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EFFECT OF SALINE ENVIRONMENT ON MECHANICAL PROPERTIES OF STRUCTURAL ADHESIVE BONDS

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Abstract

MÜLLER MIROSLAV. 2016. Effect of Saline Environment on Mechanical Properties of Structural Adhesive Bonds. *Acta Universitatis Agriculturae et Silviculturae Mendelianae Brunensis,* 64(5): 1609–1617.

This study brings new pieces of knowledge about a utilization of an inorganic filler in an area of steel adhesive bonds exposed to a degradation environment. The filler in the form of glass beads with a fraction size 90 ± 20 µm was used within the research. The aim of the research was to evaluate an influence of the degradation environment on a strength of structural two-component epoxy adhesives and a composite material. A preparation of adhesive bonds and a process of testing of the adhesive bonds were in accordance with the modified standard ČSN EN 1465. The degradation environment in a form of 5 % saline solution was used within this experiment. Adhesive bonded testing samples were subjected to a cyclic loading of the saline solution. The adhesive bonds with the filler reached up to 16 % higher adhesive bond strength than the unfilled adhesive bonds. The bonds adhesive bonded with the tested composite adhesive better resisted to the degradation process of ca. 9 %. The cyclic exposure, i.e. dipping of the testing samples into the saline solution and consequent drying significantly decreases the strength of the adhesive bond (up to 67 % in 6 weeks).

Keywords: adhesive bond strength, glass beads, filler, saline solution, scanning electron microscopy (SEM), structural two-component epoxy, steel

INTRODUCTION

An adhesive bonding technology is a prospective bonding technology of diverse materials. The adhesive bond is a complex of three layers – an adhesive bonded material (called adherent), an adhesive layer and a cohesive layer (Müller and Valášek, 2012; Müller, 2016). All three layers are significantly influenced by degradation processes, which cause a decrease in the strength of the adhesive bond and its service life. The degradation processes progressively deteriorate parameters of the adhesive bond (Müller and Herák, 2013; Cidlina *et al.* 2014). This fact shows the topicality of given problematics at simultaneously taking into consideration an increasing trend of a utilization of the adhesive bonding technology. A foreseeing or alternatively an elimination of a possibility of limit states is mainly dependent on an understanding of the process causing this state. Results of laboratory tests enable an analysis of a possible application of adhesive bonds (Müller, 2016).

Conclusions from the practice bring first distrust in the adhesive bonding technology. Frequent questions are: Will it stick together? What about demands for a technological discipline? What about a degradation, ageing? Is it better to use traditional technologies? Will we be reliable for consumers in a case of problems?

The degradation can be diagnosed on a basis of a change of a colour, a change of a state, a separation of sub-components, a decrease of a stress at which a failure in the bond started and a change of a mechanism of the bond failure (Messler, 2004; Crocombe, 1997; Doyle and Pethrick, 2009; Sargent, 2005; Müller *et al.*, 2013). Most of structural bonds come into being by connecting of a steel material. Adhesive bonds achieve good results with minimum expenses, e.g. for a treatment of an adhesive bonded surface, a choice of an adhesive etc. Their disadvantage, e.g. in a relation to aluminium alloys, is their lower resistance to a corrosion. The degradation is caused by the environment which affects the adhesive bonds. A salinity of the environment is a significant degradation element (Messler, 2004; Comyn, 1983; Müller, 2013). The adhesive bonds commonly meet the salinity e.g. at an operation of traffic means in winter months. Sources of the salinity are mainly sea environments and gritting defrosting preparations (Müller, 2016; Černý and Filípek, 2011; Černý *et al.*, 2007; Votava, 2013). A moisture of the environment in a combination e.g. with the salinity is another significant factor (Comyn, 1983). The research results proved an essentiality of a cyclic loading of the degradation environment which considerably accelerates the degradation process (Comyn, 1983; Messler, 2004; Müller, 2013). It manifests an accelerated loss of the adhesive bond strength (Müller *et al.* 2014; Müller, 2016). The research results present a huge decrease of the adhesive bond strength owing to the corrosion at exposing to effects of the increased salinity (Pereira, 2013).

Polymer materials are a significant group of technical materials. One of important requirements is that the polymer materials have to be of a sufficient resistance to the degradation (Müller, 2016). Namely in the area of connecting, i.e. a use of adhesives based on reactoplastics (two-component epoxy resins), this technology is limited by the degradation aspect. Two-component epoxy resins are widely used in joining structural components in the automotive and agronomy industry (Müller *et al.*, 2015).

Composite materials are promising structural components (Müller, 2016; Valášek, 2014). Composite materials comprise one or more discrete phases stored in the continuous phase (Müller *et al.*, 2015, Ruggiero *et al.* 2015; Valášek, 2014). Epoxies which are used as matrices for polymer composites based on reactoplastics (e.g. structural two-component epoxy) tend to similar trends (Müller *et al.*, 2015, Ruggiero *et al.* 2015; Valášek, 2014). A substantial change in mechanical properties of the resultant composite can be achieved by adding an optimum volume of a filler i.e. a reinforcement into the reactoplastics epoxy-based matrix (Ramazan *et al.*, 2008). Optimum utility properties of these composites are limited primarily by a risk of a cohesive damage caused by improper concentrations and a material of the filler. The filler of composite materials is different Al_2O_3, SiC, glass beads, minerals, various metals, rubber particles. Mechanical properties of polymer composites strongly depend on a particle size (Shao-Yun *et al.*, 2008, Müller *et al.*, 2015). The effect of the particle size on a mechanical performance of composites has been addressed by many authors. Authors (Leidner and Woodhams, 1974; Cho *et al.*, 2006; Grent and Park, 1984) demonstrated that the tensile strength of glass beads/polyester composites increased as the size of glass beads decreased.

The aim of this research was an evaluation of the influence of the degradation environment on the strength of structural two-component epoxy adhesives and a composite material.

MATERIALS AND METHODS

Adhesive bonds and a process of their testing were in accordance with the modified standard CSN EN 1465 and CSN EN ISO 9142 / Cycle D. 13 – Dipping into saline solution and drying. The degradation influence on the structural two-component adhesive (CHS Epoxy 1200) and the composite system was observed within the research. The composite system was created with the matrix (CHS Epoxy 1200) and the filler (glass beads B159).

Laboratory tests were performed using the standardized test specimens made according to the standard CSN EN 1465 (dimensions 100 ± 0.25 × 25 ± 0.25 × 1.5 ± 0.1 mm and lapped length of 12.5 ± 0.25 mm) from the carbon steel S235J0.

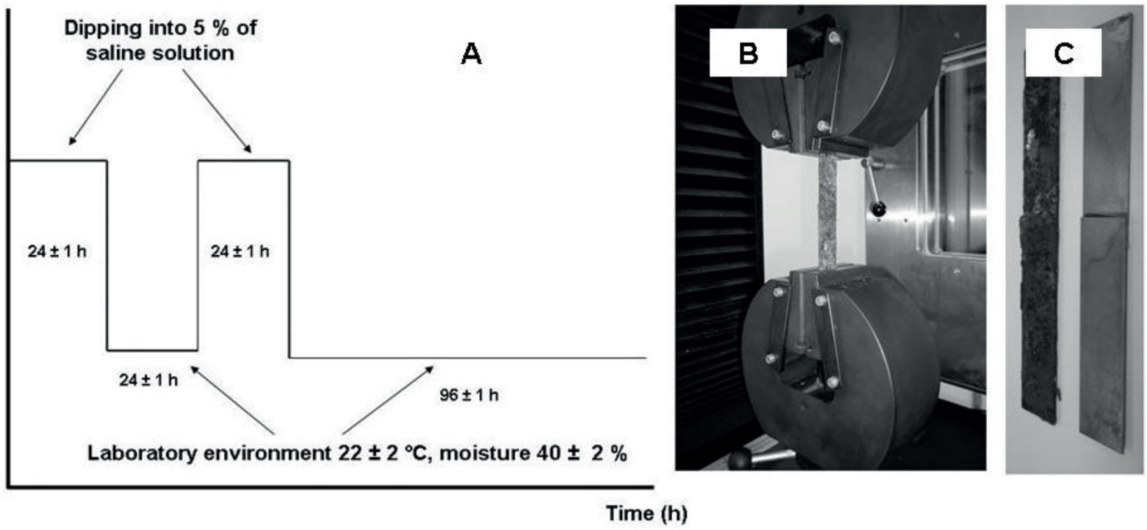
The essence of the test CSN EN 1465 is the determination of the strength by the shear loading of the simple overlapped adhesive bond between solid adherents which are loaded by the tensile force acting parallelly with an area of the conglutination and the main axis of the sample until the breach of the sample. The result of the laboratory test is the force measured at the breach of the adhesive bond.

A mixture of the adhesive was prepared by mixing of a part A (a resin) and a part B (a hardener) in a given ratio. The filler was added in the required ratio after mixing parts A (epoxy resin) and B (hardened P11 based on diethylentriamin). The determination of the concentration of the sub-components was expressed using a weight relative to 100 g of the matrix (the two-component adhesive). Weight ratios were chosen with a respect to a practical application when the filler is mixed mainly on the basis of weight ratios. An optimum filler concentration owing to the adhesive bond strength was determined in the first part of the research. The filler was added into the matrix CHS Epoxy 1200 (structural two-component reactoplastics resin) in the ratio of 10, 25, 50, 100, 150, 200 and 250 g (glass beads B159, a fraction size 90 ± 20 μm) to 100 g of matrix. The maximum filler value 250 g was chosen because of reaching a saturation of the matrix with the filler. It was ascertained that the highest adhesive bond strength was reached at the filler concentration 150 g, so the concentration 150 g of the filler was chosen for the research on the influence of the degradation environment. This composite corresponds to the weight concentration in the ratio 60 wt % of the particle filler of the glass beads (B159) and 40 wt % of the adhesive.

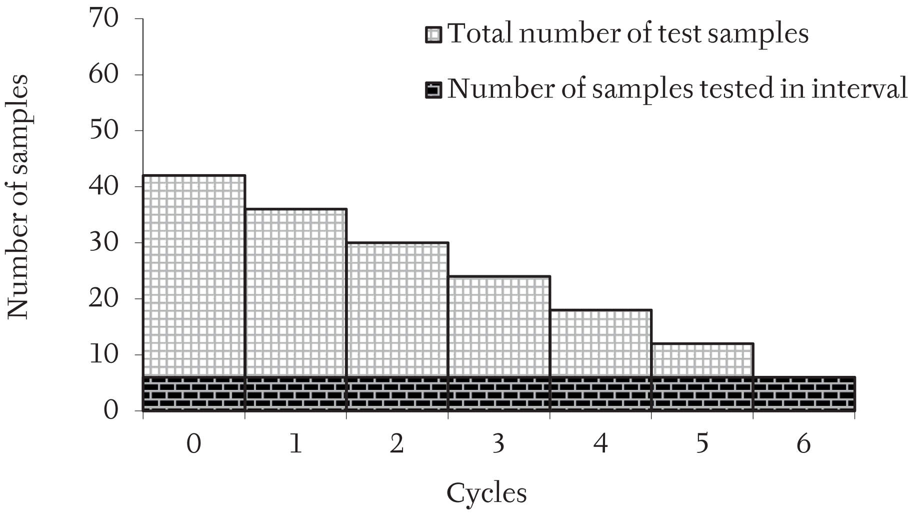
A surface treatment is essential not only in the area of the adhesive bonding technology (Müller, 2014; Novák, 2012; Dobranský *et al.*, 2015). The surface of the carbon steel S235J0 determined for the bonding was mechanically and chemically treated. The mechanical treatment consisted in a grit blasting of the bonded area by the Garnet MESH 80. The chemical cleaning consisted in removing impurities in a bath of Acetone. Roughness parameters Ra and Rz were measured on the surface of bonded adherents, in 5 partial ssections in the total trajectory 4 mm. Roughness parameters were measured with a portable profilometer Mitutoyo Surftest 301. A limit wavelength of the cut-off was set as 0.8 mm. The surface roughness parameters were measured in accordance with the standard CSN EN ISO 4288.

The adhesive was evenly applied on one bonded surface. The adhesive bonds were fixed with a weight of 750 g after applying second bonded part. The lapping was according to the standard CSN EN 1465 12.5 ± 0.25 mm. Adhesive bonds were cured for 72 ± 5 hours with a temperature 22 ± 2 °C. The reason was reaching the full strength of the adhesive bond and a minimization of the influence of the secondary hardening. Subsequently the adhesive bonds were loaded with cyclical impact of the degradation environment – saline solution.

The description of the degradation process influencing a safety of structural adhesive bonds was based on the modification of a regulation for a selection of laboratory conditions of aging for the evaluation of adhesive bonds. Chosen time periods of the exposition in the degradation environment were determined on the basis of fulfilling general ageing conditions, which are stated by the standard CSN EN ISO 9142



1: *Destructive testing of adhesive bond sample*
A – Schema of cyclic degradation of adhesive bonds, i.e. dipping into saline solution and drying, B – 6th cycle, testing machine, C – test samples



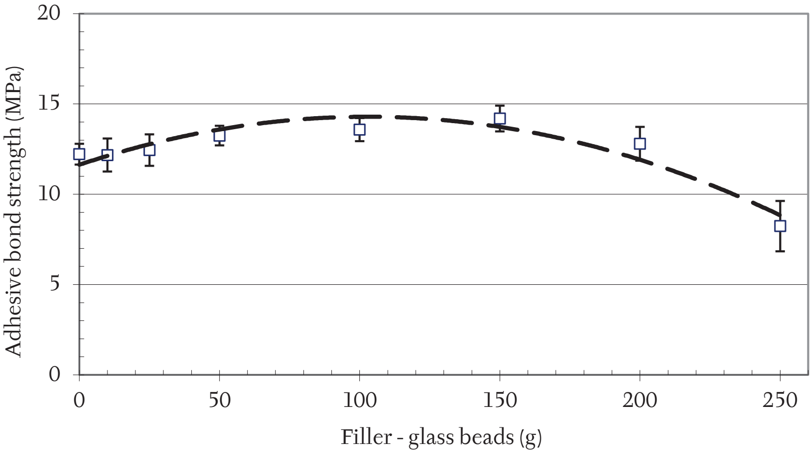
2: *Testing diagram – taking samples for destructive testing on universal testing machine LabTech5.50ST*

(Adhesives – Guide to the selection of standard laboratory ageing conditions for testing bonded joints).

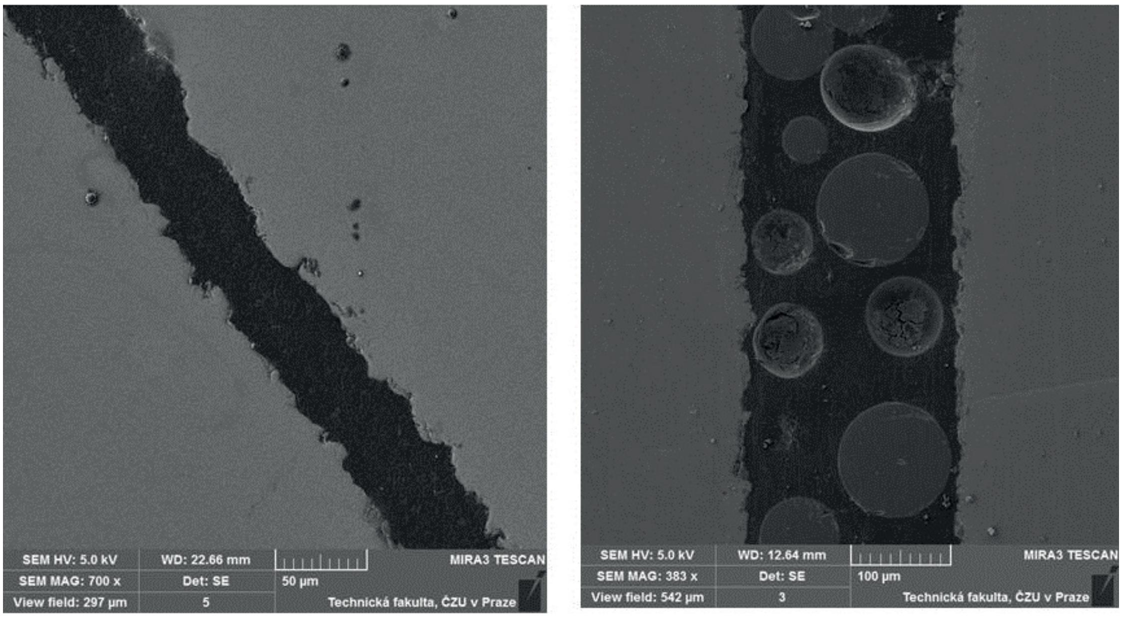
The standard CSN EN ISO 9142 describes laboratory conditions of the aging under which the adhesive bonds can be exposed to external influences. The purpose is a determination of required properties of the adhesive bond exposed to the external environment. The cycle D.13 (i.e. an immersion into to the saline solution and drying) was used within the research. The degradation environment in a form of 5 % saline solution was used. That was 5 mass portions of sodium chloride which was dissolved in 95 mass portions of water. The saline solution was mixed before putting test samples into it. Adhesive bonded test samples were subjected to the cyclical loading of the saline solution. One cycle consisted of the dipping into the saline solution and drying of the test samples which is evident from Fig. 1. The destructive testing of 6 samples was performed after the finished cycle. The remaining test samples were exposed to above described cyclical loading of the degradation environment in the form of the saline solution.

Test samples which were not exposed to the degradation environment in the form of the saline solution were used as a comparing standard. Fig. 2 shows the schema of experiments, i.e. a gradual consumption of test samples after each cycle 1 to 6. Degradation processes caused by the saline solution which influence the adhesive bond strength were continuously evaluated after finishing each cycle. Six test samples were tested in each series.

Laboratory tests were performed using the universal tensile strength testing machine LABTest 5.50ST (a sensing unit AST type KAF 50 kN, an evaluating software Test&Motion) – Fig. 1.



3: *Influence of filler concentration on adhesive bond strength (adherent S235 J0)*



4: *SEM images of microcracks in boundary adherent S235 J0 / adhesive (left), composite adhesive (right), secondary electrons*

The loading speed of the deformation corresponded to 5 mm/min. The failure type according to ISO 10365 was determined at the adhesive bonds.

The standard ISO 10365 stated a labelling of main types of the adhesive bond failure and it exemplifies the failure type by means of pictures. It can be used for all mechanical tests of adhesive bonds without a respect on the nature of the adherents and the adhesive creating the adhesive bond. The labelling of the adhesive bond failure type is used at the classification of the failure type for purpose of better assessment of the mechanical test result of the adhesive bond which is usually expressed by the quantitatively measured value.

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. An evaluation of the shape and the dimension 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. 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 ($p < 0.05$). For the correct evaluation it is also important to determine the determination index R^2. It is the problem of the correlation analysis. The values of the determination index can be from 0 to 1. So far as R^2 equals to 1, there is a perfect correlation in this sample (so there is no difference between a calculation and real values).

RESULTS

The surface roughness of the adhesive bonded material S235J0 after the mechanical treatment by a grit blasting and the chemical treatment was in the direction parallel to the loading force at the destructive testing of the adhesive bonds Ra 2.00 ± 0.16 μm, Rz 11.93 ± 0.89 μm.

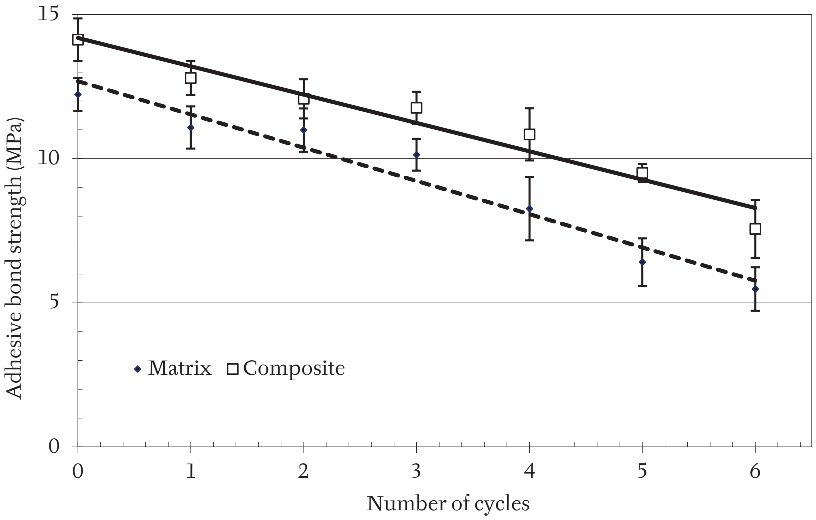
It is obvious from the adhesive bond strength results that adding the filler changes the adhesive bond strength (Fig. 3). The highest adhesive bond strength increase was at the adhesive bond with the adhesive in the form of the composite (150 g of the filler : 100 g of the matrix). The adhesive bond strength increase was 16.2 %.

It is possible to say in terms of the statistical testing of the influence of various filler concentrations on the adhesive bond strength that the concentrations are statistically non-homogeneous groups. The hypothesis H_0 was not certified, i.e. there is a difference in the adhesive bond strength in 0.05 significance level among single tested materials, i.e. the matrix and various concentrations of the filler ($p = 0.0000$).

It was used 150 g of the filler (glass beads B159) to 100 g of matrix for the research on the influence of the degradation environment. The polynomial second order function presented in Fig. 3 reaches a strong dependence of the adhesive bond strength on the filler ($R^2 = 0.89$).

Using the electron microscopy within the experimental research a presence of cracks in the boundary adherent / adhesive was not proved (Fig. 4). The experiment results proved a good wettability between the adhesive layer and the adherent. The research results proved a good wettability between the adhesive and the filler.

The results of the adhesive bond strength in dependence on the cyclical exposure to the degradation environment in the form of 5 % saline solution are visible in Fig. 5. The adhesive



5: *Influence of cyclic exposure to degradation environment of adhesive bond (adherent S235 J0, composite adhesive 150 g of glass beads B159 : 100 g of matrix)* bond strength of the structural two-component epoxy (matrix) was 12.22 ± 0.58 MPa. The strength decreased to 5.48 ± 0.75 MPa after six degradation cycles. The decrease of the adhesive bond strength was 55.2 %. The adhesive bond strength of the composite adhesive (matrix + filler) was 14.13 ± 0.74 MPa. The strength decreased to 7.56 ± 1.00 MPa after six degradation cycles. The decrease of the strength of the adhesive bond was 46.5 %. Milder decrease of the strength of the bond adhesive bonded with the composite mixture occurred owing to the degradation. The difference of the decrease of the adhesive bond strength was ca. 9 %.

The coefficient of the variation from 0 to 3rd cycle was not higher than 7 %. The coefficient of the variation from 4th to 6th cycle was in the interval from 8 to 14 %. Higher values of the coefficient of the variation give higher dispersion of results. More considerable diffusion of the liquid contaminant into the adhesive bonds significantly occurred from 4th cycle. In terms of the statistical testing of the influence of particular cycles of dipping into the saline solution and consequent drying, it is possible to say, that the cycles are statistically non-homogeneous groups, i.e. there is a difference among tested cycles.

The hypothesis H_0 was not confirmed for the structural two-component epoxy $p = 0.000$ and the composite adhesive $p = 0.0000$. There is a difference in the adhesive bond strength in the level of the significance 0.05 among particular tested cycles.

In terms of the statistical testing (T-test, $\alpha = 0.05$) of the influence of single cycles of dipping into the saline solution and drying (0th cycle $p = 0.0011$, 1st cycle $p = 0.0022$, 2nd cycle $p = 0.0379$, 3rd cycle $p = 0.0009$, 4th cycle $p = 0.0024$, 5th cycle $p = 0.0001$ and 6th cycle $p = 0.0040$) on the adhesive bond strength it is possible to say that the cycles are statistically non-homogeneous groups, i.e. there is a difference among adhesive bonds bonded with the structural two-component epoxy adhesive and the composite adhesive.

The trend of the adhesive bond strength decrease (Fig. 5) was possible to describe with a linear function (1) and (2). The linear functions stated in Fig. 5 reach strong dependence of the adhesive bond strength on the number of cycles (structural two-component adhesive $R^2_{STCA} = 0.95$ and composite adhesive $R^2_{CA} = 0.96$).

$$\tau_{STCA} = -1.1535.NC + 12.687 \tag{1}$$

$$\tau_{CA} = -0.9831.NC + 14.187 \tag{2}$$

Where:

τ_{STCA} – strength of adhesive bond – structural two-component adhesive (MPa)

τ_{CA} – strength of adhesive bond-composite adhesive (MPa)

NC – number of cycles

It was ascertained from the linear function (1) that the adhesive bonds bonded with the structural two-component adhesive ruined after 11 cycles, i.e. after 11 weeks of the exposition to the cyclic degradation. It was ascertained from the linear function (2) that the adhesive bonds bonded with the composite adhesive ruined after 15 cycles, i.e. after 15 weeks of the exposition to the cyclic degradation.

The bonds adhesive bonded with the structural two-component adhesive showed an adhesive failure type. The bonds adhesive bonded with the composite adhesive filled with the glass beads showed an adhesive-cohesive failure type of the adhesive bond (Fig. 6).

A good interaction of the filler in the form of the glass beads with the matrix in the form of the epoxy was proved using the electron microscopy (Fig. 6) within the experimental research. The residues of the matrix are visible on the surface of the glass beads filler. A good wettability of this filler follows from that.

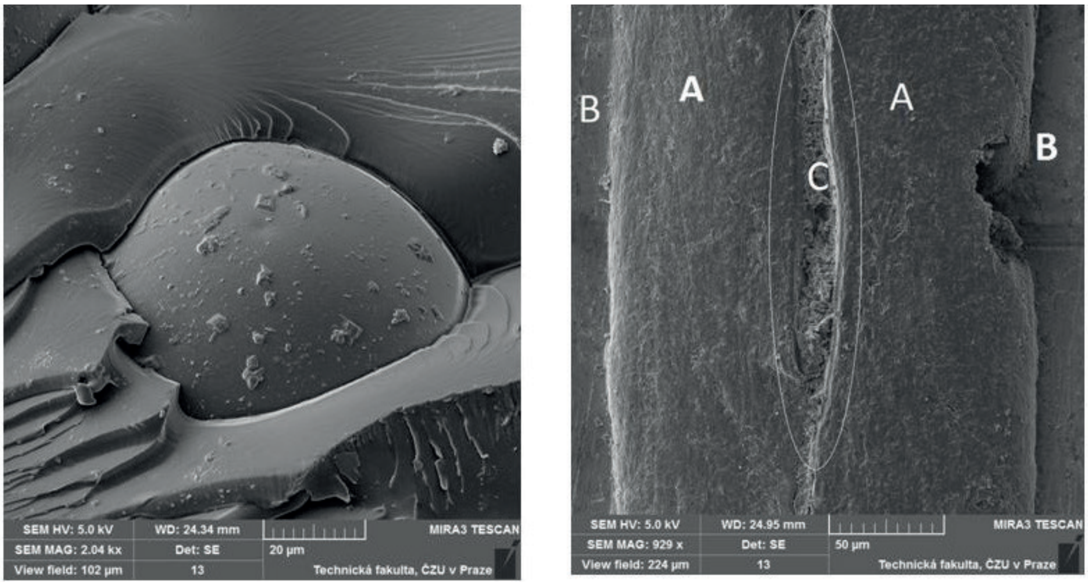
The surface of the adhesive bonded material and own adhesive bond was covered with a significant layer of corrosive products. Using the electron microscopy within the experimental research it was proved that the spontaneous initiation of the failure of the adhesive layer occurred at 4th till 6th cycle (Fig. 6). This initiation of the fracture surface occurred in the edges of the adhesive bond. Fig. 6 shows a good interaction of the adhesive and the adherent with the adhesive bonded material. It did not come to a under corrosion of the adhesive layer.

The research results proved a positive influence of adding of the glass beads filler on the adhesive bond strength, also at the degradation. Adding of the filler also considerably decreased the costs per the adhesive bond, up of more than 50 %. The price of the filler is considerably lower than the matrix, i.e. the structural two-component epoxy adhesive.

When adding the particle filler in the form of the glass beads B159 with the fraction size 90 ± 20 (the spherical shape) at the tensile shear strength test the positive influence occurred which corresponds with the conclusions of the study of authors who deduced the conclusions that adding the glass microballs with the dimension till 120 µm on average increased the tensile properties of created particle composites (Sanchez *et al.* 2007; Valášek, 2015).

DISCUSSION

The adhesive (matrix) layer thickness was measured as 39.12 ± 7.74 µm. The composite adhesive (matrix and filler) layer thickness was measured as 220.04 ± 12.64 µm. The composite materials showed greater adhesive layer thickness of 562.5 %. The optimum shear strength was reached at the two-component structural epoxy adhesive



6: *Fracture surface of adhesive bond bonded with composite adhesive*
perfect wetting of filler with resin (left), SEM images of initiation of destruction of adhesive layer caused by degradation agents: A – layer of adhesive, B – adhesive bonded material, C – initiation of destruction of layer of adhesive (right), secondary electrons

in the interval of the adhesive layer thickness 0.1 to 0.25 mm (Müller and Valášek, 2013).

From the results of the experiment it is possible to agree with the statement, that epoxy adhesives preserve their adhesive bond strength also at high concentrations of the filler (Ramazan *et al.*, 2008). The highest adhesive bond strength was reached at 60 wt. % of the filler concentration. This concentration corresponds to 150 g of the filler. A strong interaction between adhesive and particles is evident.

The assumption about a negative influence of the filler on the tensile strength was not confirmed. Cho *et al.* state that there is a decrease in the strength of the composite with increasing volume of filler particles (Cho *et al.*, 2006). This conclusion was not certified. It is possible to agree with the statement that inorganic fillers improve mechanical properties of the polymer (Farrah *et al.*, 2008). The adhesive bond in this case.

The conclusions stated by Müller *et al.* and Crocombe that the degradation of adhesive bonds depends on an adhesive type and a degradation environment were confirmed by experiments (Crocombe, 1997; Müller *et al.*, 2014).

The results of experiments stated in Fig. 6 confirm the statement of Kinloch and Court *et al.* about negative effects of different environments (Kinloch, 1987; Court *et al.*, 2001).

CONCLUSION

From the results of the experiments aimed on the evaluation of the degradation process of the structural epoxy resin (matrix) and materials on its basis, i.e. composites, it is possible to state the following conclusions:

- The highest adhesive bond strength increase was at the adhesive bond with the adhesive in the form of the composite (150 g of the filler : 100 g of the matrix). The adhesive bond strength increase was 16.2 %.
- On the basis of the evaluation of performed experiments it is possible to say, that the resulting strength of adhesive bonds decreases in the degradation environment.
- The cyclic exposure, i.e. dipping of the test samples into the saline solution and consequent drying significantly decreases the strength of the adhesive bond (up to 67 % in 6 weeks).
- The adhesive bonds bonded with tested composite adhesive better resisted to the degradation process of ca. 9%.
- Adding the filler changed the fracture surface. The number of cycles at the degradation did not change the fracture surface.

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