Influence of loading speed on a change of parameters of adhesive bonds based on cyanoacrylates

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Abstract


The paper deals with a behaviour of adhesive bonds created by means of quick-setting adhesives at different speeds of a loading. The aim of the research was to simulate a change of a bearing capacity of the adhesive bond at marginal limits foregoing the bond destruction. Application of quick-setting adhesives based on cyanoacrylates is considerably spread in a technical practice. Its advantage is speed of a hardening process. A fast creation of a bond and a possibility of fast manipulation with the adhesive bond are connected with it. The manipulation strength of the adhesive bond is reached in a few seconds. The aim of the research is to describe behaviour of quick-setting adhesives based on cyanoacrylates at different speeds of loading of the adhesive bonds in an interval of 1 to 600 mm/min. A change of the adhesive bond strength, an elongation and a failure time were evaluated within the experiments. Also the behaviour of the adhesive bonded material was observed within the research.

Keywords: adhesive bond strength; adhesive bonding technology; elongation; time of destruction

Application of quick-setting adhesives based on cyanoacrylates is considerably spread in a technical practice, e.g. constructions (Müller 2012; Müller, Mikuš 2014; Müller, Valášek 2014). A limit of these adhesives is so called “zero layer” of the adhesive (Müller 2014). This fact predetermines using the quick-setting adhesives for connecting of flat parts (Müller, Herák 2010). It is obvious from the research results that adhesive bonded areas are often deformed at production. The quick-setting adhesives based on cyanoacrylate adhesives (or superglue) are suitable for adhesive bonding of small parts and parts of medium size because they reach extremely quick fixation. These adhesives are suitable for automated systems with the necessity to reach quickly the handling strength; the disadvantage is an impossibility to fill gaps such as epoxy adhesives (Müller, Mikuš 2014).

The loading speed of the adhesive bond influences a speed of cracks spreading. According to Karac et al. (2011) the crack spreading influences the failure area of the adhesive bond. Also, destruction of the adhesive bond is connected with it, which is undesirable in the practice.

Higher loading speeds are used for elucidating the behaviour at simulation of the impact (Suresh et al. 2000). A disadvantage of the adhesive bonding technology is namely low resistance to impact dynamic loading (Müller et al. 2013). Lower values of loading enable to simulate a static view at the adhesive bond loading.

The aim of the research was to describe behaviour of quick-setting adhesives based on cyanoacrylates at different speeds of loading of the adhesive bonds in an interval of 1 to 600 mm/min. It is possible to pronounce a hypothesis that viscous adhe-
sives (liquid) and gel adhesives will be of different behaviour. A change of the adhesive bond strength, the elongation and the failure time were evaluated within the experiments. An essential factor is evaluation of changes (strength, elongation) of the adhesive bonded material.

**MATERIAL AND METHODS**

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 CSN EN 1465:2009 (Equivalent is BS 1465:2009).

To describe the process of adhesive bonds stress that affects the ultimate strength, the following cyanoacrylates were used:
- Loctite Super Bond Universal Liquid (Henkel Ltd., Dublin, Ireland) (marked 1),
- Super Ceys Instant Glue Universal (Grupo AC Marca, Barcelona, Spain) (marked 2),
- Loctite Power Flex Gel (Henkel Ltd., Dublin, Ireland) (marked 3),
- Alteco Super Glue (Alteco Chemical PTE Ltd., Tuas Avenue, Singapore) (marked 4),
- Samson Super Glue Gel (Z-trade s.r.o., Broumov, Czech Republic) (marked 5).

Specimens of all the tested materials were obtained identically – cutting from the semi-products in the hydraulic guillotine sheet metal machine. Laboratory tests of the adhesive bonds were performed using the standard test specimens made according to the standard CSN EN 1465:2009 (dimensions 100 ± 0.25 × 25 ± 0.25 × 1.6 ± 0.1 mm and lapped length of 12.5 ± 0.25 mm) from the constructional plain carbon steel S235J0 (Ferona, a.s., Prague, Czech Republic).

The surfaces of 1.5 mm thick steel sheets were at first blasted using the synthetic corundum of a fraction F80 under the angle of 90°. Using the profilograph Surftest 301 (Mitutoyo, Aurora, USA) the following values were determined: surface roughness in a cut plane \( Ra \) was \( 1.28 \pm 0.12 \, \mu \text{m} \), \( Rz = 6.2 \pm 0.86 \, \mu \text{m} \). \( Ra \) – the arithmetic mean of the
 departures of the profile from the mean line (µm), $R_z$ – the average of the maximum peak-to-valley length of five consecutive sampling lengths (µm).

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 adherent/adhesive/adherent (NOVÁK 2011; HRICOVÁ 2014). 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 real adhesive layer was measured by means of the stereoscopic microscope in cuts of the adhesive bonds. The adhesive bond cut was gradually photographed in the whole length of the cut in the microscope and it was evaluated by means of the software Quick Photo Industry (Promicra, s.r.o., Prague, Czech Republic) (Fig. 1). The adhesive layer thickness was 170.66 ± 5.81 µm.

Also the behaviour of the adhesive bonded material was observed within the research. Standard test specimens were prepared from the carbon steel S235J0 by means of an abrasive water jet (AWJ) (Fig. 2). The shape and sizes of test specimens determined for the tensile test were in accordance with the standard CSN EN ISO 6892-1: 2010. The surface roughness in a cut plane was: $R_a = 4.28 \pm 0.25$ µm, $R_z = 22.10 \pm 2.09$ µm and $R_t = 31.71 \pm 4.22$ µm. Where: $R_a$ – the arithmetic mean of the departures of the profile from the mean line (µm), $R_z$ – the average of the maximum peak-to-valley length of five consecutive sampling lengths (µm), $R_t$ – the maximum peak to valley height of the profile in the assessment length (µm).

The tensile strength test according to CSN EN ISO 6892-1: 2010 (Fig. 3a) and CSN EN 1465: 2009 (Fig. 3b) was performed using the universal tensile strength testing machine LABTest 5.50ST (Labortech s.r.o, Opava, Czech Republic) (sensing unit AST type KAF 50 kN, evaluating software Test&Motion). A speed of the loading corresponded to 1, 6, 24, 48, 100, 200, 400 and 600 mm/min. The failure type was determined at the adhesive bonds according to ISO 10365:1995.

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. So far as $R^2$ equals to 1, there is a perfect correlation in this sample (so there is no difference between calculation and real values).

### RESULT AND DISCUSSION

The increase of the tensile strength at increasing loading speed (Fig. 4) is obvious from the result of testing of the carbon steel S235J0. The fall of the elongation of the adhesive bonded material (Fig. 4) occurred at increase of the loading speed. The tensile strength of the adhesive bonded material increased by 10.12% in the observed interval of the loading speed and the elongation decreased by 13.23%.

Table 1 shows the results of the correlation analysis of results stated in Fig. 4.

A course of the dependence of the adhesive bond strength on the loading speed was best described by the power function (Fig. 5a). The function type was derived from the correlation field, which was formed by the cross points of the dependent and independent variables.

A significant trend is not visible from the experiment results. Adhesives 1 and 3 were of an increasing trend. Adhesive 1 showed the increase of 20.97% whereas adhesive 3 showed the increase of 28.69% in the tested interval. This is in accordance with the results of the adhesive bonded material (Fig. 4). Adhesives 2, 4 and 5 were of a decreasing trend; a significant fall was set at adhesive 5 (29.77%), ad-

### Table 1. Equations of functions – influence of loading speed (x) on tensile strength (y) and elongation (z)

<table>
<thead>
<tr>
<th>Adherent – steel S235J0</th>
<th>Functional equations</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile strength</td>
<td>$y = 297.89x^{0.0137}$</td>
<td>0.96</td>
</tr>
<tr>
<td>Elongation</td>
<td>$z = 69.441x^{-0.003}$</td>
<td>0.91</td>
</tr>
</tbody>
</table>

$R^2$ – index of determination

![Graph](image-url)
hesive 2 showed the fall of 19.58% and adhesive 4 showed the smallest fall of 7.58%.

Table 2 shows the results of the correlation analysis of the values stated in Fig. 5.

All tested adhesives were mutually compared using the F-test from the point of view of the influence of various loading speeds on the adhesive bond strength.

The zero hypothesis \( H_0 \) presents the state when there is no statistically significant difference \( (P > 0.05) \) among the tested sets of data as to their mean values. Adhesives 5 \( (P = 0.00007) \), 3 \( (P = 0.0000) \), 2 \( (P = 0.0087) \) and 1 \( (P = 0.0067) \) did not certify the hypothesis \( H_0 \), so there is the difference among particular tested loading speeds in relation to the adhesive bond strength on the reliability level 0.05.

Fig. 6. Cohesive failure area (a) adhesive 5 and (b) adhesive 3
The hypothesis $H_0$ was certified in the case of adhesive 4 ($P = 0.0923$), so there is no difference on the reliability level 0.05. The loading speed had no influence on the adhesive bond strength at adhesive 4; from this viewpoint, it thus comes to the statistically negligible difference in the reached values.

The adhesive bond elongation showed a decreasing trend at all adhesives (Fig. 5b). Adhesives 1, 2, 4 and 5 showed similar values. This conclusion is visible from the correlation field of the results. However, adhesive 3 (gel adhesive) showed higher values of the adhesive bond elongation.

Adhesives 1 to 5 ($P = 0.0000$, except for the adhesive 3 – $P = 0.0078$) did not certify the hypothesis $H_0$ so there is the difference among particular test loading speeds in relation to the adhesive bond elongation on the reliability level 0.05.

The significant fall of the destruction time in the interval of the loading speed 1 to 6 mm/min is obvious from the results of the experiment focused on the setting of the time of the adhesive bond destruction (Fig. 5c). This fact influences also the speed of the adhesive bond failure. The destruction occurs on average in 10 seconds at higher speeds (400 to 600 mm/min); which is significant from the viewpoint of destruction speed of the bonded construction.

The functions presented in Fig. 5c are determined by equations in Table 2. Different speeds of loading did not influence the change of the failure area type. The adhesives 1, 2 and 4 were of the adhesive failure area at the adhesive bond destruction. The adhesive 5 (Fig. 6a) was of the adhesive-cohesive failure area at the adhesive bond destruction. The adhesive 3 showed the cohesive type of the adhesive bond failure (Fig. 6b).

Different values of the loading speed are desirable for practical application. Namely high speeds of the loading are used for elucidating the behaviour at the simulation of the impact. Low and high values of the deformation speed are desirable for practical application (Müller, Mikuš 2014).

Similarly, namely high speeds of deformation are used for elucidating the behaviour at the simulation of the impact. The speed of the deformation influences the cracks spread as confirmed in the research of Karac et al. (2011). This state is undesirable in the area of the adhesive bonds. According to Karac et al. (2011) stable and unstable spreading of the crack did not influence the failure area of the adhesive bond.

**CONCLUSION**

Following conclusions of one-component quick-setting adhesives can be stated from the experiment results:

- The increase of the tensile strength at increasing loading speed is obvious from the results of the testing of the carbon steel S235J0 while the elongation of adhesive bonded material decreased.
- The loading speed influences the strength results of the tested adhesive bonds. It is not possible to set the explicit trend (decreasing, increasing, and stagnant) at one-component quick-setting adhesives.
- The loading speed has a negative influence on the elongation of the adhesive bond.
- The loading speed has a considerable influence on the resultant time of the adhesive bond destruction. The significant fall in the interval of 1 to

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Table 2. Equations of functions – influence of loading speed ($x$) on bond parameters ($y$)

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Functional equations</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bond strength</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>$y = 15.45x^{0.0302}$</td>
<td>0.86</td>
</tr>
<tr>
<td>2</td>
<td>$y = 16.572x^{-0.0219}$</td>
<td>0.52</td>
</tr>
<tr>
<td>3</td>
<td>$y = 20.735x^{0.0359}$</td>
<td>0.80</td>
</tr>
<tr>
<td>4</td>
<td>$y = 17.609x^{0.0176}$</td>
<td>0.52</td>
</tr>
<tr>
<td>5</td>
<td>$y = 18.932x^{-0.0464}$</td>
<td>0.82</td>
</tr>
<tr>
<td>Elongation of adhesive bond</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>$y = 2.4536x^{-0.1339}$</td>
<td>0.78</td>
</tr>
<tr>
<td>2</td>
<td>$y = 1.839x^{-0.082}$</td>
<td>0.68</td>
</tr>
<tr>
<td>3</td>
<td>$y = 4.6773x^{0.0621}$</td>
<td>0.76</td>
</tr>
<tr>
<td>4</td>
<td>$y = 2.9898x^{0.1499}$</td>
<td>0.91</td>
</tr>
<tr>
<td>5</td>
<td>$y = 3.3565x^{-0.2114}$</td>
<td>0.74</td>
</tr>
<tr>
<td>Time of adhesive bond destruction</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>$y = 57.138x^{-1.0122}$</td>
<td>0.99</td>
</tr>
<tr>
<td>2</td>
<td>$y = 47.82x^{0.9939}$</td>
<td>0.99</td>
</tr>
<tr>
<td>3</td>
<td>$y = 142.29x^{1.031}$</td>
<td>0.99</td>
</tr>
<tr>
<td>4</td>
<td>$y = 71.702x^{-1.0545}$</td>
<td>0.99</td>
</tr>
<tr>
<td>5</td>
<td>$y = 93.706x^{-1.1171}$</td>
<td>0.98</td>
</tr>
</tbody>
</table>

$R^2$ – index of determination
6 mm/min is obvious from the results of the experiment focused on setting of the destruction time of the adhesive bond. On average, destruction occurs in 10 s at higher speeds (400 to 600 mm/mins). This fact is important from the viewpoint of destruction speed of the bonded construction.

– The loading speed does not influence the change of the failure area.

References


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