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POSSIBILITIES OF ADHESIVES FILLING WITH MICRO-PARTICLE FILLERS – LAP-SHEAR TENSILE STRENGTH

Petr Valášek[1], Miroslav Müller[1]

[1] Department of Material Science and Manufacturing Technology, Faculty of Engineering, Czech University of Life Sciences Prague, Kamýcká 129, 165 21 Praha 6, Czech Republic

Abstract

VALÁŠEK PETR, MÜLLER MIROSLAV. 2016. Possibilities of Adhesives Filling With Micro-particle Fillers – Lap-shear Tensile Strength. *Acta Universitatis Agriculturae et Silviculturae Mendelianae Brunensis*, 64(1): 195–201.

An adhesive bonding can be ranged among technologies of materials bonding which are used in all industrial branches. It plays its important role also in an area of the construction of agricultural machines, e.g. tractors, harvestors etc. Utility properties of adhesives can be extended by using various types of fillers. These fillers increase some mechanical characteristics of adhesives and not last they can decrease resultant price. The paper focuses on a possibility to fill the adhesives showing increased lap-shear tensile strength. These adhesives are used e.g. for adhesive bonding of coach bodies in an automotive industry so that is why it is possible to apply them also in the agricultural area. Laboratory experiments describe the lap-shear tensile strength of rigid adherents which were bonded with adhesives with a variable concentration of micro-particle filler – a glass powder. T-test used for a comparison of the shear strength of the bonds created with the adhesives with 5 vol.% of the glass powder did not evidence a statistically significant difference comparing with the unfilled adhesives. This priece of knowledge opens a possible way of a material usage of the glass powder in the area of the adhesive bonding.

Keywords: agriculture, epoxy resin, glass powder, mechanical properties

INTRODUCTION

The technology of bonding materials is represented in all industrial branches. Adhesive bonds are used at the constructions of coach bodies and nowadays also of agricultural machines (Müller and Valášek, 2014). As an example a cooperation of companies developing special types of adhesives and companies focusing on the production of agricultural machines (e.g. a cooperation of Henkel and New Holland) can be mentioned. Reasons for using the adhesive bonds in practice can be following:

- a production simplicity under conditions both piece and serial production,
- an applicability at thin adherents,
- a minimization of a mass,
- prevailing shear loading (Messler, 2004; Grant *et al.*, 2009).

In the area of the agriculture the adhesive bonds can be exposed to degradation environments which can decrease their strength (Müller and Valášek, 2012). Stressing of the adhesive bonds is key from their construction point of view because adhesive bonds are only rarely of one loading type. Usually the combined tensile and shear stress occur. The nonuniform stress distribution occurs on the whole bonded surface (Müller and Herák, 2010). According to Adams (1997), owing to the nonuniform deformation the different adhesive deformation occurs through adhesive layer thickness.

Kahramana *et al.* (2008) dealt with single-lap joints bonded with aluminium powder filled epoxy. This bonds fail in a cohesive mode (failure within the adhesive) due to the high stress levels generated in the adhesive, which indicates that the adhesion to the metal surface is stronger than that of the interior part of the adhesive. Kavak and Altan (2012) investigated epoxy resins filled with particles of 63/37 Sn-Pb. Their experimental results show that bonds prepared by adhesive which was modified, adding the amount of 5 wt% of 63/37 Sn-Pb powder, are of hogher mechanical strength than bonds which are prepared by adding aluminium powder with different ratios as 5, 25, 50 wt%. Kilik and Davies (1989) determined the used adhesive as a toughened, single part epoxy which contained various amounts of admixed copper and aluminium powders. Zhai *et al.* (2006) demonstrated that incorporating epoxy adhesive with nanoparticles (nano-Al_2O_3 homogeneously dispersed in epoxy adhesive) could distinctly increase the adhesion strength of epoxy adhesive. Bahattab *et al.* (2011) increased shear adhesion strength values of polyurethane bonds by filling with nanosilicas of different particle size from the value 14.2 ± 0.1 MPa to the value 15.3 ± 0.3 MPa (100 nm, 30 wt%).

Inorganic micro-particles need not to serve only for optimizing the adhesive and cohesive characteristics of bonds but they can also according to authors (Valášek and Müller, 2013a) increase other mechanical properties. In the practice hard inorganic particles (SiC, Al_2O_3) are used which should increase the ability of the system to resist to the abrasive wear. Then these systems serve for repairs of damaged machine parts such as shafts, boxes, keyways for tongues and flanges, or of damaged parts such as casts and pipes. The glass powder also increases the resistance of epoxides to the abrasive wear (Valášek and Müller, 2013b).

The aim of the experiment is to describe the influence of the variable concentration of the glass powder to the lap-shear tensile strength of rigid adherents created with some types of high-strength epoxy adhesives.

MATERIAL AND METHODS

As adhesives one component, epoxy based adhesives specially developed for the body shop were used. The adhesives are used in the car to increase the operation durability and the body stiffness. As a matter of fact following adhesives which were hardened at increased temperature according to requirements of the producer were used:

- Betamate 1040,
- Betamate 1496f,
- Betamate 5103-3,
- SikaPower 492.

They are excellent adhesives used in the construction of the coach – work (over 40 €/kg). Stated adhesives are possible to combine with other bonding methods, e.g. a spot welding and a riveting. Tab. I presents basic characteristics of used adhesives.

The glass powder (GP) Refaglass (Recifa, s.c.) made from the used glass potsherds was used for filling of adhesives. Properties of GP stated by the producer are presented in Tab. II.

Owing to the viscosity the glass powder was mechanically mixed into pre-heated adhesives (50 °C). The density of the glass powder corresponds to 2.5 $g\cdot cm^{-3}$. Consequently the mixture of the adhesive and the glass powder was hardened and used for the adhesive bonding of single steel sheets. The mixture of the glass powder and the adhesives was prepared with different volume percentages (5–20 vol.%).

For the lap-shear strength description the lap assemblies were made (see Fig. 1). The tests were performed using the steel S235J0 specimens of dimensions 100×25×1.5 mm. At first the surfaces of 1.5 mm thick steel sheets were blasted using the synthetic corundum of the fraction F80 under the angle of 90°. In this way the average surface roughness of Ra = 1.59 ± 0.19 µm (Rz = 10.11 ± 0.54 µm) was reached. Then the surface was cleaned and degreased using acetone and prepared to the application. The surface preparation is important and should guarantee good strength on the boundary of the adherent and adhesive (Novák, 2012; Affatato and Ruggiero, 2013; Votava, 2013). An even thickness of the adhesive layer was reached by a constant pressure 0.5 MPa. The lapping was according to the standard 12.5 ± 0.25 mm.

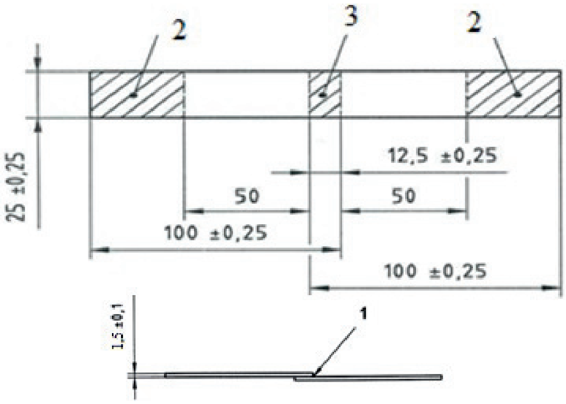
The tensile test strength (according to ČSN EN 1465) was performed using the universal tensile strength testing machine LABTest 5.50ST (50 kN). The failure type according to ISO 10365

I: *Basic characteristics of adhesives*

Adhesives	Density ($g\cdot cm^{-3}$)	Lap Shear Strength (MPa)	Viscosity 23 °C (Pa·s)	Standard curing (°C)
Betamate 1040	1.23	22	140	180/30 min
Betamate 1496f	1.19	31	160	180/30 min
Betamate 5103-3	1.50	23	100	180/30 min
SikaPower 492	1.30	20	900 (50 °C)	< 220

II: *Basic characteristics of glass powder*

Chemical composition					Particles size	Organic impurities
SO_2	CaO	MgO	Na_2CO_3	K_2O		
71%	9–11%	0.5–1.5%	14–15%	0.5%	< 90µm	< 1%



1: *Test specimen – Lap-shear tensile strength (ČSN EN 1465, 1997)*

was determined at the adhesive bonds. The representation of failed surface and adhesive layers were performed using a stereoscopic microscope. For statistical evaluation Anova and T-test were used, reliability level $\alpha = 0.05$.

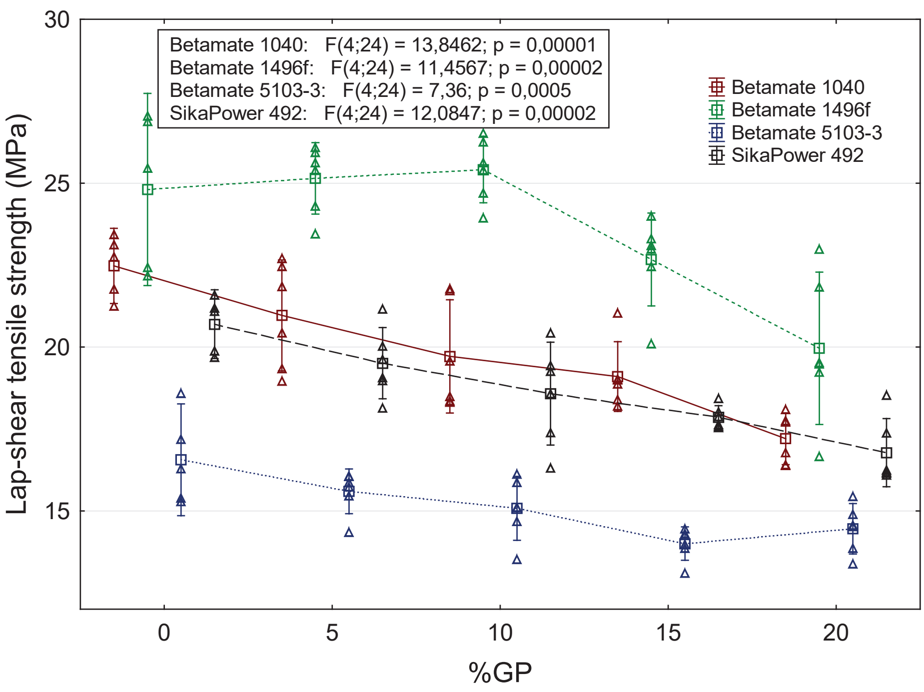
RESULTS

The theoretical density of filled adhesives coming from the densities of the adhesives and the glass powder (at an assumption of a perfect covering of the particles with the adhesive) corresponded according to the densities of the adhesive and the concentration of the glass powder to the interval $1.26–1.70\ g \cdot cm^{-3}$.

Values measured at the steel sheets of the thickness 1.5 mm were at first compared with the values stated by the producer. The adhesives Betamate 1040 (22.47 ± 0.82 MPa) and SikaPower 492 (20.70 ± 0.75 MPa) did not show a significant deviation from the values stated by the producer. On the contrary, the adhesives Betamate 1496f (24.81 ± 2.11 MPa) and Betamate 5103-3 (16.56 ± 1.23 MPa) showed significantly lower experimentally ascertained values than the values stated by the producer. Differences of measured values can differ because the producer does not state particular type of the adherent and its surface treatment. The values presented by the producer are stated for the layer thickness 0.2 mm, while the layer at reviewed bonds was defined by the constant pressure. So it was defined by own viscosity of the adhesive and at the unfilled systems the layer thickness corresponded to 0.38 ± 0.06 mm.

At the filled systems the layer thickness differed depending on the concentration of the filler. The layer thickness ranged in the interval 0.43 mm (5%GP) to 0.48 mm (20%GP) at the glass powder. The shear strength of bonds created with the adhesives with the micro-particles of the glass powder is presented in Fig. 2. Although the fall at the small concentrations of the glass powder (5%GP) was not proved, it can be generally said that with increasing rations of the glass powder (10–20%GP) the fall of the shear strength values occurred.

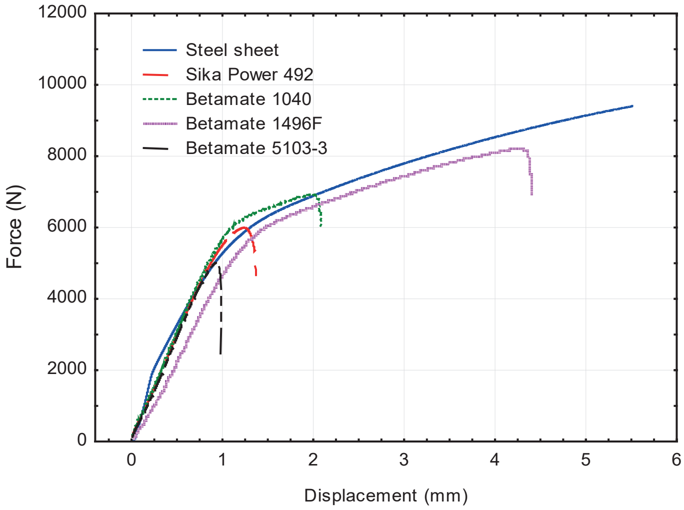
The statistical comparison of the strength values for particular concentrations of the glass powder in the adhesives with the unfilled adhesive – T-test – is presented in Tab. III. H_0 describes the zero hypothesis – there is no statistically significant difference among compared sets of data.



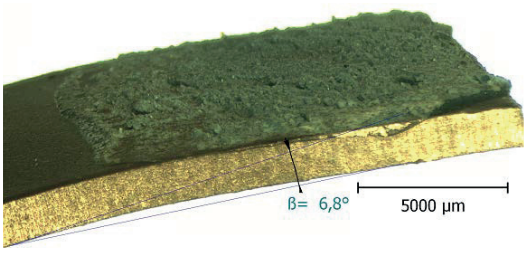
2: *Lap-shear tensile strength of systems with GP*

III: *T-test – Lap-shear tensile strength of adhesives withglass powder*

T-test H_0:(p > 0.05)	Adhesives			
	Betamate 1040	**Betamate 1496f**	**Betamate 5103-3**	**SikaPower 492**
0 : 5%GP	0.09	0.75	0.15	0.07
0 : 10%GP	0.01	0.57	0.06	0.02
0 : 15%GP	0.00	0.09	0.00	0.00
0 : 20%GP	0.00	0.00	0.01	0.00



3: *Tensile diagram (steel sheet and filled systems 5%GP)*



4: *Deformation of adherent*

As it is visible from Tab. III all adhesives with 5%GP did not show a statistically significant difference of the shear strength comparing with the unfilled adhesive. At the adhesives Betamate 1496f (p = 0.57) and Betamate 5103-3 (p = 0.06) the statistically significant fall was not recorded also at the concentration 10%GP. The value p > 0.05 was recorded also at the Betamate 1496f for the concentration 15%GP.

Described systems showed high shear strength. Forces needed for failing the adhesive bond moved already in the area of the plastic deformation of used sheet of metal (1.5 mm). This fact is proved by a tensile diagram (Fig. 3) which shows the curves for the sheet alone and for overlapped systems created whith particular filled adhesives (5%GP).

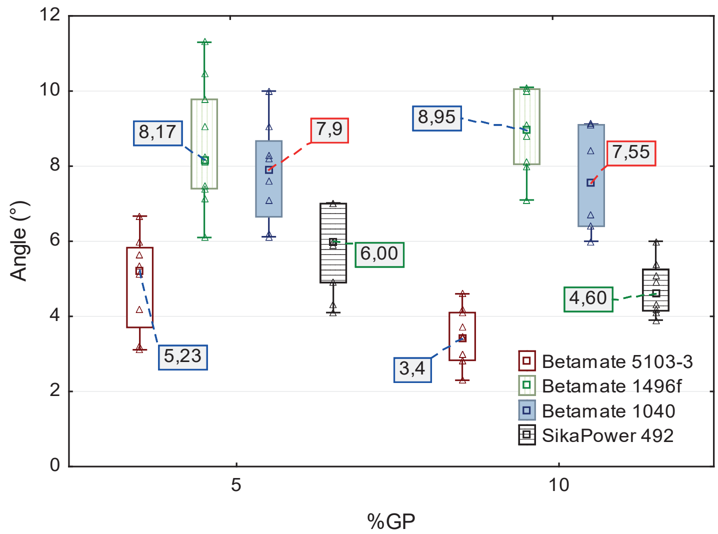
Sheets were subjected to the optical analysis on the stereoscopic microscope after the destruction testing when the measure of the sheet plastic deformation was evaluated by means of the angle (β). Results of measuring of the adherent deformation angle (a sheet of metal of 1.5 mm) are visible in Fig. 4.

The sheet plastic deformation depended on the force which is needed for failing the adhesive bond. The highest values of measured angles (ß) were recorded at the filled systems for the adhesives with 5 and 10%GP (see Fig. 5). The failure occurred in the place of the adhesive bond in all cases.

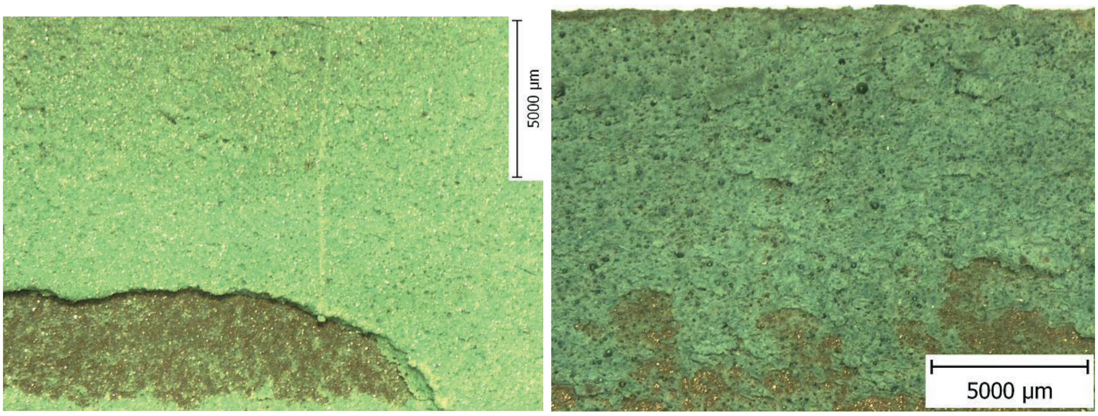
Particular failure type of adhesive bonds was evaluated on the stereoscopic microscope. At the filled adhesives the adhesive-cohesive failure type of the adhesive bond occurred (see Figs. 6, 7).

DISCUSSION

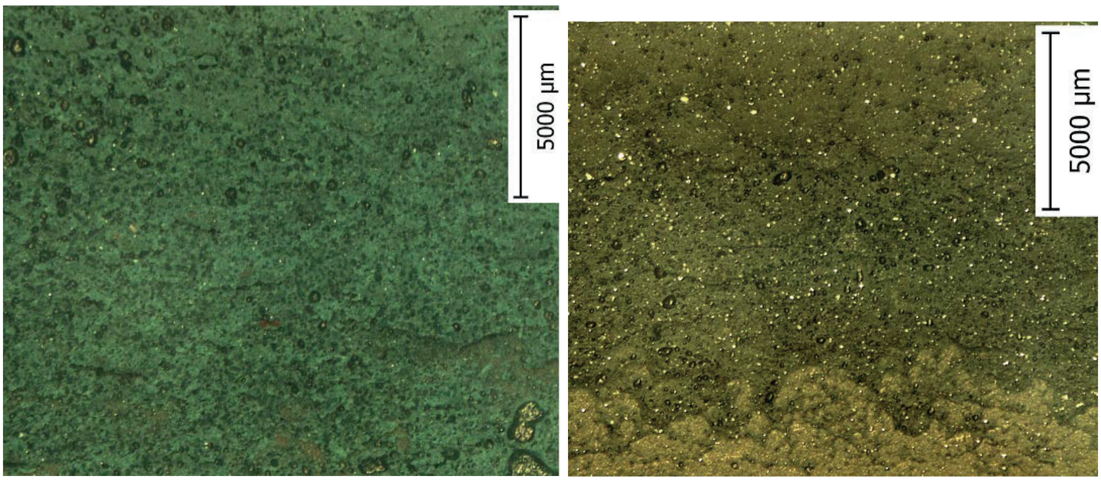
The shear strength fall by the inclusion of 5%GP into the adhesives was not proved by the statistical comparison of data. It came to a mild fall of the average strength value in most cases, however, this fall was negligible from the statistical point of view owing to the magnitude of the standard deviation. However, the arguable strength fall occurred depending on the adhesive type at the same time with higher concentration of the glass powder (10–20%GP). At the adhesive Betamate 1496f the arguable fall of the measured values (of 19.5%) was recorded not until at 20%GP. It did not come to more than 23.5% fall of the values (Betamate 1040) 12.74% (Betamate 5103-3) and 18.93% (SikaPower 492) among the unfilled adhesives and the adhesives with 20% of GP.



5: *Angles of adherent deformation (β) at particular adhesives – 5%GP*



6: *Adhesive bond failure: Betamate 5103-3, Betamate 1496f*



7: *Adhesive bond failure: Betamate 1040, SikaPower 492*

From this point of view the glass powder is possible to be used for filling the epoxy adhesives at lower concentrations – usually until 5%, according to the type of the adhesive. The presence of such amount of the filler does not influence statistically the shear strength but according to authors (Valášek and Müller, 2013b) it decreases the entire cohesive strength of the systems (the tensile strength).

The deformation of adherents during the test was certified. The mean angle of the deformation corresponded to 6° 49´ at the systems with 5% of GP in the adhesives. You *et al.* (2009) indicated the ideal adherent deformation (7°) by means of a finite element analysis in which the strength had increased of 64% comparing with the common lapped adhesive bond. So, the deformation of the adherents leads to another than purely shear stress which influences the strength of the adhesive bond. However, the influence of this deformation has not been a subject of this experiment, however, the optimum constructional setting of the bond can prevent the deformations (Habenicht, 2002).

The application of the glass powder into the epoxy adhesives can decrease the resultant price at keeping the shear strength. However, it is necessary to evaluate particular requirements at the applications and to take into regard mechanical properties of filled adhesive as the complex. Filled adhesives can be used in the agricultural area for constructions of machines bodies, where increased strength of the bond is not required and in the area of adhesive bonding and cementing. Increased wear resistance of adhesives filled with the glass powder can be used as resistant covering layers on parts of machines and equipment.

CONCLUSION

An adhesive bonding can be ranged among technologies of materials bonding which are used in all industrial branches. It plays its important role also in an area of the construction of agricultural machines. The aim of the experiment was to describe the influence of the variable concentration of the glass powder on the lap-shear tensile strength of rigid adherents created with some types of high-strength epoxy adhesives. As adhesives one component, epoxy based adhesives specially developed for the body shop were used. The adhesives are used in the car to increase the operation durability and the body stiffness. Conclusions following from performed experiment can be summed up in a following way:

- The low concentration of the glass powder (5–10 vol.%) in used adhesives did not lead to the statistically arguable decreasing of the shear strength.
- Using the glass powder as the filler can influence the possibility of combining the adhesive bonding with other ways of bonding (e.g. spot welding) in a negative way.
- The inclusion of the glass powder into the adhesives decreases the price.
- The inclusion of the glass powder into the reactoplastics is undemanding way of its utilization.

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Contact information

Petr Valášek: valasekp@tf.czu.cz
Miroslav Müller: muller@tf.czu.cz