LOW-CYCLIC FATIGUE OF ADHESIVE BONDS REINFORCED WITH FIBRES

V. Šleger, M. Müller, J. Zavrtálek

Faculty of Engineering, Czech University of Life Sciences, Prague, Czech Republic

Abstract

The paper deals with a testing of a low-cyclic fatigue of single-lap bonds reinforced with a textile waste from a process of tyres recycling. The aim of the experiment is to clarify a fatigue behaviour (low-cyclic fatigue tests) of joints adhesive bonded with a two-component structural adhesive filled with the textile waste from the process of tyres recycling applied on the structural carbon steel S235J0. The aim of the research was to evaluate a service life of the adhesive bond in terms of its fatigue stressing at a low-cyclic shear test (30 % and 60 % from a reference value of a maximum force). The number of cycles was 200, 500 and 1000. Adding of the filler in the form of the Polyamide PA microfibers from the textile waste showed in a positive way. The adhesive bond strength was increased of 6 % to 11 %.

Key words: number of cycles, Quasi-static load, structural adhesive, textile waste, tyres recycling.

INTRODUCTION

A production process differs in various industrial branches, however, it has usually one common element – a creation of the bond. The adhesive bonding technology is a prospective method of connecting different materials (MÜLLER, VALÁŠEK 2013A; MÜLLER 2011).

A research on factors influencing mechanical properties of the adhesive bond is important for a successful application of adhesive bonds. The adhesive bond fatigue is a significant factor in terms of the practical application (MÜLLER, 2015A; MÜLLER ET AL., 2013; MÜLLER, VALÁŠEK, 2013B; MESSLER 2004).

Adhesive bonds are expected to retain a significant proportion of their load bearing capacity for the entire duration of the service life of the bonded structure. Service conditions can often involve an exposure to the cyclic fatigue, which is possibly the most destructive form of a mechanical loading. The fatigue damage is an irreversible process that can occur at relatively low stress levels due to the presence of high peel and shear stresses at the overlap edges. These stresses reduce both the static strength and fatigue life of bonded structures (BROUGHTON ET AL., 1999).

A particular issue with the integrity of adhesive bonds is the presence of cracks and flaws in the manufactured adhesive bond. The presence of these defects, at least at some scale, appears inevitably and the propagation of such cracks/flaws has the potential to affect the service life of the adhesive bonds and even to cause a catastrophic failure of bonded structures in the service (HAFIZ ET AL., 2010). Hence, a better understanding of the crack propagation behaviour under realistic types of combined service loading is an important aspect of evaluating the potential performance of adhesive bonds (KELLY, 2006; HAFIZ ET AL., 2010).

Adding the suitable filler into an adhesive (a matrix) decreases a price, or it improves mechanical properties of the adhesive bond (VALÁŠEK, MÜLLER, 2015). The filler is based on both a primary raw-material, as well as the waste. In recent years, ways for efficient use of the waste from the tyres recycling have been searching (MÜLLER, 2015B; FANG ET AL., 2000; FERREIRA ET AL.; 2013). A rubber granulate, a metal waste and a textile waste come into being from the mechanical process of the waste tyres recycling (KNAPČÍKOVÁ ET AL., 2014; ACEVEDO ET AL., 2015; MÜLLER, NOVÁK, 2015). The rubber granulate is used in the area of the tyres recycling at the present. The rubber granulate is effectively used in various products. However, we cannot forget other parts of the tyre, e.g. the textile fibres (TARANU ET AL., 2013; MÜLLER, 2015B; FANG ET AL., 2000; FERREIRA ET AL., 2013). Fibres are used for a large variety of applications. Textiles, nonwovens as well as fibre reinforced composite materials are commonly used in daily life and in technical applications (BARTL ET AL., 2015).

Results of a thermal analysis of the waste fibres show that the fibres are of polyamide. After the process of tyres shredding two main types of fibres can be identified: a fibre and a microfibre. In the first case, the fibres maintain their original form (cord), while the microfibers are a consequence of different stages in the shredding process (PARRES ET AL., 2009).

The aim of the research was to set a possible utilization of the cleaned textile waste from the process of the tyres recycling in an area of the polymeric composite systems determined as a filled adhesive. The microfibres from the shredding process of the tyres were used within the research. The aim of experiments is to clarify the fatigue behaviour (low-cyclic fatigue tests) of the bond adhesive bonded with the two-component structural adhesive filled with the textile waste from the process of tyres recycling applied on the structural carbon steel S235J0. The aim of the research was to evaluate the service life of the adhesive bond in terms of its fatigue stressing at the low-cyclic shear test.

MATERIALS AND METHODS

A great proportion of impurities in a form of the rubber granulate was removed from the textile waste used for experiments. This material was used as the filler for a production of the polymeric composite material. The cleaning of the textile microfibres from the rubber particles was performed by a fluid cleaning in a firm which provided the tested filler.

The subject of performed experiments was the polymeric composite, whose continuous phase was in a form of a two-component epoxy adhesive (CHS Epoxy 1200) and a discontinuous phase (reinforcing particles) in a form of Polyamide PA microfibres (the textile waste from the process of tyres recycling).

The concentration of the filler in the matrix is indicated by the wt. fraction of the filler. 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). The filler was added into the matrix CHS Epoxy 1200 (two-component reactoplastics resin) in the ratio of 2 g and 4 g to 100 g of matrix. Weight ratios were chosen with a respect to a practical application when the filler is mixed mainly on the basis of weight ratios.

The basis of adhesive bonds laboratory testing was the determination of the tensile lap-shear strength of rigid-to-rigid bonded assemblies according to the standard ČSN EN 1465 (Equivalent is BS 1465).

Laboratory tests were performed using the standardized test specimens made according to the standard ČSN 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 carbon steel S235J0.

The surface treatment is essential not only in the area of the adhesive bonding technology (MÜLLER, 2014; NOVÁK, 2012; HOLEŠOVSKÝ ET AL., 2012; DOBRÁNSKÝ ET AL., 2015; DOBRÁNSKÝ ET AL., 2014). The surface 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.

Subsequently, the 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 and B.

Then, 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 adhesive bonds were left for 7 days in the laboratory before the destructive testing. The reason was reaching the full strength of the adhesive bond and a minimization of the influence of the secondary hardening. Adhesive bonds hardened at the laboratory temperature 22 °C ± 2 °C.

Shims of the same thickness as the bonded material were adhesive bonded to the edges of the adhesive bonds after ending the hardening process. The reason was an elimination of the bending moment at the destructive testing of the adhesive bonds.

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). The failure type according to ISO 10365 was determined at the adhesive bonds.

Five test specimens were tested in each series. The reference value of the adhesive bond strength was determined for each tested adhesive according to the standard ČSN EN 1465. The upper and lower limits for low-cyclic tests were calculated from the average value.

The test specimens were cyclically loaded in a such way the loading tension pulsated between the minimum value determined from the reference strength of the adhesive bond (i.e. 5 %) and chosen percentage value 30 % or 60 % from the reference strength of the adhesive bond with 0 g filler.

The loading speed at the static test of the adhesive bond strength was always set as $1\ mm \cdot min^{-1}$. The loading speed at the cyclic testing was always set as $30\ mm \cdot min^{-1}$. In the case that the adhesive bond was not destructive damaged after 200, 500 and 1000 cycles, the adhesive bond was subsequently broken, i.e. the testing machine developed force by the speed $1\ mm \cdot min^{-1}$ as long as the test specimens were broken.

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) and Stereoscopic microscope Arsenal.

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 hypotheses and it says that there is a statistically significant difference among tested sets of data or dependence among variables ($p < 0.05$).

RESULTS AND DISCUSSION

A width of the microfiber was 24.38 ± 6.02 µm. A similar mean of the microfibers 29.67 ± 2.3 µm was ascertained also by PARRES ET AL. (2009). The length of the microfibre was very variable – 2424.63 ± 1805.86 µm. PARRES ET AL. (2009) also state very variable length.

A good wettability between the adhesive and the adhesive bonded material (Fig. 1) was proved using the electron microscopy within the experimental research.

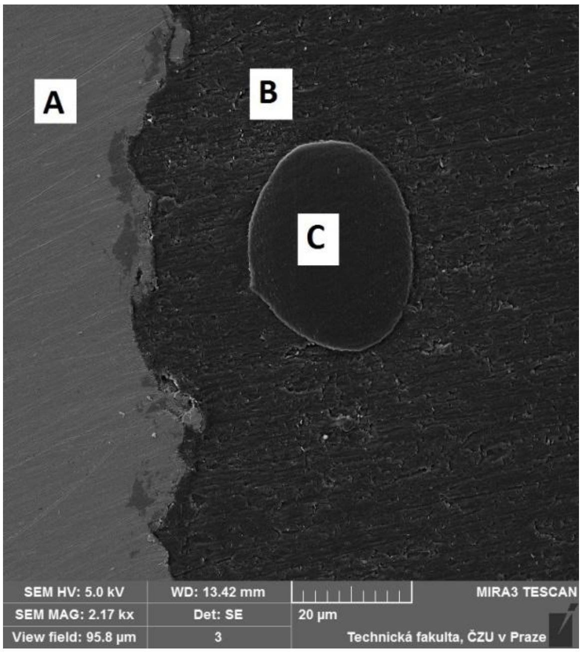

Fig. 1. – SEM images of adhesive bond cut: A - adherent S235J0, B – matrix CHS Lepox 1200 (two-component epoxy), C - microfibres of Polyamide PA from textile waste from process of tyres recycling

The surface roughness of the adhesive bonded material S235J0 after the mechanical treatment by a grit blasting was in the direction parallel to the loading force at the destructive testing of the adhesive bonds Ra 1.96 ± 0.17 µm, Rz 12.23 ± 1.31 µm.

The strength of the adhesive bond without the filler was 10.5 ± 1.4 MPa. Adding 2 g (11.1 ± 0.5 MPa) and 4 g (11.6 ± 1.0 MPa) of the filler increased the adhesive bond strength ca. of 6 % to 11 %. Adding the filler in the form of Polyamide PA microfibres from the textile waste from the process of tyres recycling showed in a positive way.

A type of a fracture surface changed after adding of the filler. The fracture surface of the matrix (the adhesive) was of the adhesive type. At the adhesive bonds (adhesive bonded with the composite system) the fracture surface was of the adhesive type. Results of the adhesive bond strength are visible from Fig. 2, 3 and 4.

It is possible to say in terms of the statistical testing of the influence of different filler concentrations that the concentrations are statistically homogeneous groups. The hypothesis H_0 was certified, i.e. there is no difference in the adhesive bond strength in the significance level 0.05 among single concentrations 0 g, 2 g and 4 g of the filler in the form of the Polyamide PA microfibres from the textile waste from the process of tyres recycling. The statistical comparison (T-test, $\alpha = 0.05$) of the influence of increasing filler concentration on the adhesive bond strength is presented in Tab. 1. It is evident from the statistical comparison of the values that increasing concentration of the filler did not statistically influence the adhesive bond strength.

It is possible to say in terms of the statistical testing of the influence of various numbers of cycles on the adhesive bond strength that the cycles are statistically homogeneous groups. The hypothesis H_0 was certified, i.e. there is no difference in the adhesive bond strength in the significance level 0.05 among single number of cycles (0, 200, 500 and 1000).

The statistical comparison (T-test, $\alpha = 0.05$) of the influence of the number of cycles on the adhesive bond strength is presented in Tab. 2. It is evident from the statistical comparison of the values presented in Fig. 2, 3 and 4 that the number of cycles does not statistically influence the adhesive bond strength.

Also a minimum difference between 30 % and 60 % of the loading from the reference value of the adhesive bond strength is obvious from the results. The average difference in the adhesive bond strength did not exceed 13 %.

Tab. 1. – Statistical comparison of influence of filler concentration on adhesive bond strength (T-test)

Loading from reference value of adhesive bond strength (%)	Number of cycles (-)	H_0: (p>0.05)
100	0	0.3259
60	200	0.2790
60	500	0.4609
60	1000	0.2973
30	200	0.3230
30	500	0.4396
30	1000	0.2922

Symbol O and C mean the outer part of root and the central part of root, respectively

Tab. 2. – Statistical comparison of influence of filler concentration and number of cycles on adhesive bond strength (T-test)

Loading from reference value of adhesive bond strength (%)	Filler concentration (g)	H_0: (p>0.05)
30	0	0.1553
30	2	0.3773
30	4	0.7025
60	0	0.2291
60	2	0.8335
60	4	0.1663

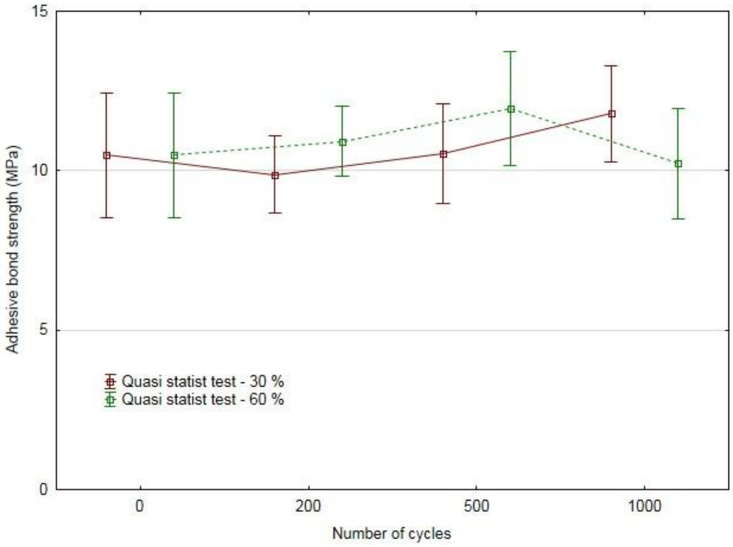

Fig. 2. – Influence of low-cyclic fatigue on adhesive bond strength (adhesive bonds with 0 g of filler)

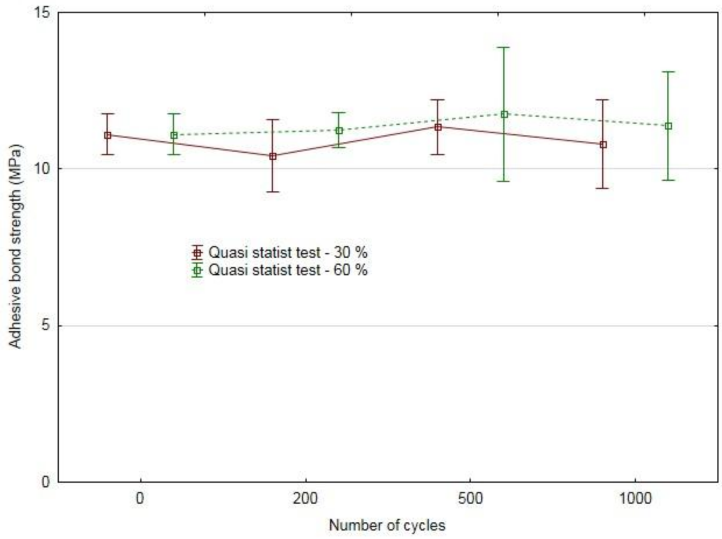

Fig. 3. – Influence of low-cyclic fatigue on adhesive bond strength (adhesive bonds with 2 g of filler)

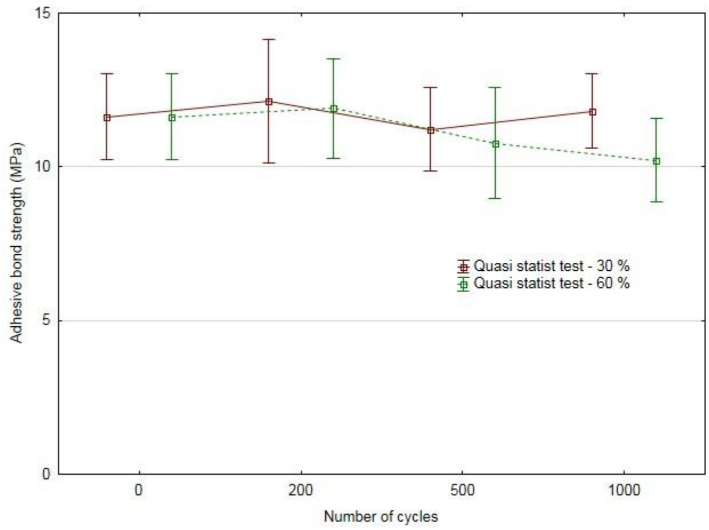

Fig. 4. – Influence of low-cyclic fatigue on adhesive bond strength (adhesive bonds with 4 g of filler)

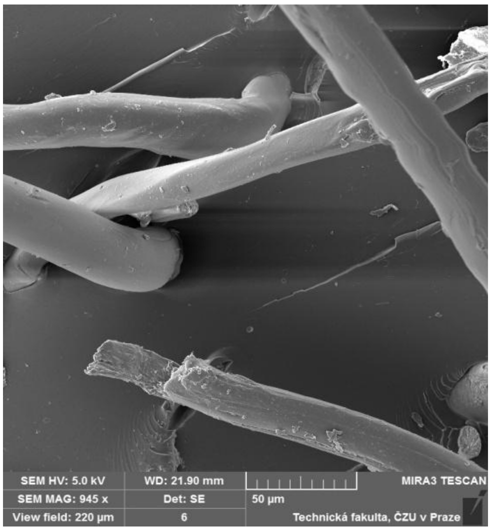

Fig. 5. – Fracture surface of sample (filler: microfibres of Polyamide PA from the textile waste from the process of tyres recycling 2 g: 100 g matrix, good wetting of filler with resin, uneven distribution of filler, secondary electrons)

A strong interaction between the adhesive and particles is evident (Fig. 1). When applying the filler into the resin, the wetting of the filler with the matrix is very important (JÄCKEL, SCHEIBNER, 1991). Results of the experiment also show the irregular stratification of filler microparticles in the matrix (Fig. 5).

CHANG AND YEIH (2001) proved in their experiments that the irregular shape of the filler ensured good interaction between the matrix and the filler. Microfibres of a regular cross-section were used as the filler within the experiment (Fig. 1). The assumption about a negative influence of the filler on the adhesive bond tensile strength was not confirmed (CHO ET AL., 2006). The course of the testing is visible from a diagram of the low-cyclic test. Fig. 6 presents the low-cyclic test at 60 %, which is finished after reaching 200 cycles by the destruction of the adhesive bond.

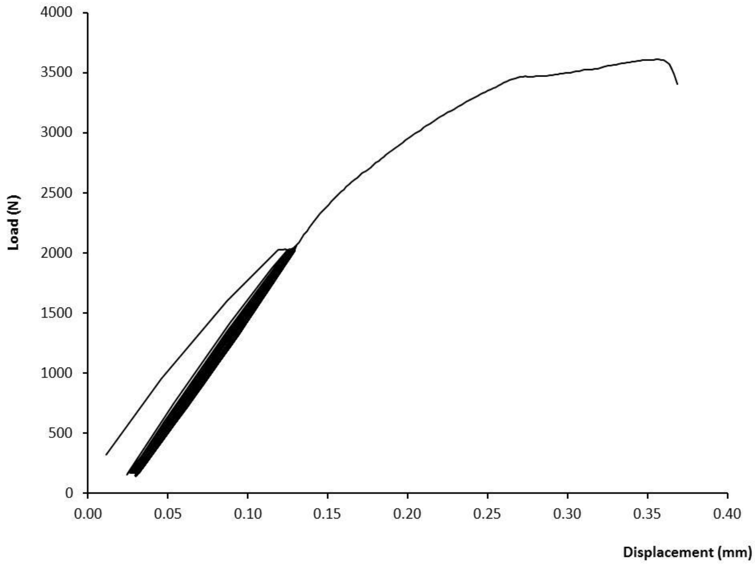

Fig. 6. – Low-cyclic test (60 %, 200 cycles, filler 4 g)

Fig. 7 presents the low-cyclic test at 60 %, which is finished after reaching 500 cycles by the destruction of the adhesive bond. A so called cyclic reinforcement occurred at some bonds after finishing the cycling. This entails an increase of the loading force after the end of the cycling.

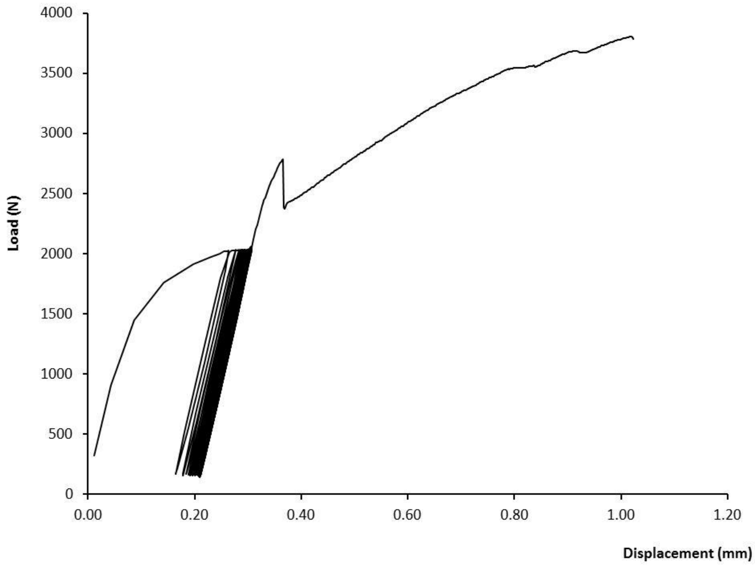

Fig. 7. – Low-cyclic test (60 %, 500 cycles, filler 0 g)

The experiment results did not confirm the assumption that the repeated cyclic loading with high value (i.e. e.g. 60 % (BROUGHTON ET AL., 1999; ŠLEGER, MÜLLER, 2015) of the reference strength of the adhesive bond) can lead to the premature failure of the adhesive bond in a relative short number of cycles. A cause can be a cumulative effect of the cyclic shear loading (ŠLEGER, MÜLLER, 2015).

CONCLUSIONS

Following conclusions can be deduced from performed experiments:

- The influence of the low-cyclic tests on the change of the fracture surface was not proved.
- The microfibres of Polyamide PA from the textile waste from the process of tyres recycling are of good wettability with the matrix.
- Adding the filler in the form of Polyamide PA from the textile waste from the process of tyres recycling showed in a positive way. It came to the increase of 6 % to 11 % of the adhesive bond strength.

It is possible to say in terms of the statistical testing of the influence of various filler concentrations and number of cycles that they both are statistically homogeneous groups, i.e. they do not influence the adhesive bond strength.

Acknowledgement

Supported by Internal grant agency of Faculty of Engineering, Czech University of Life Sciences Prague (Research on mechanical properties of multi-component polymer systems during their preparation, processing and application, 2016:31140/1312/3109).

REFERENCES

1. ACEVEDO, B., FERNÁNDEZ, A. M., BARRIOCANAL, C.: Identification of polymers in waste tyre reinforcing fibre by thermal analysis and pyrolysis. *Journal of Analytical and Applied Pyrolysis*, 111, 2015: 224-232.
2. BARTL, A., HAKL, A., MIHALYI, B., WISTUBA, M., MARINI, I.: Recycling of fibres materials. Process Safety and Environmetal Protection. *Trans. IChemE*, 83, 2015: 351-358.
3. BROUGHTON, W. R., MERA, R. D., HINOPOULOS, G.: Cyclic Fatigue Testing of Adhesive Joints, Test Method Assessment. Project PAJ3 - Combined Cyclic Loading and Hostile Environments 1996-1999, Report No 8, Centre for Materials Measurement & Technology, National Physical Laboratory, Teddington, 1999: 34 pp.
4. CHANG, J. J., YEIH. W. CH.: The effect of particle shape on bond strength improvement of epoxy particle coating composites. *Journal of Marine Science and Technology*, 9, 2001: 153-160.
5. CHO, J., JOSHI, M. S., SUN, C. T.: Effect of inclusion size on mechanical properties of polymeric composites with micro and nanoparticles. *Composites Science and Technology*, 66, 2006: 1941-1952.
6. DOBRÁNSKÝ, J., BARON, P., KOČIŠKO, M., VOJNOVÁ, E.: Monitoring of the influence of moisture content in thermoplastic granulate on rheological properties of material. *Applied Mechanics and Materials*. 616, 2014: 207-215.
7. DOBRÁNSKÝ, J., BARON, P., KOČIŠKO, M., BĚHÁLEK, L., VOJNOVÁ, E.: Solving depressions formed during production of plastic molding. *Metalurgija*, 54(3), 2015: 496-498.
8. FANG, Y., ZHAN, M., WANG, Y.: The status of recycling of waste rubber. *Materials and Design*. 22(2), 2000: 123-127.
9. FERREIRA, C. T., PEREZ, C. A. B., HIRAYAMA, D., SARON, C.: Recycling of polyamide (PA) from scrap tires as composites and blends. *Journal of Environmental Chemical Engineering*. 1, 2013: 762-767.
10. HAFIZ, T. A., ABDEL WAHAB, M. M., CROCOMBE, A. D., SMITH, P. A.: Mixed-mode fracture of adhesively bonded metallic joints under quasi-static loading. *Engineering Fracture Mechanics*, 77, 2010: 3434-3445.
11. HOLEŠOVSKÝ, F., NÁPRSTKOVÁ, N., NOVÁK, M.: GICS for grinding process optimization. *Manufacturing technology*. 12, 2012: 22-26.
12. JÄCKEL, M., SCHEIBNER, W.: Boundary layer induced modification of thermal and mechanical properties of epoxy resin composites. *Cryogenics*, 31(4), 1991: 269-272.
13. KELLY, G.: Quasi-static strength and fatigue life of hybrid (bonded/bolted) composite single-lap joints. *Composite Structures* 72, 2006: 119-129.
14. KNAPČÍKOVÁ, L., MONKA, P., HLOCH, S.: Composite Materials Reinforced with Fabric from Used Tyres. *Journal of Manufacturing and Industrial Engineering*. 12(3-4), 2014: 20-24.
15. MESSLER, R., W.: Joining of materials and structures from pragmatic process to enabling technology. Burlington, Elsevier, 2004: 816 pp.
16. MÜLLER, M.: Influence of surface integrity on bonding process. *Research in Agricultural Engineering*, 57, 2011: 153-162.
17. MÜLLER, M., HERÁK, D., VALÁŠEK, P.: Degradation limits of bonding technology depending on destinations Europe, Indonesia. *Tehnicki Vjesnik-Technical Gazette*, 20(4), 2013: 571-575.
18. MÜLLER, M., VALÁŠEK, P.: Comparison of variables influence on adhesive bonds strength calculations. *Manufacturing Technology*, 13(2), 2013a: 205-210.
19. MÜLLER, M., VALÁŠEK, P.: Influence of liquid contaminants on strength of adhesive bond on basic of two-component epoxy adhesive. In: *5th International Conference on Trends in Agricultural Engineering 2013*, Prague, Czech University of Life Sciences Prague, 2013b: pp. 441-445.
20. MÜLLER, M.: Setting of causes of adhesive bonds destruction by means of optical analysis. *Manufacturing Technology*, 14, 2014: 371-375.
21. MÜLLER, M.: Research on surface treatment of alloy AlCu4Mg adhesive bonded with structural single-component epoxy adhesives. *Manufacturing Technology*, 15(4), 2015a: 629-633.
22. MÜLLER, M.: Hybrid composite materials on basis of reactoplastic matrix reinforced with textile fibres from process of tyres recyclation. *Agronomy Research*, 13(3), 2015b: 700-708.
23. MÜLLER, M., NOVÁK, P.: Researches of liquid contaminants influence on change of hardness of agricultural tyre tread. *Research in Agricultural Engineering*, 61(1), 2015: 14-20.
24. NOVÁK, M.: Surfaces with high precision of roughness after grinding. *Manufacturing technology*. 12, 2012: 66 -70.
25. PARRES, F., CRESPO-AMORÓS, J. E., NADAL-GISBERT,

A.: Mechanical properties analysis of plaster reinforced with fiber and microfiber obtained from shredded tires. *Construction and Building Materials*, 23, 2009: 3182-3188.
26. ŠLEGER, V., MÜLLER, M.: Quasi Static Tests of Adhesive Bonds of Alloy AlCu4Mg. *Manufacturing Technology*, 15(4), 2015: 694-698.
27. TARANU, N., BANU, D., OPRISAN, G., BUDESCU, M., BEJAN, L.: Strengthening of thin reinforced concrete slabs with composite strips. *Romanian Journal of Materials,* 43(1), 2013: 3-13.
28. VALÁŠEK, P., MÜLLER, M.: Abrasive wear in three-phase waste-based polymeric particle composites. *Tehnicki Vjesnik-Technical Gazette,* 12(2), 2015: 257-262.

Corresponding author:
doc. Ing. Vladimír Šleger, CSc., Department of Mechanical Engineering, Faculty of Engineering, Czech University of Life Sciences Prague, Kamýcká 129, Praha 6, Prague, 16521, Czech Republic, phone: +420 22438 3173, e-mail: sleger@tf.czu.cz