing element of the fabric. On the Fig. 4, A the warp shape of the cotton is apparent. The diameter of the cotton warp was  $14.99 \pm 4.04 \mu\text{m}$ . From the Fig. 4, C the flax warp shape is apparent. The diameter of the flax warp was  $9.55 \pm 3.36 \mu\text{m}$ . From the Fig. 4, B and 4, C a bad wetting of the warp with the resin is visible. Bad wetting of the reinforcing cotton fabric (warp) and resin was ascertained with the optical analysis, this was showed by the distance  $1.16 \pm 0.86 \mu\text{m}$  (Fig. 4, A). Bad wetting of the reinforcing flax fabric (warp) and resin was ascertained with the optical analysis, this was showed by the distance  $0.98 \pm 0.43 \mu\text{m}$ . The adhesion was improved by the plasma treatment of biological fibres, i.e. the distance between the warp and the resin was decreased dramatically (Fig. 4, D; 4, E).



**Figure 4.** SEM images of cut through adhesive bond reinforced with biological reinforcing fabric (secondary electrons): A: resin reinforced with fabric from cotton MAG 222 x, B: cut through adhesive bond, poor wettability of reinforcing fabric (cotton) with resin (interaction of warp and resin) MAG 3.77 kx, C: cut through adhesive bond, poor wettability of reinforcing fabric (flax) with resin (interaction of warp and resin) MAG 3.02 kx, D: cut through adhesive bond, satisfactory wettability of reinforcing fabric (cotton) with resin after 30 seconds, reinforcing fabric treated by plasma (interaction of warp and resin) MAG 1.77 kx, E: cut through adhesive bond, good wettability of reinforcing fabric (cotton) with resin after 90 seconds reinforcing fabric treated by plasma (interaction of warp and resin) MAG 2.17 kx.

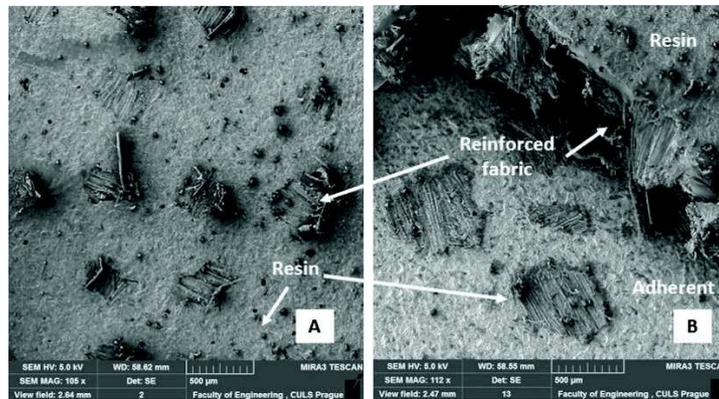
It is obvious from the results shown in Table 2 that the surface plasma treatment improved the adhesion of the resin and biological reinforcing fabric. A significant fall of the distance between the untreated and the plasma treated biological reinforcing fabrics is visible from the experiment results. More significant fall of the distance was at the cotton, up of 75% already at 15 s. The fall was milder at the flax.

**Table 2.** Impact of surface plasma treatment on distance between biological reinforcing fabrics (warp) and resin

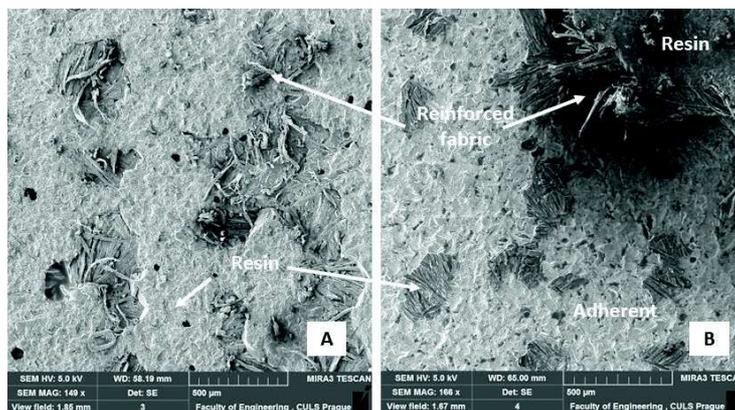
Time of effect of plasma treating of biological material surface (s)	0	15	30	60	90
Cotton Mean ( $\mu\text{m}$ )	1.16	0.29	0.26	0.20	0.15
Standard deviation ( $\mu\text{m}$ )	0.86	0.15	0.09	0.12	0.10
Variation coefficient (%)	74.51	51.72	35.75	60.00	65.65
Flax Mean ( $\mu\text{m}$ )	0.98	0.68	0.46	0.25	0.23
Standard deviation ( $\mu\text{m}$ )	0.43	0.34	0.20	0.10	0.08
Variation coefficient (%)	44.26	50.00	43.73	40.00	33.58

The adhesive type of the adhesive bond failure was at the resin. Adhesive bonds with the reinforcing phase were of adhesive (85–95%) – cohesive (5–15%) type (Fig. 5, A & 6, A). It was proved by the SEM that the resin was broken away from the reinforcing fabric at the adhesive bond destruction (Fig. 5, A & 6, A). The cohesive fracture surface was increased up to 80% after reinforcing fabric surface treating by the

plasma (Fig. 5, B & 6, B). This caused much better strength increase of the adhesive bond reinforced with the reinforcing fabrics from cotton. The strength increased by 22.5%. There was no significant strength increase of adhesive bonds reinforced with the flax when they were treated by the plasma. The strength was increased only by 1.5%. However there was the adhesive material deformation when adhesive bonds were weighted, which caused peeling off that consequently causes the formation of the fracture surface. It is obvious from the cohesive/adhesive fracture surface that there is destruction in the adhesive layer (Figs 5, B & 6, B). The destruction in the adhesive layer is caused by a bending moment at the deformation of the adhesive bonded material. This state is caused at thinner adhesive bonded material above all.



**Figure 5.** SEM images of fracture surface of adhesive bond reinforced with cotton (secondary electrons): A: Adhesive/cohesive failure – reinforcing fabric without plasma treatment MAG 105 x, B: Cohesive/adhesive failure – reinforcing fabric treated by plasma for over then 90 s MAG 112 x.



**Figure 6.** SEM images of fracture surface of adhesive bond reinforced with flax (secondary electrons): A: Adhesive/cohesive failure – reinforcing fabric without plasma treatment MAG 149 x, B: Cohesive/adhesive failure – reinforcing fabric treated by plasma for over then 90 s MAG 166 x.

A positive influence on the adhesive bond strength was proved by using the biological reinforcement. This positive effect was increased by the plasma treatment of the cotton fabric surface. Analogous effect was observed also at synthetic fibres (Müller & Cidlina, 2015; Müller et al., 2016; Zavrtašek et al., 2016; Zavrtašek & Müller, 2016a). Results proved the statement about the positive effect of natural and synthetic fabrics in the adhesive layout. SEM analysis proved worse wetting of the biological reinforcement without previous modification (flax, cotton).

## CONCLUSIONS

The experiment results proved a benefit of the reinforcing biological fabric in the layer of the adhesive. The adhesive bond strength was increased of 15.5% at the reinforcing layer based on the cotton and up of 43.2% at the reinforcing layer based on the flax. The reinforcing of the adhesive bond by the layer of the flax and the cotton showed itself in the positive way. However, the biological reinforcement was not fully wetted with structural epoxy adhesive, which did not fundamentally influence the adhesive bond quality. A fracture surface of the adhesive and the adhesive reinforced with the fabric was of the adhesive type, i.e. the strength of the adhesive was not fully utilized. The fracture surface changed when using the plasma treating of the fabric surface. The fracture surface was of the cohesive/adhesive type with prevailing cohesive failure. Ca. 20% increase of the adhesive bond strength at the reinforcement based on the cotton is connected with it. The plasma surface treatment of biological fibres improved the adhesion i.e. the distance between the warp and resin was decreased significantly.

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