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Impact strength behaviour of structural adhesives

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Abstract. A cohesive force at an adhesive bond is one of the limit for a strength of an adhesive bonding. This study is focused on an impact force of an adhesive. Samples without a notch were cast in the casting mould at a laboratory temperature with a normal pressure. The instrumentation microcharpy test equipment was used for the evaluation of the impact force. The samples were tempered at a laboratory temperature, 40 °C and 60 °C. Results showed that the temperature of the specimens influenced the impact strength, the toughness and the maximum deformation of the adhesives. Higher temperature decreased the impact force but it increased the toughness. The hardness Shore D of commercial filled two-component epoxies is comparable. A non-homogeneity of adhesives distinguished for a porosity was found by the investigation of a fracture surface.

Keyword: Deformation, hardness, impact force, impact energy, microcharpy test, temperature.

INTRODUCTION

The production process in various industrial sectors divers, but it usually has one common element and that is forming a bond (Müller, 2011). One of important requirements is, that the products have to reach a sufficient resistance to the dynamic load (Hayashida et al., 2015).

A promising method of creating the bond is the adhesive bonding technology. This technology is often used as a complementary method of joining (Müller, 2013).

According to Messler (2004), the bonding is a process of joining materials with the aid of acting of a chemical agent which is able to keep together these materials via surface attractive forces. Materials which are joined are called adherents, while the bonding factor is an adhesive. The forces, which allow the surface attraction, arise from one or more sources, mostly chemical, but some may be mechanical or even electrostatic. These forces give rise to that what is called an adhesion, i.e. bonding of different materials together (Messler, 2004). The adhesive layer forms the cohesive strength. A summary of forces can be considered as the cohesion. They bind the adhesive particles to each other by a valence mutual action and molecular forces of an attraction (Müller, 2013).

The environmental temperature is a significant factor which influences the behaviour of the adhesive, i.e. polymer (Müller & Valášek, 2012; Messler, 2004). An effective application of adhesive bonds depends also on their resistance to an effect of different temperatures. This means, that their ability to retain their properties during the long-term exposure to increased or decreased temperature is significant for the practical application.

It is not possible to perform a general evaluation of the temperature influence on the adhesive strength and adhesive bonds. The critical decrease of the strength of all adhesives is usually in the range from 60 to 100 °C (Messler, 2004). Hu et al. (2013) discovered, that the long-term exposure of adhesive bonds to increased temperature causes a decrease in the strength.

The temperature is a significant factor which affects the mechanical properties of the adhesive. Influencing of the adhesive occurs already during a storage, a transportation or an application on the adhesive bonded material (Valášek & Müller, 2015). The impact resistance is another significant limit of the effective application of adhesive bonds.

In spite of the increasing use of adhesives in the aircraft and automotive industries the crashworthiness of structures joined with adhesives is still a challenging subject.

Following considerations are made for adhesive bonded structures:

- The impact performance of the bond and whether it is different from the quasi-static performance.
- The capability of any energy absorption of the adhesives can contribute to the impacted system.
- Understanding of the impact loading of the bonds so that they can be designed for such conditions (Hayashida et al., 2015).

There are several dynamic tests enable to simulate the impact force. These dynamics tests are mainly based on the principle of tests according to Izod and Charpy.

Adhesives are sensitive to the dynamic loading and it is proved that the strength decreases with the temperature (Adamvalli & Parameswaran, 2008).

The adhesives filled with a filler are used in the automotive industry. The reason is particularly the decrease of a price and the increase of the impact strength (Valášek & Müller, 2015).

A substantial change in mechanical properties can be achieved by adding an optimum volume of the filler i.e. the reinforcement (Kahramana et al., 2008). Optimum utility properties of these composites are limited primarily by a risk of a cohesive failure caused by an improper amount and the material of the filler.

Composites can include a reinforcing phase in various sizes. The reinforcement in a form of hard and thermodynamically stable chemical compounds produced primarily as a filler is used for particle composites.

The addition of fillers to the epoxy adhesive does not produce a definite improvement or deterioration in the impact strength according to the research of various authors. E.g. Dadfar & Ghadami (2013) indicate an improvement in the toughness due to the increased content of the rubber modifier. Furthermore, the addition of aluminium oxide fillers has on the contrary a negative effect (Kejval & Müller, 2013).

Another feasible method for the elimination of the brittleness of polymers (reactoplastics) is the waste utilization from the process of tyre recovery (Müller, 2015).

The polyamide fibres from the process of the tyres recovery are recommended to remove the brittleness of the materials (Parres et al., 2009). This assumption was certified within the research. The impact strength significantly increased when adding the filler.

The aim of this research was to evaluate mechanical properties of structural adhesives used in the automotive industry.

MATERIALS AND METHODS

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

- Bison metal epoxy (marked BM) – Two-component epoxy adhesive filled with metal microparticles, temperature range -60 to 100 °C.
- Alteco 3 Ton epoxy adhesive (marked A30M) – Two-component epoxy adhesive filled with metal microparticles, temperature range -20 to 120 °C.
- Alteco 3 Ton quick adhesive (marked A4M) – Two-component epoxy adhesive filled with metal microparticles, temperature range -20 to 120 °C.
- Perma oxy 5 minutes (marked PA) – Two-component methylmetacrylate adhesive, temperature range -50 to 120 °C.

Mechanical properties of adhesives evaluated in this paper were – impact force, elongation, hardness.

The experimental procedure of the microcharpy test contains a few steps: Making samples of the microcharpy type – width 5 mm, height 5 mm and length 27 mm, specimens for the impact test are without a notch. The samples were cast in a casting mould which was made from Lukapren N.

Samples without the notch were cast in the casting mould at a laboratory temperature with a normal pressure. The instrumentation microcharpy test equipment was used for the evaluation of the impact force. The samples were tempered at laboratory temperature 22 ± 2 °C, 40 ± 2 °C and 60 ± 2 °C. Test specimens were tempered for 60 minutes to achieve required temperature in the laboratory chamber Memmert UF55 before own testing process.

Instrumented microcharpy tester (Fig. 1) with the nominal energy 25 J was used for the evaluation of the impact force and the impact toughness of adhesives. An instrumental edge with a semiconductor strain gauge was jointed to PCI 1716 card with a maximum samples rate 200k per second (Advantech Company), the software was developed with using Microsoft Visual Basic Express 2010.

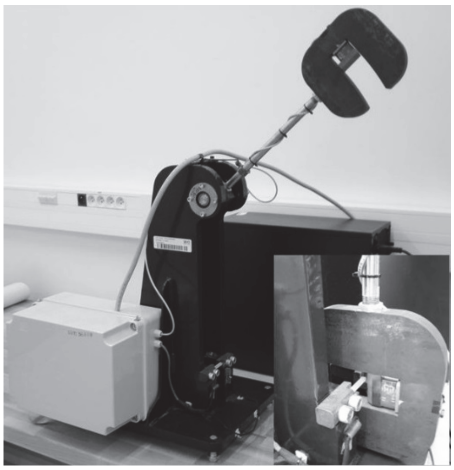

Figure 1. Instrumented Microcharpy test.

The impact energy was determined from discrete output data. The deflection was calculated from Eq. 1:

$$s(t) = v_0.t - \frac{L_p.g}{M_H}\int_0^{t_1}\int_0^{t} F(t)dtdt_1 \tag{1}$$

where:v_0 – impact velocity (m s^{-1}); t – time after impact (s) when deflection is calculated; L_p – length of pendulum (m); M_H – horizontal pendulum moment (N.m); $F(t$ – force (N) at time after impact; $s(t)$ – deflection of sample (m) at time after impact; g – gravitation accelerate (m s^{-2}).

The impact energy (W) was calculated from Eq. 2:

$$W = \sum_{i=0}^{t}\left(\frac{F_i + F_{i+1}}{2}\right).(s_{i+1} - s_i) \tag{2}$$

where: W – impact energy (J); F – force (N); s – deflection (m); t – time (s).

Five samples were used for each of the temperature. Their mean value is presented in the results.

Hardness SHORE D: The material hardness was measured by Shore D i.e. by pressing the tip of the instrument durometer Shito HT. The hardness SHORE D was measured according to the standard CSN EN ISO 868. Test specimens were 5 mm high.

The statistical survey was used for the evaluation – ANOVA F-test in the level of significance $\alpha = 0.05$. As the null hypothesis H_0 it was determined the state, when there is not a statistically significant difference among each comparing data sets in term of their mean values: $p > 0.05$. A coefficient of the variation is defined as a ratio of the standard deviation and the arithmetic mean.

RESULTS AND DISCUSSION

The results focused on the evaluation of the impact force are evident in Fig. 2. The coefficient of the variation among particular tested adhesives at the evaluation of the impact force was in the interval from 1.5 to 18.0%.

It can be stated on the basis of the statistical testing that tested adhesives are statistically non-homogeneous, i.e. there is a difference among tested temperatures which have the influence on the impact force. For adhesives A4M $p = 0.0318$, A30M $p = 0.0000$, BM $p = 0.0023$ and PA $p = 0.0046$. The hypothesis H_0 was not confirmed, i.e. there is a difference in the level of significance 0.05 among particular tested temperatures 20, 40 and 60 °C.

An increase of the average value of the impact force was measured at the temperature 40 °C for adhesives A4M, BM and PA. All adhesives showed a decrease of the impact force at the temperature 60 °C. There was a decrease in the impact force for the adhesive BM due to the increased temperature.

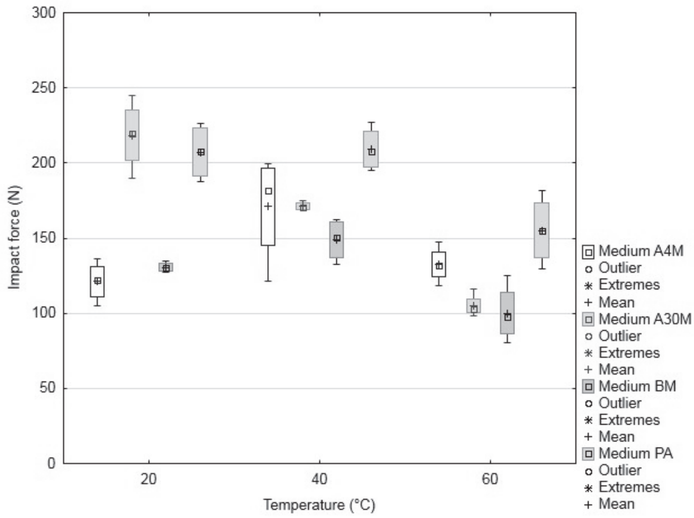

Figure 2. Effect of environmental temperature on impact force for tested adhesives.

Results of the tests focused on the evaluation of the impact energy are evident from Fig. 3. The coefficient of the variation for particular tested adhesives during the evaluation of the impact energy ranged in the interval 7.2 to 23.1%.

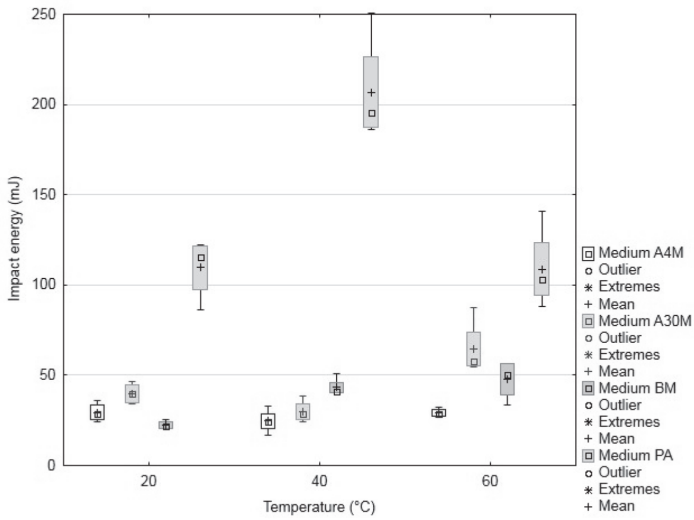

Figure 3. Effect of environmental temperature on impact energy of tested adhesives.

It can be stated from the statistical survey point of view that adhesives A30M, BM and PA are non-homogeneous, i.e. there is a difference among tested temperatures influencing the impact energy. For adhesives A30M $p = 0.0029$, BM $p = 0.0015$ and PA $p = 0.0003$. The hypothesis H_0 was not confirmed, i.e. there is a difference in the level of significance 0.05 among particular temperatures 20, 40 and 60 °C.

The difference in the impact energy due to the different temperature was not statistically proved for the adhesive A4M ($p = 0.3711$). The hypothesis H_0 was confirmed, i.e. there is not a difference in the level of significance 0.05 among particular tested temperatures 20, 40 and 60 °C. It is possible to state with a respect to the statistical testing that tested adhesives are statistically homogeneous groups.

The increase of values of the impact energy was measured for adhesives A30M, A4M and BM due to the effect of the temperature. The increase of the impact energy was measured for the adhesive PA (methylmetacrylate adhesive) at the temperature 40 °C and a subsequent decrease followed at the temperature 60 °C.

Tested samples evince a behaviour common for the brittle material, i.e. reactoplastics at the laboratory temperature and at the temperature 40 °. Spreading of an unstable crack propagation in a cross-section of the sample occurred after reaching the maximum impact force. There was an increase of deformation values in the interval from 47 to 80% for the testing temperature 60 °C (for tested adhesives with the filler, i.e. A4M and A30). There was also a significant decrease of values of the impact force.

There is evident an effect of the impact force and the time of different tested adhesives in the Figs 4, 5, 6 and 7. The laminating is also evident in particular layers of the tested sample from the progress of the graphic dependence visible from Figs 4, 5, 6 and 7. After the failure of one layer the following layer deforms until the failure occurs in the whole cross-section. It is possible to characterise the whole process as a failure of first layer distinguished by a decrease and subsequent increase of the impact force in the deflection. This state repeats until reaching the local maximum. A typical course of given dependence is presented in Figs 6 and 7.

The significance of the temperature factor at the application of the adhesive bonds was certified (Messler, 2004). Increased temperature influences not only the fall of the adhesive strength, but it also decreases the impact force (Hu et al., 2013). The conclusions of authors Müller et al. (2015) on the sensitivity of adhesives to the dynamic loading were also confirmed. It is also possible to agree with their results stating that the force decreases owing to the increased temperature (Adamvalli & Parameswaran, 2008).

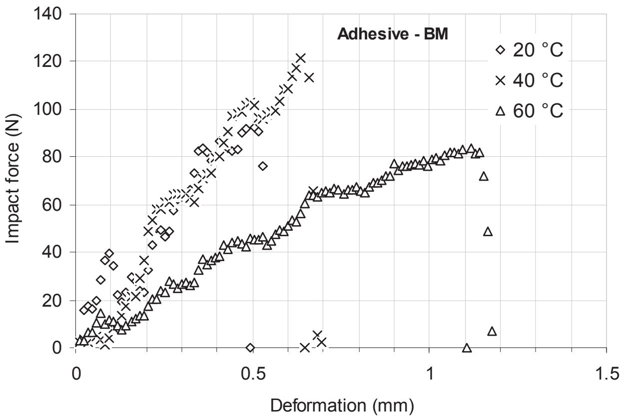

Figure 4. Dependence between impact force and deflection of tested adhesive BM at various temperatures.

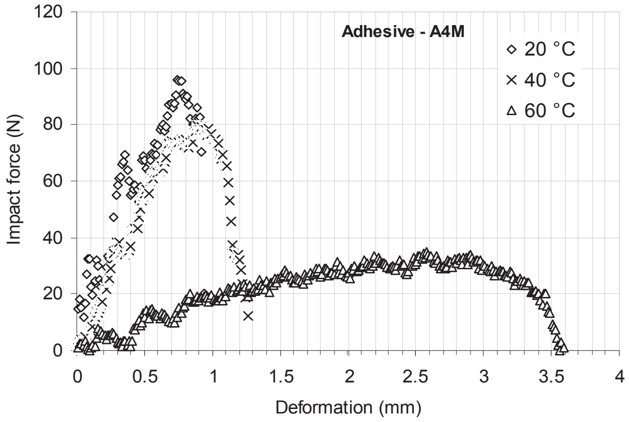

Figure 5. Dependence between impact force and deflection of tested adhesive A4M at various temperatures.

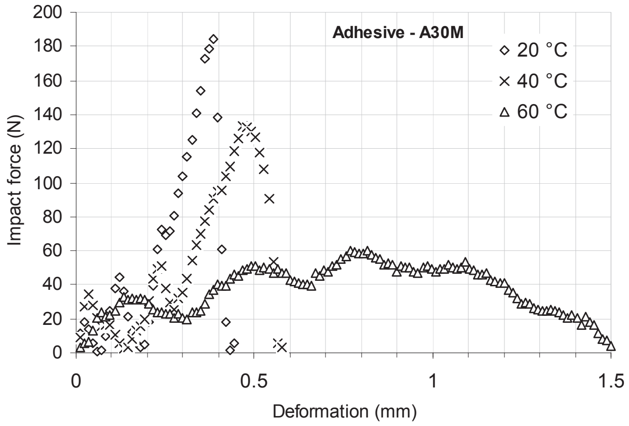

Figure 6. Dependence between impact force and deflection of tested adhesive A30M at various temperatures.

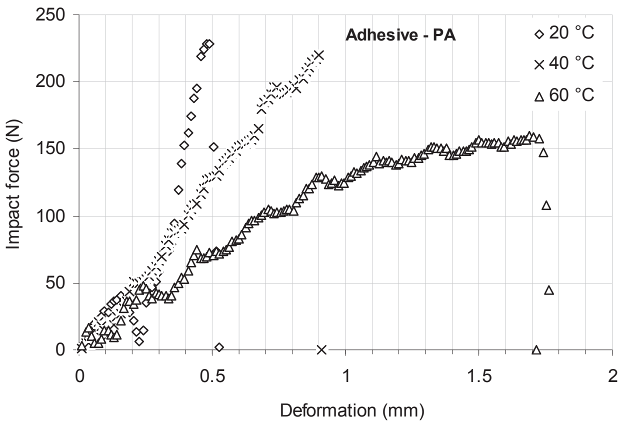

Figure 7. Dependence between impact force and deflection of tested adhesive PA at various temperatures.

The results of this experiment confirmed the assumption that the decrease of mechanical properties of adhesive bonds occurs with the increasing temperature, i.e. mainly the decrease of the strength of the adhesive bond occurs. The instrumental Charpy test has shown that the comparison of adhesives from the point of view of values of the impact energy can be deceptive. But if it is compared with the impact strength, we can obtain significant data for an engineering.

Fig. 8 shows the results of the adhesive hardness measured at the laboratory temperature 20 °C. Two-component epoxy adhesives (A30M, A4M and BM) reached higher values of the hardness Shore D comparing to the methylmetacrylate adhesive PA. The difference was 41.3 to 43.4%. The reason is, that two-component epoxy adhesives are filled with filler.

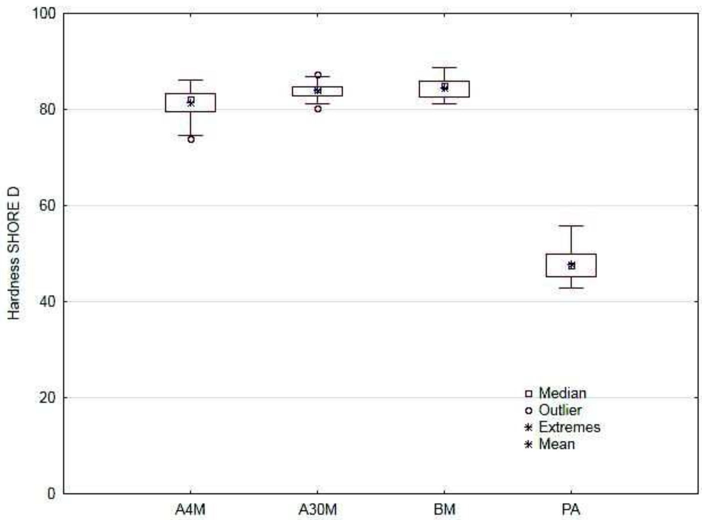

Figure 8. Hardness of tested adhesives at laboratory temperature.

When comparing the results of the hardness Shore D and the impact force it is apparent, that the adhesive with smaller hardness reached higher values of the impact strength. Similar results were observed also when adhesives were tested on the machine Dynstat characterized by an analogous principle of testing as according to Charpy.

Considering the higher complexity of the failure process during the Charpy test of adhesives the scanning electron microscopy was used in the fracture surface. From the picture of the fracture surface of the filled two-component epoxy adhesive it is possible to indicate a correct connection between the adhesive and the filler (Figs 9 and 10). It was found, that the fracture surface is rough and irregular. Also the non-homogeneity of the adhesive, which is distinguished for its spherical shape, is evident from the fracture surface. These are air bubbles which came into being during a process of mixing of the two-component epoxy adhesive and during a curing process (Fig. 9).

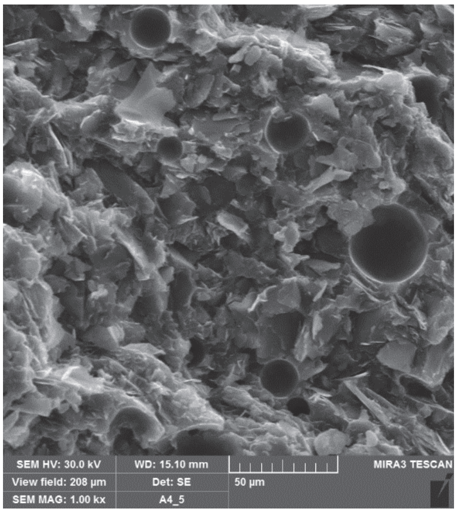

Figure 9. Fracture surface of sample – adhesive A30M, secondary electrons.

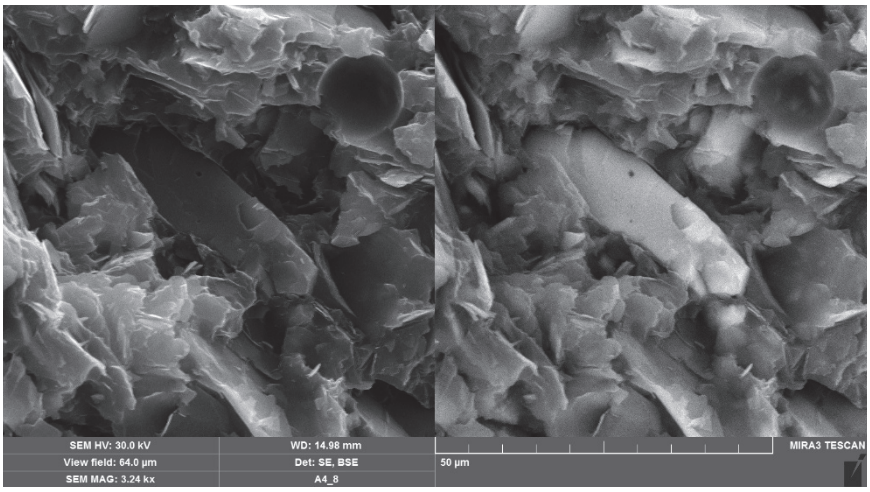

Figure 10. Fracture surface of sample – adhesive A4M, left-secondary electrons, right-back scattered electrons.

CONCLUSIONS

Results of measurements were obtained at the device Microcharpy test which is visible in Fig. 1. It is a suitable method for a characterisation of the dynamic loading of adhesives. It is possible to state these conclusions on the basis of obtained data:

- results showed that the temperature of the specimens influenced the impact force, the toughness and the maximum deformation of the adhesives. Higher temperature decreased the impact force but it increased the toughness. This result should be taken in account at the structure, i.e. in the real application of adhesive bonds,
- the hardness Shore D of commercial filled two-component epoxies is comparable,

- the non-homogeneity of adhesives distinguished for a porosity was found by the investigation of the fracture surface.

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