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Mechanical properties of resin reinforced with glass beads

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Abstract. The research was focused on the evaluation of a loading speed and a size of the particle filler in a form of glass beads B159 and B112 on resultant behaviour of a composite material and during its application in a structural adhesive bond. A fall of the tensile strength of the composite material of ca. 60% is obvious from the experiment results when adding both fillers (B159 as well as B112). This composite material showed in the positive way as the adhesive at the adhesive bonds. The experiment results proved the positive influence of adding the particle filler of the spherical shape – glass beads B159 (the fraction size 85.23 ± 31.23 μm) on the adhesive bond strength. The adhesive bond strength was increased up of 14% at the filler glass beads B159. However, adding the filler into the resin proved that this filler eliminated the influence of various loading speeds. Adding the filler into the resin changed a fracture surface. An analysis of a scanning electron microscopy (SEM) proved a good wettability of the filler, the resin and the adhesive bonded material (a structural carbon steel S235J0). A crack propagation was concentrated around the filler B112 (151.59 ± 53.04 μm), namely at higher value of the loading speed, i.e. 10 mm min^{-1}. The crack propagation is a consequence of this. Higher particles show in a negative way namely at an initiation of the fracture surface.

Key words: Loading speed, particle filler, SEM, strength.

INTRODUCTION

The use of the adhesive bonding technology in the construction of transport means and agricultural machines secures a stiffness comparable with mechanical fasteners or spot-welds (Borselino et al., 2009). Further, the adhesive bonding technology increases an energy absorption reducing a noise and vibrations (Borselino et al., 2009).

The adhesive bonding strengthens its position in a number of agricultural machinery and tools. It can be mentioned e.g. the cooperation of the firms Henkel and New Holland in the area of the agriculture (Müller, 2013; Müller & Valášek, 2013). An 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 (Müller, 2013).

The adhesive bonding technology belongs among significant bonding methods of diverse materials (Šleger & Müller, 2016). A present trend is to add a filler into the adhesive. A reason is mainly a decrease of the adhesive price and an improvement of mechanical properties (Kim & Khamis, 2001; Kawaguchi & Pearson, 2003; Agoudjil et al., 2008; Ramazan et al., 2008; Müller & Valášek, 2012; Müller et al., 2015; Valášek &

Müller, 2015; Šleger & Müller, 2016). Some polymers can be toughened by rigid inorganic fillers (Lee & Yeeb, 2000).

Polymer particle composites are usually applied in a renovation area where they are used namely as cements (Cho et al., 2006; Ruggiero et al., 2015; Valášek, 2015a; Valášek, 2015b; Müller, 2016). An improvement of the adhesive bond properties can be reached at a suitably chosen filler type, its size and a concentration. The properties of the composite mixture are similar as of the adhesive itself. Particle-reinforced polymers are widely used in load-carrying applications (Tjernlund et al., 2006). Fillers of various dimensions are frequently added to make polymers stiffer, cheaper and improve on properties such as heat resistance, dimensional stability, fracture toughness and colour control (Tjernlund et al., 2006; Kawaguchi & Pearson, 2003).

The strength strongly depends on the stress transfer between the particles and the matrix (Shao-Yun et al., 2008). For well-bonded particles, the applied stress can be effectively transferred to the particles from the matrix, this clearly improves the strength (Shao-Yun et al., 2008). However, for poorly bonded microparticles, the strength reductions occur by adding particles (Shao-Yun et al., 2008).

The mechanical properties of polymer particle composites depend strongly on a particle size, a particle–matrix interface adhesion (wettability) and a particle loading (Kawaguchi & Pearson, 2003). A crack propagation along the interface between the filler and the epoxy matrix threatens at the use of the filler (Tjernlund et al., 2006). This propagation largely depends on the size and the shape of the particles.

According to this mechanism, particles in a brittle matrix can resist the crack propagation by making the crack front bow out between particles (Lee & Yeeb, 2000). The amount of the line energy in the bowed crack front reflects the toughness increase due to the existence of particles (Lee & Yeeb, 2000). This mechanism explains the common generalization on the effect of the inherent matrix toughness, i.e. the toughening effect of the inorganic particle incorporation into polymers decreases as the inherent matrix toughness increases (Lee & Yeeb, 2000).

The former research results proved a positive influence of adding particle filler of the spherical shape – glass beads B159 with the fraction size 90 ± 20 μm on the adhesive bond strength (Müller, 2016). The highest increase of the adhesive bond strength was at the adhesive bond with the adhesive in the form of the composite (150 g of filler: 100 g of matrix) (Müller, 2016). Also Sanchez-Soto et al. (2007) came to similar conclusions that adding glass microballs with the dimension to the diameter 120 μm increases tensile properties of resulting particle composites. The filler of composite materials is different Al_2O_3, SiC, glass beads, minerals, various metals, rubber particles. Although the use of particles as fillers in many polymers is widespread, the knowledge about the fracture behaviour of inorganic particle filled composites has developed very slowly (Lee & Yeeb, 2000).

The research subject is the influence of the particle filler (glass beads) on mechanical properties of the composite mixture with the evaluation of the interaction of the matrix and the reinforcement by means of the fracture surface at the same time. The research aim was not to determine the influence of the filler concentration. This had been already done and the pieces of knowledge were used from another study.

The research focused on the evaluation of the influence of the loading speed and the particle size on the resultant behaviour of the composite material and during its application in the area of the structural adhesive bond. The influence of the fracture

surface at various loading speeds on the composite system strength was investigated by means of SEM. The composite system strength is based on the transfer of the stress between the matrix and the filler. Also the adhesive layer between the adhesive and the adhesive bonded material is the essential factor at the adhesive bond (Shao-Yun et al., 2008).

MATERIALS AND METHODS

The subject of performed experiments was the polymer composite whose continuous phase was in a form of the structural two-component adhesive CHS Epoxy 1200 (resin) and a discontinuous phase / reinforcing particles in a form of glass beads B159 (a fraction size 85.23 ± 31.23 μm) and B112 (a fraction size 151.59 ± 53.04 μm) which was applied on the structural carbon steel S235J0. Figs 1 and 2 show a histogram of a frequency of glass beads filler. It is obvious from the results that the highest portion was at the glass beads B112 among 100 to 200 μm and B159 among 60 to 140 μm. The polymer filler was of very different dimensions.

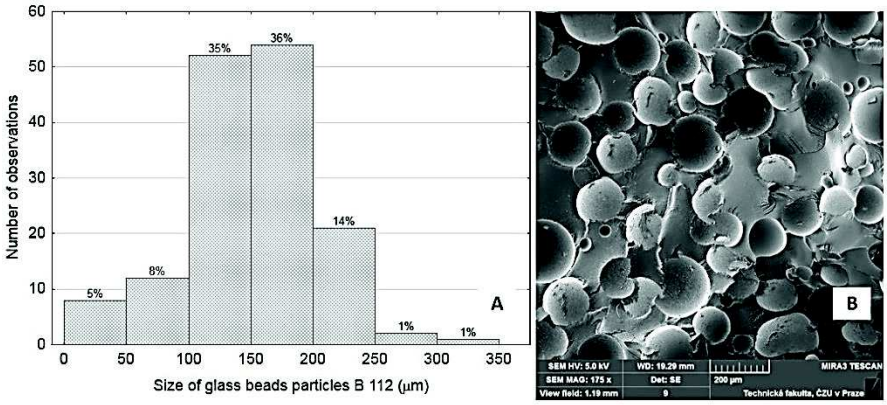

Figure 1. Filler in form of glass beads B112: A) Histogram, B) composite with spherical particles (SEM image, secondary electrons, MAG 175 x).

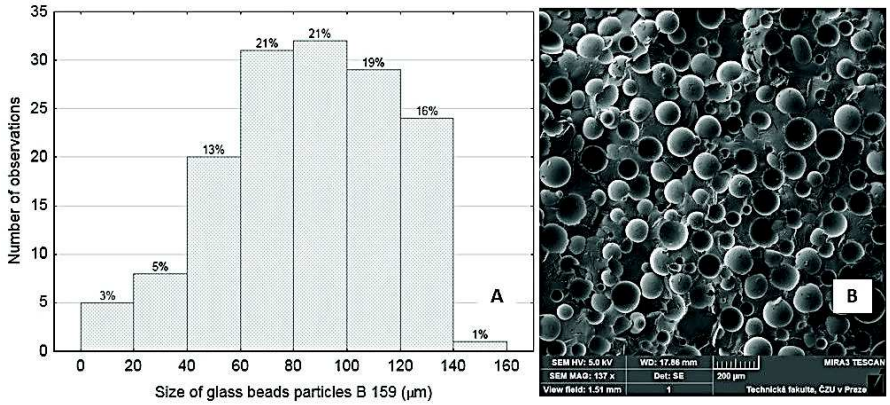

Figure 2. Filler in form of glass beads B159: A) Histogram, B) composite with spherical particles (SEM image, secondary electrons, MAG 137 x).

The filler concentration 40 vol.% was used at the research on the influence of the loading speed on mechanical properties of the composite material. This concentration corresponded to reaching the optimum composite strength (Müller, 2016).

The research focused on the evaluation of the influence of the loading speed and the particle size on resultant behaviour of the composite material and during its application in the area of the structural adhesive bond by means of the static tensile test and the adhesive bond strength.

The test specimens for the tensile properties determination according to the standard CSN EN ISO 527-1 (Plastics – Determination of tensile properties – Part 1: General principles) were prepared according to the standard CSN EN ISO 3167 (Plastics – Multipurpose test specimens, Czech Standard Institution). The tensile strength was the evaluated parameter. The test specimens were cast into the moulds from Lukapren N which corresponded to the requirements of the standard by their shape and dimensions. The universal tensile strength testing machine LABTest 5.50ST (a sensing unit AST type KAF 50 kN, an evaluating software Test&Motion) was used for the determination of the tensile strength. The loading speed of the deformation corresponded to 1, 5 and 10 mm min^{-1}.

The hardness of the tested materials was determined on the base of the standard CSN EN ISO 2039 by means of a ball of a diameter 5 mm on the device Durajet (the Struers company), a loading force corresponded to 961 N.

The adhesive bond strength was analysed owing to a verification of the composite mixture behaviour in the interaction with the adhesive bonded material.

Laboratory tests were performed using the standardized test specimens made according to the standard CSN EN 1465 (dimensions 100 ± 0.25 x 25 ± 0.25 x 1.5 ± 0.1 mm and lapped length of 12.5 ± 0.25 mm) from the structural carbon steel S235J0. The adhesive bonded surface was mechanically treated (grit blasted by Garnet MESH 80, a fraction size 0.1–0.3 mm) and chemically treated (cleaned in the acetone bath). The roughness parameters Ra and Rz were measured on the surface of grit blasted adherents, $Ra = 1.76 \pm 0.16$ μm, Rz 10.92 ± 0.85 μm. Roughness parameters were measured with a portable profilometer Mitutoyo Surftest 301. A limit wavelength of the cut-off was set as 0.8 mm. Adhesive bonds were hardened for 72 ± 5 hours with a temperature 22 ± 2 °C.

The universal tensile strength testing machine LABTest 5.50ST (a sensing unit AST type KAF 50 kN, an evaluating software Test&Motion) was used for the determination of the adhesive bond strength. The loading speed of the deformation corresponded to 1, 5 and 10 mm min^{-1}. A fracture surface of the adhesive bonds was evaluated according to the standard ISO 10365.

Fracture surfaces and an adhesive bond cut was examined with SEM (scanning electron microscopy) using a microscope MIRA 3 TESCAN at the accelerating voltage of the pack (HV) 5.0 kV. The samples were dusted with gold by means of the equipment Quorum Q150R ES – Sputtering Deposition Rate using Gold.

Data were also evaluated 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

The hardness of the tested materials is following: resin 153.85 ± 4.78, composite B159 256 ± 9.74 and composite B112 275 ± 16.09. Composite mixture was considerably increased with the resin (matrix), of ca. 67 to 79%.

The results of the loading speed influence of the resin and the composite material based on the glass beads on the static tensile strength are visible in Fig. 3. The fall of the tensile strength by adding both fillers glass beads B159 as well as B112 is visible from the experiment results. The tensile strength at the polymer particle composite reinforced with the glass beads B159 was reduced of 61.5% and with B112 of 55.2%.

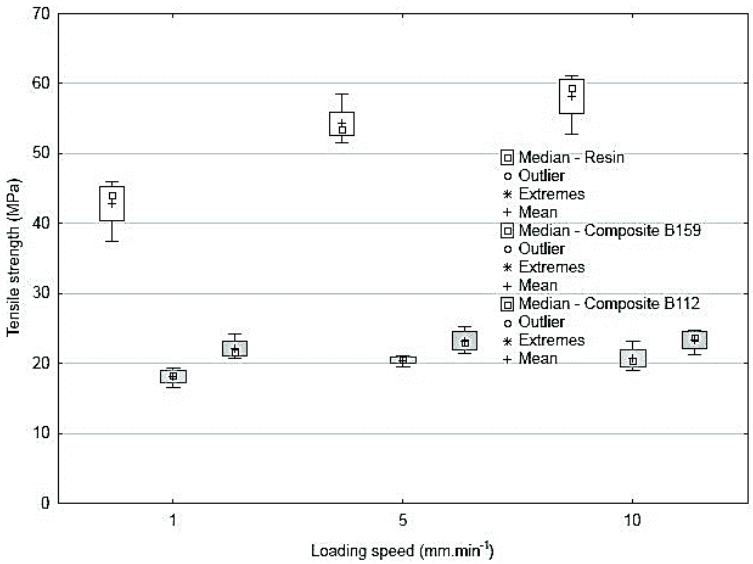

Figure 3. Influence of loading speed on tensile strength of resin and polymeric particle composite.

It is possible to say in terms of the statistical testing of the resin that the loading speeds are statistically non-homogeneous groups ($p = 0.004$), i.e. there is the difference in the resultant tensile strength among single loading speeds 1, 5 and 10 mm min^{-1}. The hypothesis H_0 was not certified in the significance level 0.05. It is obvious from the results that different loading speed influences the tensile strength. The experiment results proved the increase of the tensile strength of the resin due to increasing loading speed of 27 to 3%.

It is possible to say in terms of the statistical testing of the polymer particle composites based on the glass beads B159 and B112 that the loading speeds are statistically homogeneous groups ($p_{B159} = 0.336$, $p_{B112} = 0.489$), i.e. there is no difference in the resultant tensile strength among single loading speeds 1, 5 and 10 mm min^{-1}. The hypothesis H_0 was certified in the significance level 0.05. It is obvious from the results that different loading speed does not influence the tensile strength. The loading speed cannot be regarded as the statistically significant at the application of the tested polymer particle composite.

Other parameters determined from the tensile test (Elongation and Time of destruction) are presented in Table 1. An average elongation at the resin was 6.81 ± 1.02%. The average elongation at the composite B159 and B112 was 1.96 ± 0.18%. The elongation did not significantly change owing to the loading speed. The loading speed considerably influenced the time of the destruction. By adding the filler, the elongation and the loading speed were considerably changed (decreased).

Table 1. Tensile test (CSN EN ISO 527-1) (**Elongation ε, Time of destruction t**)

Abbr.	Properties	Resin			Composite B 159			Composite B 112		
	Loading speed (mm min^{-1})	1	5	10	1	5	10	1	5	10
ε	Mean (%)	5.41	7.19	7.82	1.44	1.79	1.75	1.37	1.47	1.77
	Standard deviation (%)	0.41	0.55	0.51	0.13	0.14	0.16	0.11	0.15	0.11
	Variation coefficient (%)	7.55	7.66	6.55	8.74	7.65	9.23	8.21	9.99	6.11
t	Mean (s)	163	46.7	23.7	59.7	10.7	5.27	36.1	7.09	4.48
	Standard deviation (s)	11.5	2.47	1.78	6.47	0.82	0.49	2.45	0.72	0.5
	Variation coefficient (%)	7.04	5.29	7.49	10.8	7.66	9.2	6.79	10.2	11.22

SEM (scanning electron microscopy) analysis was used for the study of fracture surfaces and cuts through the adhesive bonds. SEM analysis enabled to display a quality of the interaction of the reinforcing phase (glass beads) and the resin. A better understanding of the tested material behaviour was the reason for the research by means of SEM. The fracture surface of the resin and the composite systems after the tensile test are visible in Fig. 4. The interaction between the filler and the resin is good (Fig. 4, A & B). However, a reduction of the wetting was possible to observe in some places, i.e. the interaction between the resin and the glass beads B 159 and B 112 was lower 0.13 ± 0.06 μm (Fig. 4, B).

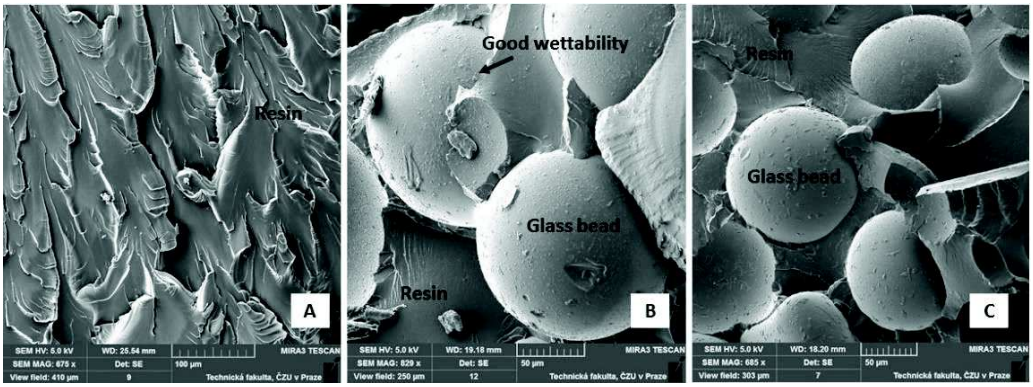

Figure 4. SEM images of fracture surface after tensile test (secondary electrons): A: Resin (MAG 675 x), B: interaction of resin and glass beads B112 (MAG 829 x), C: interaction of resin and glass beads B159 (MAG 829 x)

Results of the influence of the loading speed of the resin and the composite material based on glass beads on the adhesive bond strength are visible in Fig. 5. The fall of the adhesive bond strength of ca. 1.5% by adding the glass beads B112 filler is obvious from the experiment results. The increase of the adhesive bond strength of ca. 14.3% by

adding the glass beads B159 filler is obvious from the experiment results. The adhesive bond strength was increased by adding the spherical filler of smaller dimension.

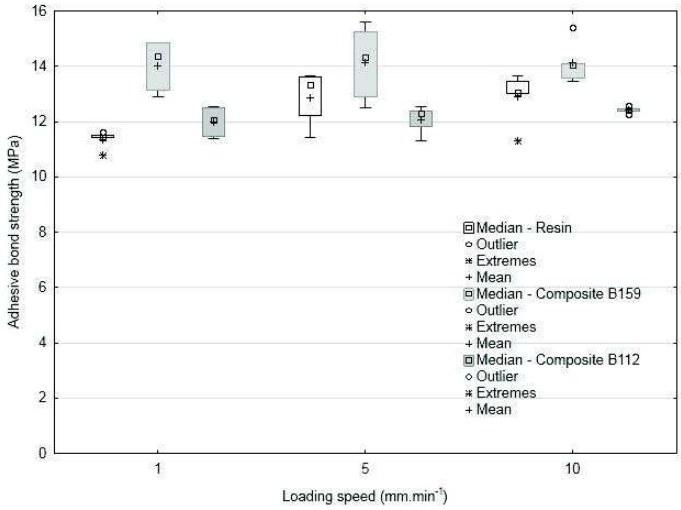

Figure 5. Influence of loading speed on adhesive bond strength of resin and polymer particle composite.

It is possible to say in terms of the statistical testing of the resin that the loading speeds are statistically non-homogeneous groups ($p = 0.015$), i.e. there is the difference in the resultant adhesive bond strength among single loading speeds 1, 5 and 10 mm min^{-1}. The hypothesis H_0 was not certified in the significance level *0.05*. It is obvious from the results that different loading speed influences the adhesive bond strength. The experiment results proved the increase of the adhesive bond strength at the resin owing to the loading speed of ca. 13%. The considerable difference in the adhesive bond strength did not occur at the loading speeds 5 and 10 mm min^{-1}.

It is possible to say in terms of the statistical testing of the polymeric particle composites based on glass beads B 159 and B112 that the loading speeds are statistically homogeneous groups ($p_{B159} = 0.986$, $p_{B112} = 0.290$), i.e. there is no difference in the resultant adhesive bond strength among single loading speeds 1, 5 and 10 mm min^{-1}. The hypothesis H_0 was certified in the significance level *0.05*. It is evident from the results that different loading speed did not influence the strength of the composite adhesive bond. The loading speed cannot be regarded as statistically significant at the application of the tested polymer particle composite.

The layer thickness of the adhesive is essential (Naito et al., 2012). The layer thickness of the adhesive was considerably different depending on the filler type. The highest adhesive bond thickness 291.56 ± 12.29 μm was measured at the adhesive bonds reinforced with glass beads B 112. It was 224.32 ± 12.46 μm at the adhesive bonds reinforced with glass beads B159. The layer thickness was the smallest at the adhesive bonds bonded only with the resin – 162.25 ± 48.56 μm. However, it is evident from the results that the layer of the adhesive bond was uneven, i.e. the variation coefficient was 29.9%. The layer was even at the adhesive bonds reinforced with the filler, i.e. the variation coefficient was 4.2 to 5.6%.

The type of the fracture surface differed for various variants of the experiment. The adhesive bond failure was of the adhesive type, i.e. the adhesive bond failure occurred between the adhesive bonded material (adherent) and the resin. The adhesive bonds reinforced with the glass beads showed the adhesive – cohesive type of the fracture surface. The loading speed did not changed the fracture surface.

Other parameters determined from the adhesive bonds testing are presented in Table 2. The average elongation was 2.46 ± 0.16%. The considerable changes of the elongation owing to the loading speed occurred at the resin and the composite B112. The loading speed significantly influenced the time of the destruction. A considerable change occurred between the speed 1 and 5 mm min^{-1}.

Table 2. Adhesive bond strength (CSN EN 1465) (Elongation ε, Time of destruction t)

Abbr.	Properties	Resin			Composite B 159			Composite B 112		
	Loading speed (mm min^{-1})	1	5	10	1	5	10	1	5	10
ε	Mean (%)	2.20	2.39	2.48	2.50	2.49	2.54	2.24	2.52	2.77
	Standard deviation (%)	0.27	0.25	0.11	0.18	0.30	0.17	0.18	0.21	0.20
	Variation coefficient (%)	12.29	10.4	4.44	7.36	12.1	6.88	8.13	8.42	7.07
t	Mean (s)	62.06	14.3	6.84	82.56	14.5	7.83	63.55	14.2	8.45
	Standard deviation (s)	3.84	1.08	0.94	8.96	2.02	0.22	6.31	2.02	0.30
	Variation coefficient (%)	6.18	7.57	13.8	10.85	13.9	2.81	9.94	14.22	3.57

SEM analysis proved a good wettability of the adhesive and the adhesive bonded material (carbon steel S235J0) (Fig. 6 A, B & 7, A).

This conclusion is essential because the wettability of the adhesive bonded surfaces is crucial for good adhesive strength (Rudawska, 2012; Müller & Valášek, 2013; Rudawska et al., 2016; Šleger & Müller, 2016). The fracture surface of the adhesive bond is visible in Fig. 6, C. The fracture surface was concentrated around the filler B 112, namely at higher values of the loading speed, i.e. 10 mm min^{-1} (Fig. 7, B). A spreading of the fracture surface is a consequence of this. Higher particles show themselves in a negative way.

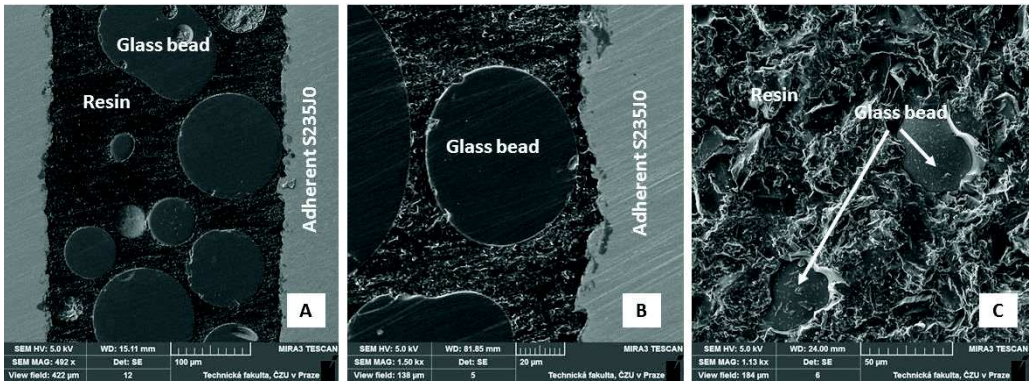

Figure 6. SEM images of cut through adhesive bond reinforced with glass beads (secondary electrons): A: cut through adhesive bond of resin reinforced with glass beads B 112 (MAG 492 x); B: good wettability of glass beads B 112 filler with resin (MAG 1.50 kx); C: adhesive – cohesive type of fracture surface of adhesive bond (MAG 1.13 kx).

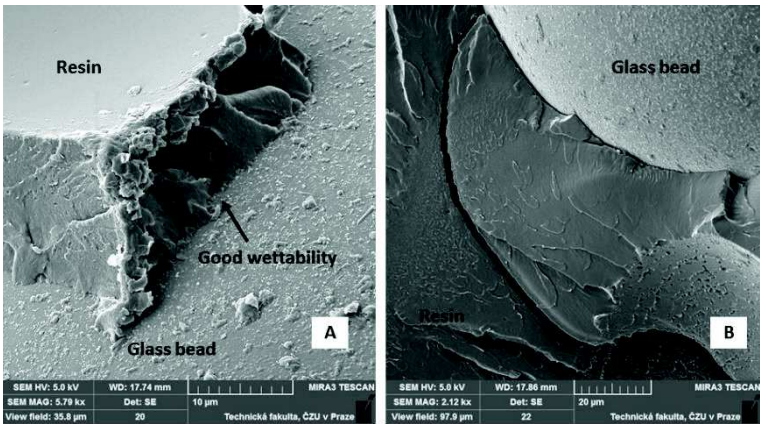

Figure 7. SEM images of fracture surface after shear tensile strength test of adhesive bonds (secondary electrons): A: good interaction of resin and glass beads B112, loading speed 1 mm min^{-1} (MAG 579 kx); B: fracture surface within adhesive layer stopped by glass beads B112 filler, loading speed 10 mm min^{-1} (MAG 212 kx).

The tensile strength is decreased at the epoxies which filled with glass beads when increasing the loading speed of the composite material (Gurusideswar & Velmurugan, 2014). The presence of the filler considerably influences the mechanical properties. The experiment results proved significant fall of the tensile strength by adding the filler. The fall of the tensile strength was of ca. 62% at the filler B159 and ca. 55% at the filler B112 (Valášek, 2011). Similar results were gained also in other researches which state to 80% fall of the tensile strength at using the glass beads B159 (Valášek, 2011). The fall of the composite strength with increasing mass portion is attributed to an agglomeration and poor interfacial bonds between the matrix and the filler Gurusideswar & Velmurugan, 2014). A visual check of tested samples shows significant changes in the fracture surface with increased loading speed Gurusideswar & Velmurugan, 2014). It is obvious from the results that the loading speed of the adhesive bonds has a positive influence on the strength results of tested adhesive bonds (Müller et al., 2016).

The increase of the adhesive bond strength by adding the glass beads B 159 filler of ca. 14.3% is obvious from the experiment results. The adhesive bond strength was increased by adding the spherical filler of smaller dimensions. The glass beads B 112 filler which is of greater fraction did not caused the increase of the adhesive bond strength values. Mild fall of the adhesive bond strength was proved also at Fe based particles (Valášek et al., 2016). Similar increase of the adhesive bond strength of ca. 14% occurred also in other studies (Müller, 2016).

The assumption that the stress in the polymer particle composite filled with the glass beads is concentrated around the filler of higher dimensions was certified (Lee & Yeeb, 2000). Spreading of the fracture surface is the consequence of this. This state was visible in SEM image – see Fig. 7, B.

The cohesive strength can be related to the strain energy release rate of a material through a decohesion process occurring at the crack tip (Kawaguchi & Pearson, 2003). Yield behaviour is also related to the strain energy release rate through plastic zone formation at the crack tip (Kawaguchi & Pearson, 2003).

Even thickness of the adhesive layer is essential (Naito et al., 2012). The experiment results certified that the adhesive bond without the filler is of huge uneven thickness of the adhesive layer, the difference is up to ca. 30% (Lee & Yeeb, 2000). On the contrary when using the filler the dispersion of the layer thickness was reduced to max. 5.6%.

CONCLUSIONS

The experiment subject is the behaviour of the particle filler (glass beads) in relation to mechanical properties of the composite mixture at simultaneous evaluation of the interaction of the matrix and the reinforcement by means of the fracture surface. The experiment focused on the evaluation of the influence of the loading speed and the particle size on resultant behaviour of the composite material and at its application in the structural adhesive bond. Following conclusions can be deduced from the experiment results:

- A fall of the tensile strength by adding the filler – glass beads B159 and B112 is obvious from the experiment results. The tensile strength of the polymeric particle composite reinforced with the glass beads B159 filler was reduced of 61.5% and with the glass beads B112 of 55.2%. It is obvious from the results that different loading speed influenced the tensile strength of the resin. It is also obvious that different loading speed does not influence the tensile strength of the resin filled with the glass bead microparticles. The loading speed cannot be regarded as the statistically significant at the application of tested polymer particle composite. The loading speed considerably influenced the time of the destruction and the elongation. By adding the filler the significant change (fall) of the elongation and the loading speed occurred.
- The experiment results proved a positive influence of adding the particle filler of the spherical shape – glass beads B159 on the adhesive bond strength. The adhesive bond strength was increased up of 14%. The glass beads B112 filler which is of greater fraction did not influence the adhesive bond strength (neither an increase nor a fall). The adhesive bond strength was similar as at the resin. It is obvious from the results that different loading speed influences the adhesive bond strength. The experiment results proved the increase of the adhesive bond strength at the resin owing to the loading speed of ca. 13%. The adhesive bond strength was not so different at the loading speed 5 and 10 mm min^{-1}. However, adding the filler into the resin proved that this filler eliminated the influence of different loading speed. The loading speed cannot be regarded as the statistically significant at the application of tested polymer particle composite. The loading speed considerably influenced the time of the destruction and the elongation, namely at the resin and the composite B112. The loading speed did not change the fracture surface. Adding the filler into the resin changed the fracture surface.
- SEM analysis proved good wettability of the filler and the adhesive bonded material (adherent – carbon steel S235J0) with the resin. The fracture surface spreading was concentrated around the filler B112, namely at higher values of the loading speed, i.e. 10 mm min^{-1}. Greater particles show themselves in a negative way, namely at the initiation of the cracks.

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