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Research on influence of loading speed of structural two-component epoxy adhesives on adhesive bond strength

Müller, Miroslav^{a*}, Valášek, Petr^a, Ruggiero, Alessandro^b, D'Amato, Roberto^c

^aCzech University of Life Sciences Prague, Faculty of Engineering, Department of Material Science and Manufacturing Technology, Prague, Czech Republic

^bUniversità di Salerno, Department of Industrial Engineering, Salerno, Italy

^cUniversidad Politécnica de Madrid, Departamento de Ingeniería Mecánica y Construcción, Madrid, Spain

Abstract

Adhesive bonds belong among a prospective technology of materials connecting, they are applied in many industrial branches when single applications use many advantages of these constructional bonds among which also good shear strength can be ranked. Just the shear strength of the adhesive bond belongs among factors determining an applicability of single adhesives. Standards describing the experimental determination of the adhesive bond shear strength specify the time which is needed for reaching the bond failure. For an extension of adhesives specification for given application the definition of the shear strength can be spread also of other speeds. The paper deals just with the determination of the time influence which is necessary for breaking a steel test adherent on the resultant shear strength of structural adhesives. An electron microscopy was used for an evaluation of fracture surfaces. The aim of the performed experiment is to describe the influence of changeable rate of deformation of the adhesive bond on the shear strength. From the result of the constructional carbon steel S235J0 testing the increase of the breaking strength and yield strength at increasing loading speed is obvious. At increasing loading speed the fall of the elongation of the adhesive bonded material occurred. From the results it is obvious that the loading speed of the adhesive bonds has a positive influence on the strength results of tested adhesive bonds. It came to the increase of the adhesive bond strength up of 21-57% at the speed 500 mm·min⁻¹.

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1. Introduction

The adhesive bonding technology can be ranked among constantly developing method of the material connecting which manages to compete with classical methods of the material connecting. However, when applying the adhesive bonds it is necessary to take into regard their advantages and disadvantages. Some of the disadvantages are a necessity of the surface treatments before putting the adhesive on the adherent, low peel strength and low resistance to temperature changes. When applying the adhesive bond a minimum increase of a mass occurs, a character of the adhesive bond can be optimized as a follow-up to the application requirements (damping of vibrations etc.), different types of materials can be adhesive bonded without a thermal influence of a basic material. [1-2]

In single industrial branches the production process is very various. One element is common, namely the joint creation with contemporary monitoring of the whole production process to follow simplicity and effectiveness. That is the reason of connection between continual development and searching of new prospective technologies. In this way the production process will be easier.

* Miroslav Müller. Tel.: +420 224 383 261.
E-mail address: muller@tf.czu.cz

On the basis of this presumption it was possible to pick up again on research and conclusions published in contemporary scientific papers. The performed of the tests according to the standard CSN EN 1465 (adhesives – Determination of the tensile lap-shear strength of rigid-to-rigid bonded assemblies) was the basis of bonded joints laboratory testing. According to the report elaborated by Broughton and Mera from “Centre for Materials Measurement & Technology” [3] the above mentioned test is the most used method of destructive tests having the relationship to the concrete manufacturing program. The authors performed tests according to the standard BS EN 1465: 1995 “Adhesives – Determination of Tensile Lap-Shear Strength of Rigid-to-Rigid Bonded Assemblies”, which is identical with the standard CSN EN 1465.

It is obvious from the experimental results that the loading speed influences the increase of the adhesive bond strength of one-component epoxy adhesives used in automotive industry [4].

The aim of the performed experiment is to describe the influence of changeable rate of deformation of the adhesive bond on the shear strength. Performed experiment also evaluates fracture surfaces (by means of the electron microscopy). The experiment also describes a bearing capacity of single adherents depending on the loading speed, i.e. their tensile characteristics.

2. Methods

There is an effort to get closer to the loading of adhesive bonds in a practice with higher strain rate. 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).

As a basic standard CSN EN 1465 is used, there the rate of deformation is specified in the interval of 65 ± 20 seconds till the failure time. The strain rate is on the order of $0.1-1 \text{ mm} \cdot \text{min}^{-1}$ to comply with the limit of 65 ± 20 seconds.

In the laboratory tests three kinds of two-component epoxy adhesives (Loctite Nordbak 7256 (marked A), 3-TON Epoxy adhesive 30 min (marked B) and GlueEpox Rapid (C)) that cure at laboratory temperature ($23 \pm 2 \text{ }^\circ\text{C}$) were used. The use of epoxy adhesives in this experiment was due to their representation as a structural adhesives and huge utilization in industry.

The tests were performed using the steel S235J0 specimens, the surfaces of 1.5 mm thick steel sheets were at first blasted using synthetic corundum fraction F80 under an angle of 90° . Using the profilograph Surftest 301 the following values were determined: $R_a 1.39 \pm 0.19 \text{ } \mu\text{m}$, $R_z 9.3 \pm 1.01 \text{ } \mu\text{m}$.

Then the surface was cleaned and degreased using acetone and prepared for the application. The surface preparation is important and should guarantee good strength on the boundary of adherents [5-14]. An even thickness of the layer of the adhesive was reached by a constant pressure 0.5 MPa. The lapping was according to the standard ČSN EN 1465, $12.5 \pm 0.25 \text{ mm}$.

Within the research also the influence of the adhesive bonded material behaviour depending on the loading speed 1, 5, 10, 20, 50, 100, 300 and $500 \text{ mm} \cdot \text{min}^{-1}$ was observed. Standardized test specimens were prepared for the carbon steel S235J0 by means of the water jet (Fig. 1).

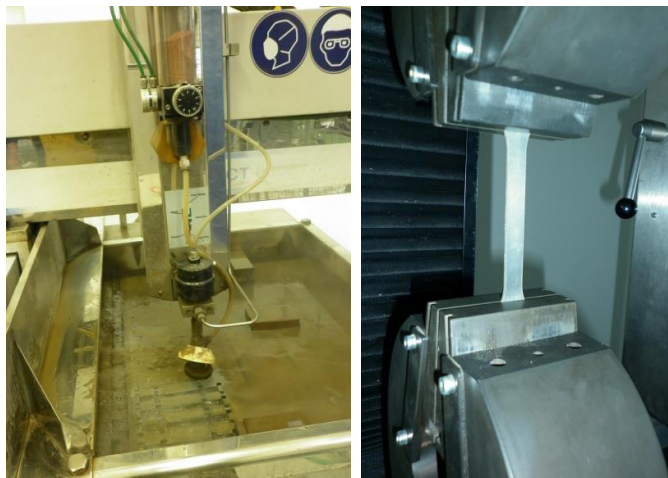


Fig. 1. Static tensile strength test - Production of steel test specimens by means of cutting by water jet (left), test specimen of carbon steel S235J0 (right).

3. Results

Mechanical characteristics of used steel adherent are presented in Fig. 2 and 3. It is obvious from measured values that the loading speed of the adherent influences a breaking strength, yield point and an elongation of the adherent. However, it is necessary to say that the adhesive bond strength is considerably smaller than the breaking strength and yield point. With increasing loading speed of the adherent, R_e , R and ε are increased. Presented conclusions confirm also the results of other

authors. [15, 16]

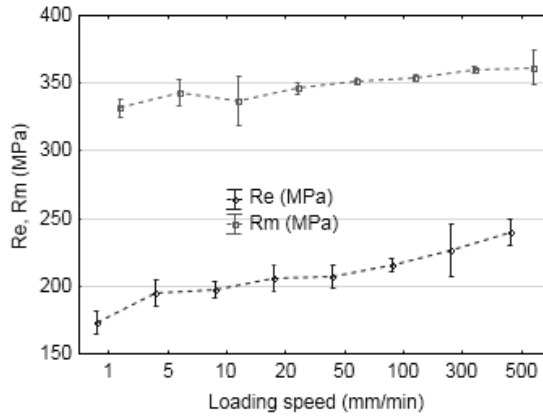


Fig. 2. Tensile characteristics R_e , R_m of used adherent at changeable loading speed.

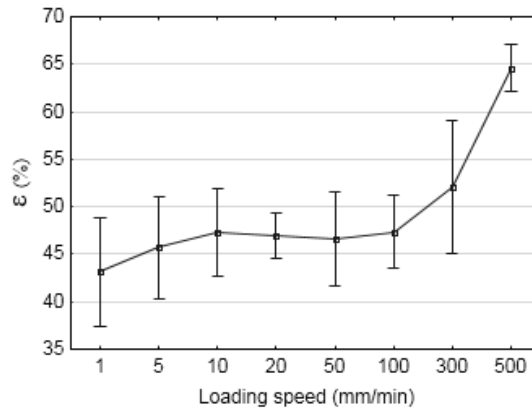


Fig. 3. Tensile characteristic ϵ of used adherent at changeable loading speed.

A graphic presentation of the results of the shear tensile strength of adhesive bonds destruction can be seen from Fig. 4. A course of the dependence was best described by the logarithmic function. The function type was derived from the correlation field, which was formed by the cross points of the dependent and independent variables.

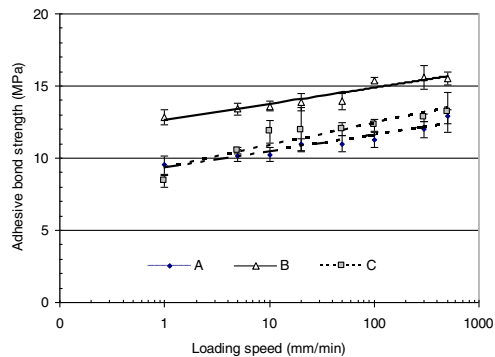


Fig. 4. Influence of loading speed on adhesive bond strength – adhesive A, B, C.

For the correct evaluation it is also important to determine the determination index R^2 . It is the problem of the correlation analysis. The values of the determination index can be from 0 to 1. The functions presented in Fig. 4 are determined by equations in table 1.

Tab. 1. Equations of functions – influence of loading speed (x) on adhesive bond strength (y).

Adhesive	Functional equations	R^2
A	$y = 0.4923\text{Ln}(x) + 9.3216$	0.94
B	$y = 0.4896\text{Ln}(x) + 12.597$	0.91
C	$y = 0.6661\text{Ln}(x) + 9.3493$	0.86

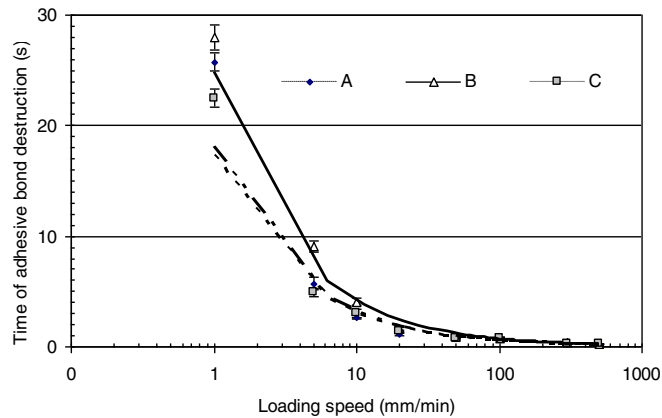


Fig. 5. Influence of loading speed on time of adhesive bond destruction – adhesive A, B, C.

The functions presented in Fig. 5 are determined by equations in table 2.

Tab. 2. Equations of functions – influence of loading speed (x) on time of adhesive bond destruction (y).

Adhesive	Functional equations	R^2
A	$y = 17.987x^{-0.7533}$	0.97
B	$y = 24.773x^{-0.7759}$	0.98
C	$y = 17.246x^{-0.7433}$	0.97

Tested adhesives were mutually compared using F-test from the influence of various loading speeds on the adhesive bond strength point of view. The zero hypothesis H_0 presents the state when there is no statistically significant difference ($p > 0.05$) among tested sets of data from their mean values point of view.

Adhesives A ($p = 0.0002$), B ($p = 0.0002$) and c ($p = 0.0009$) did not certify the hypothesis H_0 , so there is the difference among particular tested loading speeds in relation to the adhesive bond strength in the reliability level 0.05.

A graphic presentation of the results of the time of adhesive bonds destruction can be seen from Fig. 5. A course of the dependence was best described by the power function. The function type was derived from the correlation field, which was formed by the cross points of the dependent and independent variables. The adhesive bond destruction was faster than at one – component epoxies. [15]

A statistical comparison (T-test, $\alpha=0.05$) of the influence of increasing loading speed of the adhesive bonds on their shear strength is presented in tab. 3. It is visible from the statistical comparison of the values that increasing loading speed at all adhesive bonds considerably statistically influences the shear strength. At the adhesive A the statistically provable increase of the shear strength occurs with increasing loading speed from the speed $100 \text{ mm} \cdot \text{min}^{-1}$ (comparing with the speed $1 \text{ mm} \cdot \text{min}^{-1}$), at the adhesive B from the same speed (till $100 \text{ mm} \cdot \text{min}^{-1}$ $p>0.07$) and at the epoxy adhesive C any increase of the loading speed led to the statistically significant increase of the shear strength values.

Tab. 3. Statistical comparison of influence of loading speed of adhesive bond on its shear strength (T-test).

$H_0: \mu_1 = \mu_2$ ($p > 0.05$)	Used adhesives		
Comparison	A	B	C
1 : 5 $\text{mm} \cdot \text{min}^{-1}$	0.30	0.29	0.00
1:10 $\text{mm} \cdot \text{min}^{-1}$	0.27	0.16	0.00
1:20 $\text{mm} \cdot \text{min}^{-1}$	0.06	0.12	0.03
1:50 $\text{mm} \cdot \text{min}^{-1}$	0.07	0.12	0.00
1:100 $\text{mm} \cdot \text{min}^{-1}$	0.04	0.00	0.00
1:300 $\text{mm} \cdot \text{min}^{-1}$	0.01	0.01	0.00
1:500 $\text{mm} \cdot \text{min}^{-1}$	0.00	0.00	0.00

A type of the fracture surface differed depending on the type of the adhesive. The adhesives A and B showed a cohesive type of the fracture surface. The adhesive C showed an adhesive type of the fracture surface. Owing to the loading speed, the type of the fracture surface did not change. Fig. 6 shows the fracture surface of the adhesive C. The destruction of the adhesive bond occurred in the boundary of adhesive bonded material / adhesive. Fig. 7 presents the cohesive failure of the layer of the adhesive A. The destruction of the adhesive bond occurred in the layer of the adhesive.

Experiment results proved a definite increasing trend of the adhesive bond strength depending on the increasing loading speed. This trend was similar also at one-component adhesives. This trend was not definite at quick-setting cyanoacrylate adhesives. [15, 16]

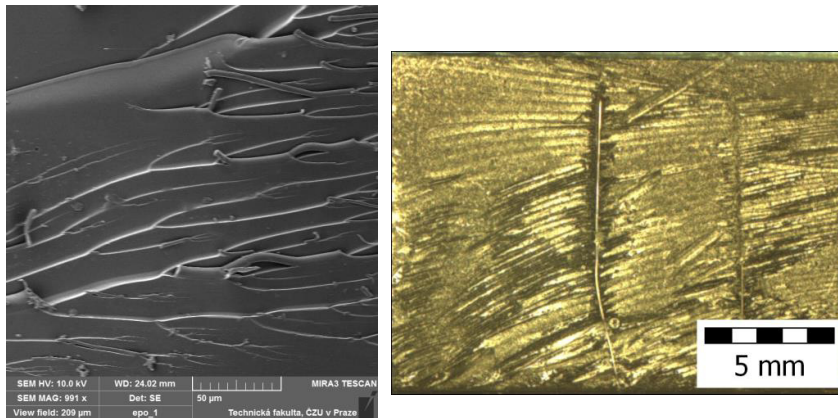


Fig. 6. Fracture surface of adhesive type (adhesive C): macroscopic view on fracture surface (right), SEM fracture surface (left).

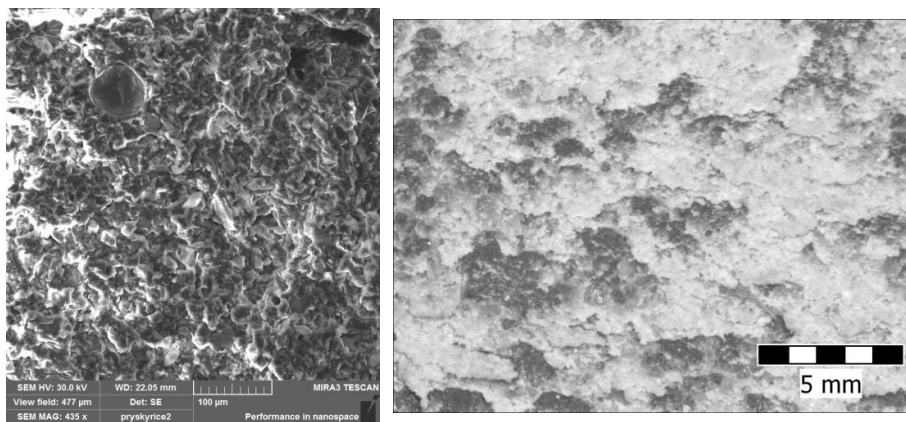


Fig. 7. Fracture surface of cohesive type (adhesive A): macroscopic view on fracture surface (right), SEM fracture surface (left).

4. Results

Following conclusions can be deduced from the experiment results:

- From the result of the constructional carbon steel S235J0 testing the increase of the breaking strength and yield strength at increasing loading speed is obvious. At increasing loading speed the fall of the elongation of the adhesive bonded material occurred. Considerably increase of the elongation of the adhesive bonded material occurred at the speeds over $100 \text{ mm} \cdot \text{min}^{-1}$.
- From the results it is obvious that the loading speed of the adhesive bonds has a positive influence on the strength results of tested adhesive bonds. It came to the increase of the adhesive bond strength up of 21-57% at the speed $500 \text{ mm} \cdot \text{min}^{-1}$. The most significant increase of the adhesive bond strength occurred at the adhesive C, up of 57% at the loading speed $500 \text{ mm} \cdot \text{min}^{-1}$.
- The speed of the elongation at the adhesive bond loading has a considerable influence on resultant time of the adhesive bond destruction. Decreasing time trend needed for the destruction of the adhesive bond is the result.
- The adhesive bond loading speed does not influence the change of the fracture surface

Acknowledgements

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