TWO-BODY ABRASION OF FE-BASED PARTICLE EPOXY COMPOSITES - EXPERIMENTAL APPROACH

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Abstract
It is possible to efficiently change the mechanical characteristics of polymer materials and simultaneously optimize its cost via micro particle fillers. Inclusion of hard inorganics particles on the basis of metal powders mostly leads to increase of hardness and wear resistance of polymers. These properties can be used in many application areas, for example in agriculture. Optimization of adhesive and cohesive characteristic is possible with these fillers under certain conditions. This paper describes the possibility of filling the reactoplastics represented with epoxy resin filled with hard inorganic powder on the Fe-basis, when the wear resistance and fundamental adhesive characteristics are evaluated. Inclusion of Fe-based powder led to increased resistance against to two-body abrasion of 69% while the adhesive characteristics stayed preserved.

Key words: adhesion, metal powder, strength, wear.

INTRODUCTION

The inclusion of inorganics fillers into the polymer matrix creates the composite system. It is possible to define these systems as polymer particle composites. It is possible to utilize inherent corrosion resistance of polymer particle composites in combination with their excellent mechanical properties and low density in various industry areas (PERREUX AND SURI, 1997). Polymer matrix can by created by reactoplastic or thermoplastic. Reactoplastics have a wide spread in the area of experimental description of composite behavior – they have excellent mechanical properties and the preparation of experiment does not require complicated technological equipment comparing to thermoplastics. Moreover, many reactoplastics, as an epoxy resin for instance, are used in the area of structural adhesive bonding, where the fillers can play a significant factor in optimization (MÜLLER ET AL., 2008; VALÁŠEK AND MÜLLER, 2015; RUGGIERO ET AL., 2015B).

Utilization of aluminium powder in the shape of spherical particles (smaller than 50 µm) for description of shear characteristics on the aluminium sheets is described by KAHRAMANA ET AL. (2008), who state, that even though the finite element analysis shows higher stresses at the adhesive-metal substrate interface, actual failure occurs within the adhesive indicating that the strength of adhesion to the metal substrate surface is stronger than the strength of the adhesive itself. For optimization of the adhesive strength of the joints can be used also different particle on the metal basis. (KILIK AND DAVIES, 1989).

KAVAK AND ALTAN (2012) for example state, Sn-Pb powder. OHSAKO AND YOSHIZAWA (2011) state that it is possible to use the inorganics particles for optimization on the interface – interaction between composite system and adherent to whom the system is applied. This interaction is as important as the ability of matrices to wet the particles, thus mutual semi phase interaction (ZHAI ET AL., 2006).

It is possible to use also hard inorganic particles on the Fe-basis for optimization of plastics matrices (GUNGOR, 2007; GAO ET AL., 2016). GUNGOR (2006) states the comparison of Fe/HDPE composite with the mechanical properties of unfilled HDPE, Fe filled polymer composites showed lower yield and tensile strength, % elongation, and Izod impact strength, while the modulus of elasticity and hardness of the composites were higher than those of HDPE. Interesting possibility of usage ferrite particles are their magnetics properties, which can be according to FULCOA ET AL. (2016) used in polymer matrices in the combination with different kinds of fillers, for example with fibers (ZOIS ET AL., 2003).

TIAN ET AL. (2014) also use Fe-Si particles in the interaction with epoxy resin to optimize magnetic properties of matrix with different temperatures (-60°-

140 °C), but at the same time they state, that impact strength of Fe-Si/epoxy composites increases with increasing the Fe-Si content.

Presence of hard inorganic particles in polymer material increases wear resistance (RUGGIERO ET AL., 2015A). Morphology of particles ranks among significant parameters, which effect the adhesion between filler and the used resin – CHENG ET AL. (2002) state as the most important parameter the roughness of particles and they say that important is also the shape of particles and also their size (LAU ET AL., 2006). Filled reactoplastic in form of so called liquid metals is possible to use for renovation of functional parts of machines and equipment (VALÁŠEK ET AL., 2015).

This experiment has as objective to describe the resistance of particle composite on the basis of Fe-based powder and epoxy resin (Fe-based/Epoxy) to two-body abrasion. Description of adhesive characteristic is important from the viewpoint of interaction of composite and surface to which is the system applied. Remaining mechanical properties and picture analysis of fracture areas are shown to clarify the behavior of composite.

MATERIALS AND METHODS

Special technological procedure (such as vacuum) has not been used for the preparation of composite mixtures. Procedure of preparation was chosen with respect to minimization of resulting price of composite and with respect to practise where the composites – liquid metals – determined for renovation of machine parts are applied straight on the renovated area.

Two-component epoxy resin Glue Epox Rapid was used. The particles on the Fe basis were mechanically mixed into the resin. Composites were prepared with different volume percentage of filler because of determination of effect of particle inclusion on mechanical properties as: 5%, 10%, 15%, 20% and 25%.

For optical and chemical analysis of Fe-based powder and optical analysis of fracture areas was used electron microscope - SEM - (Tescan Mira 3 GXM) equipped with an energy dispersive X-ray on the surface of each sample using vacuum coating (Quorum Q150R ES) which scan system (Oxford X-MaxN). For analysis of adherent surface was used fluorescence confocal microscope (Axio Imager Zeiss LSM 800).

The two-body abrasion was tested on a rotating cylindrical drum device with the abrasive cloth of the different grain size (P120, P220 and P400 - Al_2O_3 grains) according to the standard CSN 62 1466. The testing specimen is in the contact with the abrasive cloth and it covers the distance of 60 m. During one drum turn of 360° it is provoked the testing specimen left above the abrasive cloth surface. Consequent impact of the testing specimen simulates the concussion. The pressure force is 10 N. The mean of the testing specimens was 15.5 ± 0.1 mm and their height was 20.0 ± 0.1 mm. The mass decreases were measured on analytic scales weighing on 0.1 mg. The volume decreases were calculated on the basis of the found out volume and the density of the composite systems. The highest temperature value observed in the interface of the testing sample and the abrasive cloth was recorded by thermal camera. The hardness of test specimens was measured by the method Shore D (CSN EN ISO 868).

Adhesion characteristics were assessed on aluminium adherents with a thickness of 1.5 mm in accordance with the standard CSN EN 1465. The surfaces of aluminium sheets were blasted using the synthetic corundum (Al_2O_3) of the fraction F80 under the angle of 90°. Then the surface was cleaned and degreased using perchlorethylene and prepared to the composite application. Adequate surface treatment of adherents is required before applying resins (VALÁŠEK AND MÜLLER, 2014). The lapping was according to the standard 12.5 ± 0.25 mm (see Fig. 1).

For statistical comparison the T-test and ANOVA were used when the zero hypothesis H_0 ($p > 0.05$) states an agreement of the statistical sets of data.

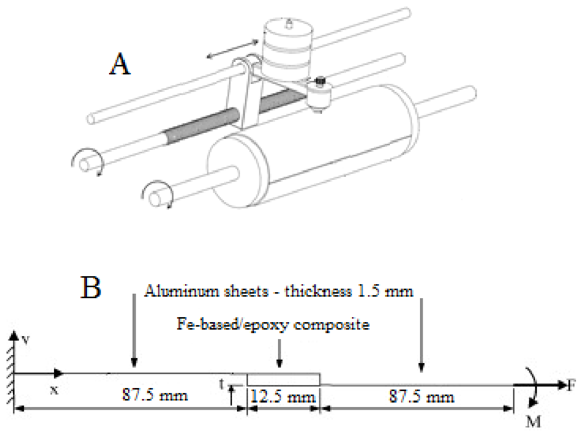


Fig. 1. – a) Schema of equipment for two-body abrasive wear testing, b) Model of lap-shear testing

RESULTS

Specification of fillers (particles) before application into the matrix, i.e. determination of chemical content and morphology of particles, is essential from the point of view of understanding the semi phase interaction. Specific chemical composition of particles was determined experimentally by the Oxford X-MaxN EDS system, resolution at 5.9 keV – 124 eV (Fe 98.2%, see Fig. 2).

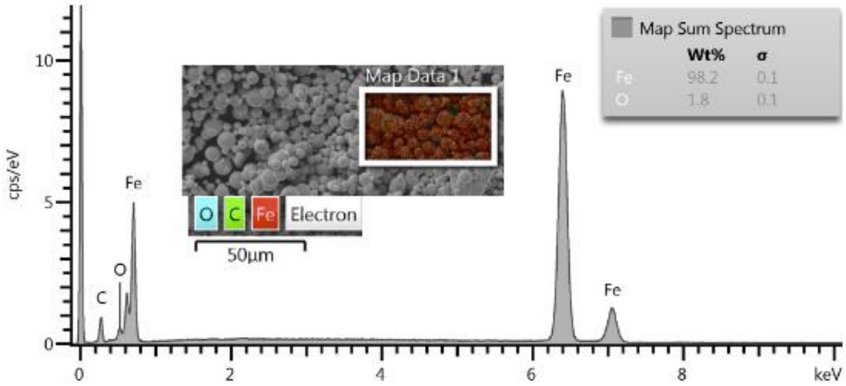


Fig. 2. – Results of EDS particle analysis of Fe-based powder

Powdery density of Fe-based powder is from 3.0 to 5.0 g·cm^{-3}, pycnometrics density 7.0 g·cm^{-3}. Perfect wetting of filler with resin (1.15 g·cm^{-3}) is considered when preparing the composite systems, therefore the pycnometrics density is used for calculations (theoretical density). Dimension of particles was determined via optical analysis (see histogram Fig. 3-a). Particles evinced the spherical shape, see image from electronic microscope (Fig. 3-b).

Among significant qualitative factors of composite systems ranks the porosity *P*, which calculation follows from the comparison of theoretical and real density of composite. Higher porosity shows the excessive presence of air bubbles or bad distribution of the filler (see Tab. 1). Another mechanical property which can be in correlation with assessed wear resistance is hardness (see Tab. 1) - stated properties were assessed on testing samples for resistance to abrasive wear, hardness was assessed in their sedimented parts. Presence of Fe-based powder in epoxy resin increased the hardness according to Shore D from the value 89.63 ± 1.06 (0%) up to value 93.42 ± 1.32 (25%).

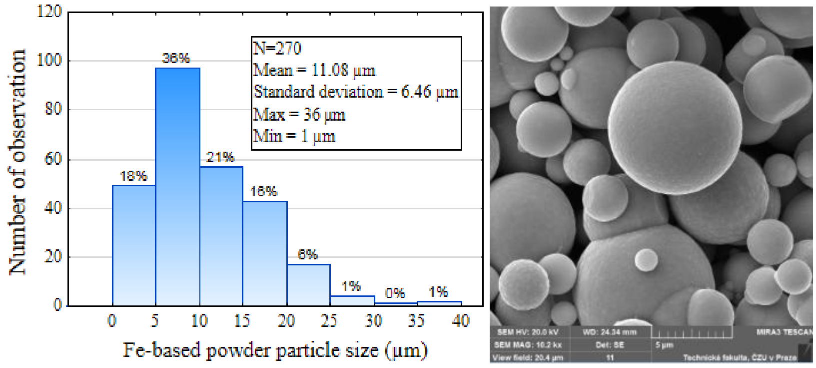


Fig. 3. – a) Histogram (left), b) Spherical Fe-based particles (right)

Tab. 1. – Density, porosity and hardness

Properties	0%	5%	10%	15%	20%	25%
Theoretical density (g·cm^{-3})	1.15	1.44	1.71	2.03	2.32	2.61
Porosity (%)	0	5.6	6.5	4.9	6.0	7.3
Hardness Shore D (-)	89.63	91.05	91.13	92.65	92.62	93.42
Standard deviation (-)	1.06	1.70	1.14	0.91	1.47	1.32
Variation coeficient (%)	1.2	1.9	1.3	1.0	1.6	1.4

The fluorescence confocal microscope Axio Imager Zeiss LSM 800, objective with magnification 10x and numerical aperture 0.45 was used for description of surface of aluminium sheets before application of composite system (see Fig. 4). Significant parameters of roughness measured by described procedure states Tab. 2 according to standard ISO 25178.

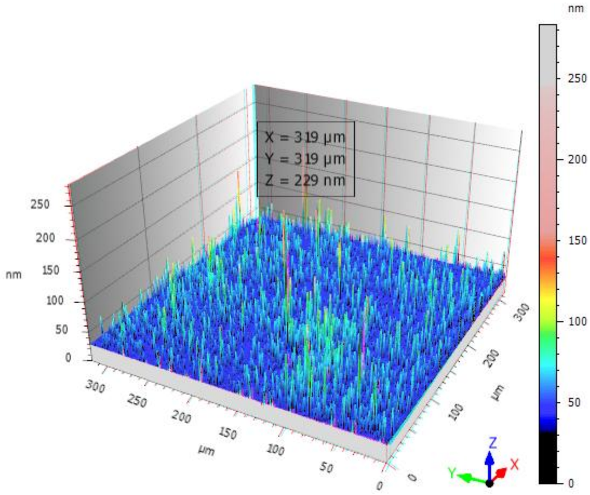


Fig. 4. – Topography of surface of Al sheet before application of composite

Tab. 2. – Roughness parameters (ISO 25178)

Properties	Mean	SD	Min	Max
Arithmetical mean height of the surface Sa (nm)	16.6	13.9	1.89	42.6
Maximum height of the surface Sz (nm)	255	0.53	251	255
Maximum height of valleys Sv (nm)	12.1	11.6	1.0	36.0
Maximum height of peaks Sp (nm)	243	11.6	219	254
Root mean square height of the surface Sq (nm)	28.7	19.3	6.10	61.5

Fig. 5 a, b and c represents the cut of the bonded joint via SEM analysis. Very good wetting in the interface of composite system and adherent is visible in the cut of the joints. Good interaction of particular phases and whole composite system with adherent is crucial for corresponding mechanical properties.

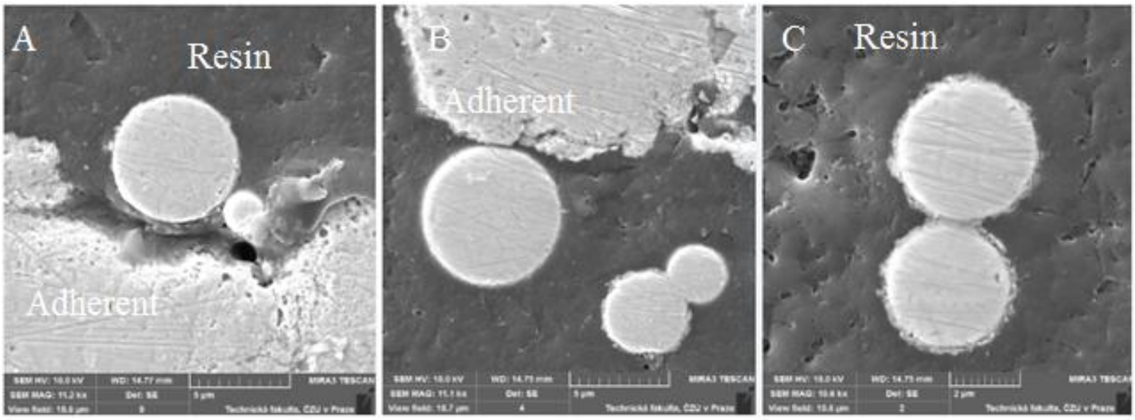


Fig. 5. – Cut of the bonded joint (SEM): a - Wetting with presence of bubble, b - Perfect wetting of adherent and particles, c - Wetting of particles

Resin without filler reached shear strength 8.82 ± 0.44 MPa on the aluminium adherent. From statistical analysis provided with T-test is possible to confirm the null hypothesis H_0: $\mu 1=\mu 2$ up to concentration of filler 15% ($p > 0.06$). It is possible to state that increased concentration of filler in the resin (20 and 25%) led to statistically decrease of values in shear strength.

Arrangement of particles of Fe-based powder in the composite system on the cohesive fracture is pictured via SEM analysis on Fig. 6 a, b and c. Good mutual interaction between particles of Fe-based powder and used epoxy resin is visible from the images.

Tab. 3. – Lap-shear tensile strength

Properties	0%	5%	10%	15%	20%	25%
Shear strength (MPa)	8.82	8.19	8.03	7.98	7.58	7.60
Standard deviation (MPa)	0.44	0.50	0.80	0.72	0.55	0.77
Variation coeficient (%)	5.0	6.1	10.0	9.0	7.2	10.1
T-test (*p*) compared with 0% (-)	1	0.06	0.08	0.06	0.00	0.00

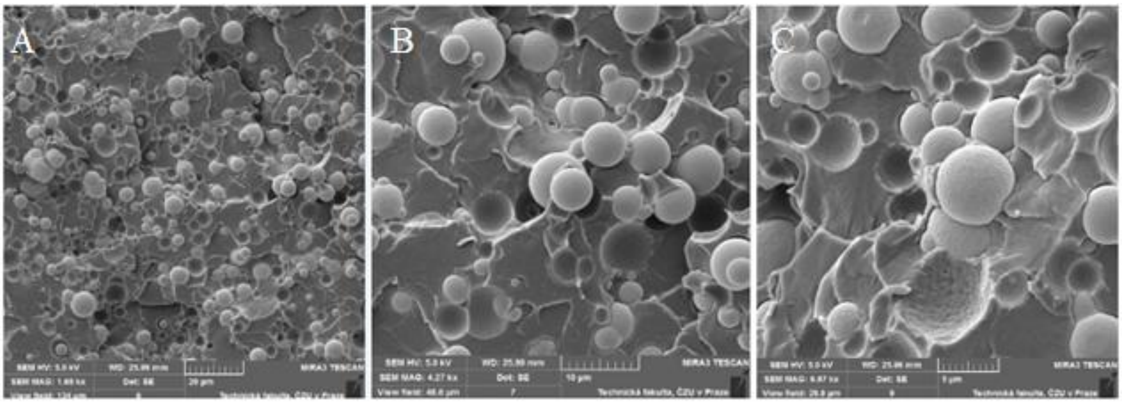
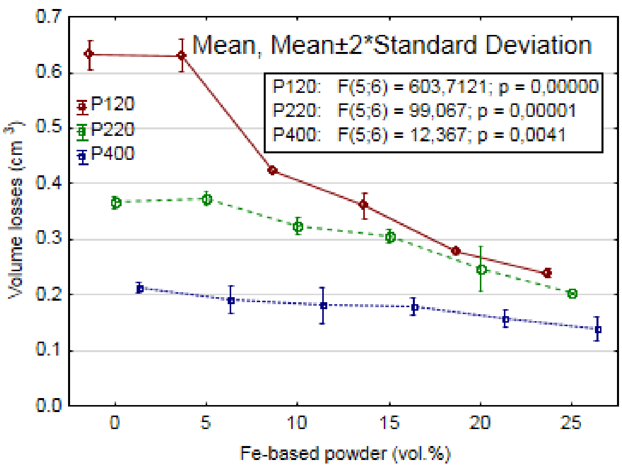


Fig. 6. – Analysis of cohesive fracture (SEM): a) MAG 1.68 kx, b) MAG 4.27 kx, c) MAG. 6.97 kx

Results of resistance to two-body abrasion shows Fig. 7-a, from the graph is visible that presence of Fe-based powder increased the ability of the system to resist to two-body abrasion. Volume losses of unfilled resin corresponded to 0.631 ± 0.009 cm^3 (P120), 0.367 ± 0.004 cm^3 (P220) and 0.213 ± 0.003 cm^3 (P400). During the test of resistance to abrasive wear was measured temperature of testing sample via thermal camera, the temperature did not exceed 41.4 °C (Fig. 7-b).



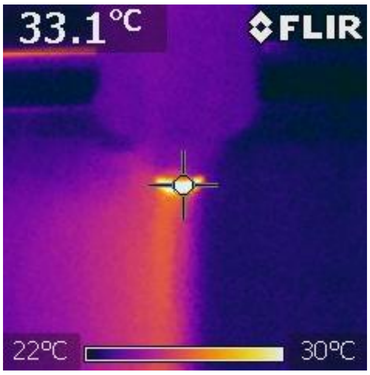


Fig. 7 – Resistance to two-body abrasion: a) Results (left), b) Image from thermal camera: course of the test (right)

DISCUSSION

Chosen technological procedure of preparation of composites without using the vacuum follows from the practical requirements, i.e. simple and fast application on the functional surfaces, however this procedure has an impact on demonstrable presence of pores in the material (on an average 6%). High porosity negatively affects the assessed mechanical properties, limitation of porosity can lead to optimization of monitored characteristics. On the scale of Shore D was the increase of hardness by inclusion of Fe-based powder about 2.6% and it was in compliance with conclusions of many authors (SATAPATHY AND BIJWE, 2002; VALASEK ET AL., 2015) who describe the inclusion of hard inorganic microparticles into the polymers. Resistance to abrasive wear increased with increasing amount of filler in the matrix, which is in compliance with many authors (SATAPATHY AND BIJWE, 2007; XUE QUNJI AND WANG QUIHA, 1997), who used hard inorganic particles on the basis of corundum, silicon carbide or powder on the metal basis to increase the resistance of polymers. Ability to increase the resistance to abrasion was the most manifested on cloth with bigger abrasive grains (P120), where the volume losses decreased up to 62% (for cloth P220 was decrease 45% and for cloth P400 was decrease 34%). In the interval with concentration of Fe-based powder in resin 5-15% was not statistically significant decrease of values of shear strength, there was a decrease of values of shear strength max. 15% with higher concentration of the filler.

CONCLUSIONS

Described composite systems preserve adhesive properties assessed via shear strength with inclusion of Fe-based powder up to 15%. There is a slight decrease of this characteristic from this concentration. Inclusion of Fe-based powder increased the hardness and wear resistance. Described properties correspond to requirements of filled polymers used for renovation of functional areas of machines and equipment – there is an increase of resistance to abrasive wear with preservation of adhesive characteristic. Abrasive wear ranks among the most represented kinds of wear in the area of agriculture.

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