

# Analysis of production parameters of single-lap bonds adhesive bonded with composites based on aluminium filler

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## Abstract

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In the area of bonding of sheets of metals, mainly in construction of transport and agricultural machines, single-lap bonds are used. In manufacturing corporations focused on bonding of the metal sheets the technologies such as riveting, welding and adhesive bonding are particularly used. These methods are frequently combined. The aim of the research was the evaluation of lap length of alloy AlCu4Mg adhesively bonded using two component epoxy adhesive, which is commonly used in construction of machines and its modification based in addition of filler in form of aluminium microparticles. The secondary aim of the research was to ascertain the influence of microparticle volume of aluminium filler on mechanical properties of polymer particle composite. Strength of adhesively bonded joint depends on the thickness of the bonded material. Strength of the adhesively bonded joint is dependent on the lapping length of adhesively bonded material. The highest values of strength of adhesively bonded joint were reached with the coefficient of the proportional length  $0.27 \pm 0.01$ . The assumption about negative effect of filler on tensile strength during the experiments was not confirmed.

**Keywords:** AlCu4Mg; adhesive bond strength; lapping length; sheet of metal; particle reinforced composite; scanning electron microscopy (SEM)

The adhesive bonding technology is a promising method of bonding (MÜLLER 2013b, MÜLLER et al. 2013). Aluminium alloys belong to the significant technical materials, that are suitable for the adhesive bonding. The technology of adhesive bonding is inexpensive and fast method of bonding and it is used almost in all areas (PEREIRA et al. 2010). Adhesive bonding of aluminium and its alloys was introduced in England in 1940 (PEREIRA et al. 2010).

The adhesive bonding strengthens its position in a number of agricultural machinery and tools. It can be mentioned e.g. the cooperation of the com-

panies Henkel and New Holland in the area of the agriculture (MÜLLER, VALÁŠEK 2013a). Single-lap adhesive bonds are used in the area of bonding of sheets of metals, e.g. in a construction of agricultural machines (MÜLLER 2013a). Another example of the adhesive bonding application in the agriculture is an adhesive bonding of a breakwater in an agricultural fertilizer sprayer and holders for fixing of plough shares on a plough body (BENEŠ 2012; MÜLLER 2013). A decrease of the mass of the construction is required in case of the cars and agricultural machines etc. (BORSELLINO et al. 2009; MÜL-

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LER 2013a). This requirement is secured namely by a use of aluminium materials in the constructions of machines. The aluminium constructions are lighter than traditional steel ones. The aluminium is of worse mechanical properties than the steel (BORSELLINO et al. 2009). However, the aluminium constructions are comparable with the steel ones thanks to special constructional adjustments and with a respect to the bonding methods (BORSELLINO et al. 2009). A classical welding is a typical method for the bonding of steel materials. It is difficult for the bonding of aluminium alloys (BORSELLINO et al. 2009). The adhesive bonding technology is an alternative method for the connecting of the aluminium alloys. The use of the adhesive bonding technology in the construction of transport means and agricultural machines secures stiffness comparable with mechanical fasteners or spot-welds. Further, the adhesive bonding technology increases an energy absorption reducing a noise and vibrations (BORSELLINO et al. 2009).

Adhesively bonded single-lap joints are from point of manufacturing view less expensive and in many cases they satisfy strength requirement, while in situations, where there is a requirement of higher strength of adhesively bonded joints, the special construction modifications are applied. These modifications however, require higher expenses and they are more difficult from the technology point of view (VALÁŠEK, MÜLLER 2015).

Considerable part of the research on single-lap adhesive bonds is focused on geometrical design of adhesive bonds, thickness of adhesive layer and on mechanical properties of adherents (MÜLLER, HERÁK 2010; MÜLLER, VALÁŠEK 2013b).

Geometrical parameters of adhesively bonded joints are significant for determination of constructional parameters and economical expenses (HERÁK et al. 2009). When shorter length of lap is chosen, the failure occurs in adhesively bonded joints and the max. bearing capacity of bonded material is not used (MÜLLER, HERÁK 2010). On the contrary, when the lap length is longer than the optimum length, the disruption occurs in the bonded material. Simultaneously total weight of adhesively bonded set increases (MÜLLER, HERÁK 2010).

One of the possibilities to achieve the optimum constructional modification is increasing the thickness of bonded material. As a consequence of smaller deformation of thicker bonded material the adhesive layer is less deformed (MÜLLER, HERÁK 2010).

Adherence and cohesiveness themselves are not able to enable long-term service life of bond due to other outer factors. One of the outer factors is action of outer load, therefore it is important to pay attention to all phases of adhesively bonded joint from design, i.e. geometrical parameters of the joint, with exclusion or minimization of peel loading or bending, to which they have low extent of resistance (EITNER, RENDLER 2014).

When the adhesively bonded joints are created, the negative part is a significant consumption of expensive adhesives. One option is adding the filler into the adhesive. Other options are different construction modifications. OLIA and ROSSETTOS (1996) introduced an analytical study of gap effectiveness in single-lap joints. A target of their experiments was the minimization of expenses of adhesives by using longer lengths of lap. Their results showed that presence of gaps created an origin of significant peeling forces on spare ends of gaps. There could be the influence of strength up to 25% (OLIA, ROSSETTOS 1996).

With respect to the construction, the length optimization of adhesively bonded joint is problematic. The reason is, that with the increasing length of lap also economical expenses increase. Adhesively bonded joint becomes ineffective. The consumption of adhesive and bonded material increases, while strength of adhesive bond is not increasing (LOCTITE 1998).

Mutual interaction of the length and the width of the lap on the strength of bond was compared by EBNEAJJAD and LANDROCK (2014). They presented straight linear proportion of increasing width on strength of adhesively bonded joint. The same proportion is not valid for the length.

The treatment of the surface is also a significant factor influencing the manufacturing expenses (NOVÁK 2012; HRICOVA 2014). Nevertheless, recent researches show, that effectiveness of mechanical and chemical treatments is not that fundamental for the strength of the adhesively bonded joint (BOCKEN et al. 2002). The solution is particularly the development of „new” adhesives.

The aim of the research was the evaluation of lap length of alloy AlCu4Mg adhesively bonded using two component epoxy adhesive, which is commonly used in construction of machines and its modification based in addition of filler in form of aluminium microparticles.

Secondary aim of the research was to ascertain the influence of microparticle volume of aluminium filler on mechanical properties of polymer particle

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composite. The object of performed experiments was polymer particle composite, whose continuous phase was in form of two component epoxy resin and the discontinuous phase (reinforcement particles) were microparticles of aluminium smaller than 45  $\mu\text{m}$ .

Adding the filler into reactoplastics matrix on the base of epoxies influences the mechanical properties of resulting composite (KIM, KHAMIS 2001; GALUSEK et al. 2007; SHAO et al. 2008; KEJVAL, MÜLLER 2013; VALÁŠEK 2015). Optimum utility properties of these composites are limited primarily by the risk of origin of the cohesive failure, which is caused by both, inappropriate concentration and material of the filler.

The synergic effect is not always achieved, i.e. significant improvement of mechanical properties. In some cases it is possible to achieve decrease of price of adhesive on the base of composite at keeping comparable properties with unfilled adhesive.

For instance there is a research of KAHRAMANA et al. (2008) who found that by adding the aluminium particles the strength of adhesively bonded joint increased. They used composite mixture on base of aluminium microparticles for the adhesive bonding.

High volume of filler can cause deterioration of mechanical properties of adhesive, i.e. composite material. KAHRAMANA et al. (2008) state, that strength of adhesive bond did not significantly decrease with adding the aluminium powder (0 to 50% vol.).

## MATERIAL AND METHODS

Standard ČSN EN 1465:2009 was followed when performing the experiments. Laboratory experiments were performed on normalized testing samples of alloy AlCu4Mg, prepared under standard ČSN EN 1465:2009 by cutting the metallurgical semi-finished product in form of metal sheet.

The testing samples without the mechanical treatment of the surface were used for the adhesive bonding. The adhesively bonded surface was chemically treated – degreased with Acetone before the process of adhesive bonding. The untreated samples were used due to minimizing the factors effecting the preparation of bonded surface. This trend is significant particularly in operations, where the automatization is implemented (NOVÁK 2012; HRICOVA 2014).

The object of performed experiments was polymer particle composite, whose continuous phase was in form of two component epoxy adhesive

ChS Epoxy 1200 (hardener P11 – Diethylentriamin) (Sincolor a.s., Zibohlav, Czech Republic) and discontinuous phase (reinforcement particle) were aluminium microparticles (ca. 90 % of particles were smaller than 45  $\mu\text{m}$ ).

The concentration of filler in the matrix was indicated by the volume fraction of filler  $v_p$ . Another possibility of indication is the expression of mass fraction of  $v_m$ . Mass fraction is not optimum due to the different densities of the matrix and filler. This difference is often diametrical. Concentration of filler in the matrix is considered as one of the most important parameters affecting the final properties of composite systems (VALÁŠEK 2014). The volume fraction of the filler is calculated according to Eq.(1). Epoxy density was 1.15 g/cm<sup>3</sup> and 2.7 g/cm<sup>3</sup> for the filler:

$$v_p = V_p / V_c \quad (1)$$

where:  $v_p$  – volume fraction of filler (-);  $V_p$  – volume of filler (cm<sup>3</sup>);  $V_c$  – volume of composite (cm<sup>3</sup>)

Determination of the concentration of the sub-components was expressed in volume percentages of 10, 15 and 20%.

The roughness parameters *Ra* and *Rz* were measured on the adherent's surface designated for adhesive bonding. Roughness parameters were measured with portable profilometer Mitutoyo SurfTest 301 (Mitutoyo, Michigan, USA). Boundary wave length of cut-off was placed to 0.8 mm. The measurement was performed on 150 places of 20 adherents Al-Cu4Mg. Three thicknesses of adhesively bonded adherent were tested 1.52 ± 0.02 mm (adherent A), 1.90 ± 0.03 mm (adherent B) and 3.01 ± 0.04 mm (adherent C). The lapping was according to the standard 9 ± 0.25 mm, 12 ± 0.25 mm, 24 ± 0.25 mm, 36 ± 0.25 mm and 48 ± 0.25 mm. An even thickness of the adhesive layer was reached by a constant pressure of 0.5 MPa.

The tested samples were kept with laboratory temperature 22 ± 2°C for 48 h after the fixation of adhesively bonded joint. After that the destructive testing followed.

The failure type according to ISO 10365:1995 was determined at the adhesives bonds. The tensile strength and the elongation test 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 (both Labortech s.r.o, Opava, Czech Republic). A speed of the deformation corresponded to 6 mm/min.

After the adhesive bond destruction the maximum force was read, the lapping of the surface was measured, the failure type according to ISO 10365:1995 was determined and the adhesive bond strength was calculated according to the Eq. (2). standard CSN EN 1465:2009:

$$\tau = F/(b \times l_u) \quad (2)$$

where:  $\tau$  – tensile shear strength (MPa);  $F$  – maximum force (N);  $b$  – lapping width (mm);  $l_u$  – lapping length (mm)

In order to increase the explanatory power following from the lap geometry of adhesively bonded joint it is possible to make a calculation of coefficient of proportional length. According to the Eq. (3) not only the lapping length but also the widths of the lapping are taken into the regard:

$$\lambda_p = l_u/b \quad (3)$$

where:  $\lambda_p$  – coefficient of proportional length (–);  $l_u$  – lapping length (mm);  $b$  – lapping width (mm)

Volume of impurities on the adhesively bonded material was determined on the base of weighing scales (Kern; Kern&Sohn GmbH, Balingen, Germany). Testing samples were weighed before and after chemical treatment in the acetone bath. Weight of the impurities was compared with the weight of the testing sample.

For the correct evaluation it is also important for the statement of the index of the determination  $R^2$ . It is the problem of the correlation analysis. The values of the determination index can range 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).

The tested sets were mutually compared using F-test from the point of view of the influence on mechanical properties. The zero hypothesis  $H_0$  presents the state when there is no statistically significant difference ( $P > 0.05$ ) among tested sets of data from their mean values point of view.

## RESULTS AND DISCUSSION

Using the profilograph SurfTest 301 (Mitutoyo, Michigan, USA) the following values were determined:  $Ra$   $0.37 \pm 0.08 \mu\text{m}$ ,  $Rz$   $2.41 \pm 0.44 \mu\text{m}$ .

Thickness of the adhesive layer was measured  $0.27 \pm 0.04$  mm. For two component epoxy adhe-

sives the thickness of adhesive layer reached the optimum of shear strength in the interval 0.1 to 0.25 mm (MÜLLER, VALÁŠEK 2013b).

Within this research the mechanical treatment of the surface was not used regarding to findings of TAMAI and ARATANIC (1972). They found, that under terms of certain limits the higher roughness of the samples caused a decrease of their wetting. Simple explanation is that projections of long elevation create the barriers, which prevent the drops spreading. The similar conclusions were found also by PEREIRA et al. (2010). They state that mechanical properties of untreated aluminium were better than properties of mechanically treated one. On the basis of these conclusions the testing samples of alloy AlCu4Mg without mechanical treatment of adhesively bonded surface were used for the research.

Testing samples prepared for adhesive bonding contained significant amount of impurities on their surface. It was observed, that they contain  $0.0202 \pm 0.0024\%$  of impurities per one testing sample. This percentage is related to the weight of testing sample. From the results it is obvious, that testing samples without chemical treatment of the surface contain significant amount of the impurities on their surface. The results of the testing focused on the evaluation of the influence of lap length, thicknesses of adherent and filler on the strength of adhesively bonded joint are visible in Fig 1.

From the results of this experiment it is apparent, that the strength of the adhesively bonded joint exponentially decreases with the lapping length.

One possibility of reaching the optimum construction modification is the increase of thickness of the bonded material. Another possibility is the increase of width of adhesively bonded joint. This construction modification, however, does not change the unevenness of the load distribution, which increases proportionally with the bond width (LANG, MALLICK 1999; MESSLER 2004; VALÁŠEK, MÜLLER 2015). The reason is the bending moment, which causes the tensile loading predominantly at the ends of the bond. The origin of peeling forces is subsequently a consequence of it.

Strength of the adhesively bonded joint was in the interval 0.85 to 9.34 MPa in the dependence on thickness of the bonded material, the filler volume and the lapping length. The highest strength of adhesively bonded joint was reached with the thickness of the adhesively bonded material marked as:

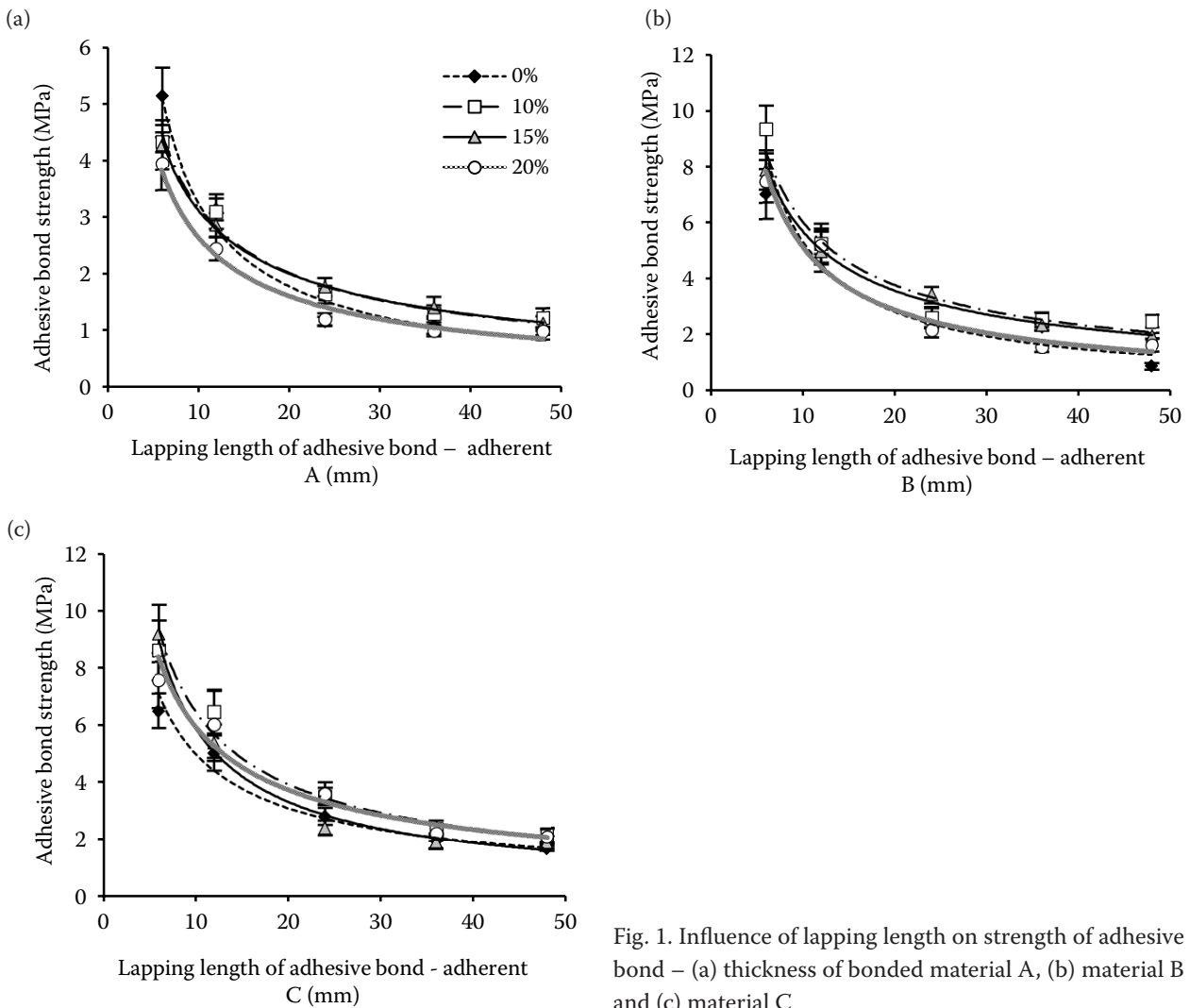


Fig. 1. Influence of lapping length on strength of adhesive bond – (a) thickness of bonded material A, (b) material B and (c) material C

- A ( $1.52 \pm 0.02$  mm)  $5.14 \pm 0.51$  MPa with filler concentration 0%,
- B ( $1.90 \pm 0.03$  mm)  $9.34 \pm 0.86$  MPa with filler concentration 10%,
- C ( $3.01 \pm 0.04$ )  $9.20 \pm 1.01$  MPa with filler concentration 15%.

From the statistical point of view of the testing of the influence of the different thickness of the adhesively bonded material it is possible to state, that these are statistically non-homogeneous groups, i.e. there is a difference between tested thicknesses of adhesively bonded materials A, B and C. For adhesively bonded joints bonded with two-component epoxy resin the values were  $P = 0.0165$  for concentration of filler 0%,  $P = 0.0003$  for concentration of filler 10%,  $P = 0.0018$  for concentration of filler 15%, and  $P = 0.00007$  for concentration 20%. Hypothesis  $H_0$  was not confirmed. i.e. there is a dif-

ference in the strength in the significance level 0.05 among particular tested thicknesses of adhesively bonded materials A, B and C.

With the respect to the statistical testing of the influence of different thicknesses of adhesively bonded material (compared individually B ( $1.90 \pm 0.03$  mm) and C ( $3.01 \pm 0.04$ )) it is possible to state, that these are statistically homogeneous groups, i.e. there is not difference between tested thicknesses of adhesively bonded materials B and C. For adhesively bonded joints using the two component epoxy resin containing 0% concentration of filler  $P$  was 0.9809, for filler concentration 10%  $P$  was 0.7851, for filler concentration 15%  $P$  was 0.9332, and for filler concentration 20%  $P$  was 0.2519. Hypothesis  $H_0$  was confirmed, i.e. there is not a difference in strength of adhesively bonded joints in the significance level 0.05 between par-

ticular tested thicknesses of adhesively bonded materials B and C.

From the above stated the significant conclusion for dimension of adhesive bond follows. With respect to strength of the adhesively bonded joint, there is a significant difference among thicknesses of the materials A ( $1.52 \pm 0.02$  mm), B ( $1.90 \pm 0.03$  mm) and C ( $3.01 \pm 0.04$ ). For tested material AlCu4Mg there is not significant increase of strength of adhesively bonded joint from the thickness 1.90 mm.

For tested materials B and C, i.e. thickness of adhesively bonded material ca. 2 to 3 mm, there was not evident any deformation of adhesively bonded material at the failure of adhesive layer. For tested material with thickness 1.5 mm the significant deformation was evident already before failure inside the adhesive layer.

Due to influence of deformation (bending moment) of adhesively bonded material A (ca. 1.5 mm) the failure of adhesively bonded joint occurred in the interface of adhesive and bonded material. Deformation of the bonded material shows negatively via peeling forces, which decrease the strength of the bonded set. Bonded material A evinced deformation  $1^{\circ}49' \pm 6'$  after destruction.

The analogous results were reached with construction steel when 1.5 and 3 mm thicknesses of bonded material were compared (VALÁŠEK, MÜLLER 2015).

On the basis of results and statistical survey the conclusions were confirmed, that in consequence of smaller deformation of thicker bonded material the adhesive layer is less deformed (MESSLER 2004; MÜLLER, VALÁŠEK 2013b).

From the viewpoint of the statistical survey of influence on different lapping lengths of bonded material it is possible to state, that these are statistically non-homogeneous groups, i.e. there is a difference among tested thicknesses of bonded materials A, B and C and filler concentrations. For all tested variants  $P$  was 0.0000. Hypothesis  $H_0$  was not confirmed, i.e. there is a difference in strength of adhesively bonded joint in the significance level 0.05 among particular tested lapping lengths and filler concentrations in form of aluminium microparticles.

From the viewpoint of statistical survey of influence on different concentration of aluminium microparticles it is possible to state, that these are statistically homogeneous groups, i.e. there is not difference among tested thicknesses of adhesively bonded materials A, B and C. For adhesively bond-

ed joints with material thicknesses A  $P = 0.6129$ , B  $P = 0.4810$ , and C  $P = 0.4461$ . Hypothesis  $H_0$  was confirmed, i.e. there is not difference in strength of adhesively bonded joint in the significance level 0.05 among particular filler concentrations 0, 10, 15 and 20%.

From the results of the experiment it is possible to agree with statement, that epoxy adhesives preserve their strength even with 50 % vol. of aluminium filler (KAHRAMANA et al. 2008). At the experiments concentrations up to 20% vol. percentage were tested. From the results of strength of adhesively bonded joint and also from the statistical survey it is evident, that by adding the filler there is not significant change in strength of adhesively bonded joint (KAHRAMANA et al. 2008).

Results of the experiments confirmed conclusions of KAHRAMANA et al. (2008), where they state, that there was a slight increase of strength of adhesively bonded joint of testing samples from aluminium alloy with adding the aluminium microparticles with size approximately 50  $\mu\text{m}$ . The aluminium microparticles with size less than 45  $\mu\text{m}$  were used within the experiment.

The assumption about negative influence of filler on tensile strength was not confirmed. CHO et al. (2006) state that with increasing volume of filler particles there is a decrease in strength of composite.

The highest values of strength of adhesively bonded joint were reached with coefficient of proportional length  $0.27 \pm 0.01$ . With increasing coefficient of proportional length there is a decrease of strength of adhesively bonded joint. The lowest values of strength of adhesively bonded joint were reached with coefficient of proportional length  $1.94 \pm 0.03$ .

The functions presented in Fig 1. are determined by equations in Table 1. A strong dependence is obvious from the values stated in Table 1.

Adhesively bonded joints evinced both, adhesive and special cohesive type of fracture surface. Adding the filler in form of aluminium microparticles there caused change in the failure. Therefore it is not possible to agree with statement of KAHRAMANA et al. (2008), that filled epoxy resin improved the adhesion to bonded surface, i.e. there was not cohesive failure of adhesively bonded joint.

Increase of percentage representation of special cohesive failure was arisen due to change of thickness of bonded material. The special cohesive failure of adhesively bonded joint was found more of frequently in adhesively bonded materials B and C.

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Table 1. Equations of functions – influence of lapping length ( $x$ ) on adhesive bond strength ( $y$ )

Adherent	Adhesive	Functional equations	$R^2$
A	adhesive	$y = 24.431x^{-0.8748}$	0.955
A	composite 10%	$y = 14.424x^{-0.6579}$	0.980
A	composite 15%	$y = 13.746x^{-0.6432}$	0.999
A	composite 20%	$y = 13.962x^{-0.7219}$	0.966
B	adhesive	$y = 43.357x^{-0.9131}$	0.879
B	composite 10%	$y = 28.524x^{-0.6763}$	0.928
B	composite 15%	$y = 26.467x^{-0.6694}$	0.994
B	composite 20%	$y = 34.419x^{-0.8281}$	0.959
C	adhesive	$y = 24.427x^{-0.689}$	0.978
C	composite 10%	$y = 33.769x^{-0.7174}$	0.980
C	composite 15%	$y = 39.196x^{-0.8235}$	0.968
C	composite 20%	$y = 27.653x^{-0.6677}$	0.962

 $R^2$  – determination index

For cohesive strength of own adhesive the semi-phase interface is important. Particularly considering the option of application of adhesively bonding technology as construction bonded element in the area of agricultural machines. There is a high risk of contamination of adhesive bond in the agro manufacturing field, for instance by fertilizers etc. (MÜLLER, VALÁŠEK 2012; MÜLLER 2013a). The diffusion of liquid contaminants into the adhesive occurs (MÜLLER 2013a). Liquid contaminants can disrupt phase interface between filler and epoxy resin.

The presence of cracks in the interface adherent/adhesive (Fig. 2a) was proved within the experimental research by means of the electron microscopy. The adhesive failure of adhesive bonds had its origin also in these cracks. It came to the decrease of the adhesive strength of the adhesive bond.

The presence of diversely large aluminium particles was proven by using the electron microscopy within experimental research (Fig. 2b). The microparticles had regular spherical shape. There was a weak interaction between adhesive and particles. Many authors dealt with this research. When applying the filler into resin, the wetting of filler with matrix is very important (JACKEL, SCHEIBNER 1991). Results of the experiment also shows the irregular stratification of filler microparticles in the matrix.

From the fracture surface the non-homogeneity of adhesive is also evident. This is caused by air bubbles arisen during both, mixing process of two

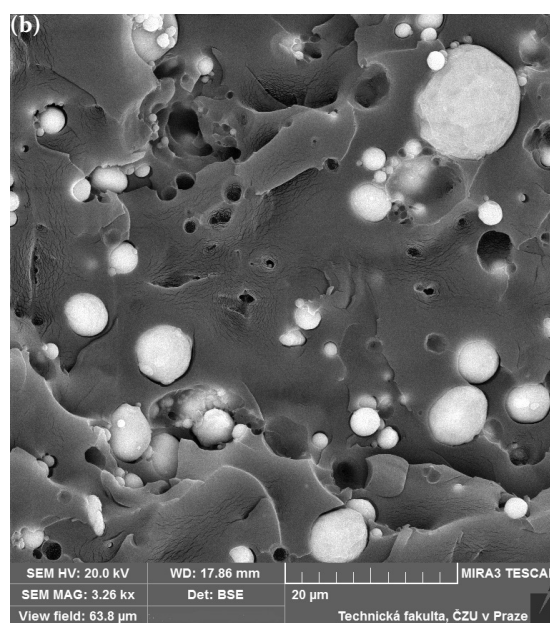
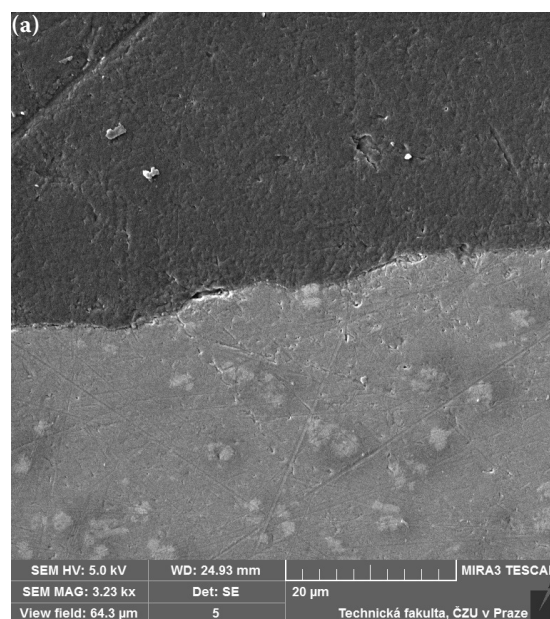


Fig. 2. SEM images of (a) micro-cracks in interface of adherent/adhesive (0 % filler) and (b) of microparticles with 20 vol % of aluminium particles

component epoxy adhesive and during hardening without using vacuum.

## CONCLUSION

From the results of the experiments aimed at the analysis of production parameters of single-lap bonds adhesively bonded using composites on the

aluminium based microparticles it is possible to state following conclusions:

Strength of adhesively bonded joint is depended on the thickness of bonded material. There is not significant increase of strength of adhesive bond with thickness from ca. 1.90 mm at tested material AlCu4Mg.

There was not apparent significant deformation of bonded material at the failure of adhesive layer causing the bending moment which decreases strength of the adhesively bonded joint for tested materials B ( $1.90 \pm 0.03$  mm) and C ( $3.01 \pm 0.04$  mm).

Strength of adhesively bonded joint is dependent on lapping length of adhesively bonded material. The highest values of strength of adhesively bonded joint were reached with the coefficient of proportional length  $0.27 \pm 0.01$  mm. There was a decrease of strength of adhesively bonded joint with increasing coefficient of proportional length.

There was not confirmed assumption about negative effect of filler on tensile strength during the experiments. There is a minimum effect of different filler concentration of aluminium microparticle, i.e. 0, 10, 15 and 20 vol. % on strength of adhesively bonded joint.

Adhesively bonded joints evinced both, adhesive and special cohesive type of fracture surface. By adding the filler in form of aluminium microparticles there was not a change of failure type. The increase of percentage representation of special cohesive failure occurred due to change of thickness of bonded material. The special cohesive failure of adhesively bonded joint occurred more frequently at bonded materials B ( $1.90 \pm 0.03$  mm) and C ( $3.01 \pm 0.04$  mm).

The presence of diversely large aluminium microparticles was proven using SEM within experimental research. The microparticles had regular spherical shape. There is a weak interaction between adhesive and microparticles.

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