Laminated safety glass and adhesives: A literature survey on experimental techniques and experimental data

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Abstract

This literature survey presents some of the available information in the literature regarding experimental methods and experimental data for mechanical testing of laminated safety glass and windscreen adhesives for the automotive industry.

When testing laminated safety glass several methods exist that involve dynamic loading. The evaluation of these tests does not normally involve acquisition of loads or displacements but do only include evaluation of the fracture appearance. Some methods, like split Hopkinson bar test, do include acquisition and analysis of loads and strains.

The literature survey indicates that there is a lack in windscreen adhesive data for higher strain rates. Articles describing testing at high strain rates exists regarding structural adhesives. Methods for high strain rate testing, both in compression and tension, have thus been used for testing of structural adhesives. Data from adhesive suppliers are mainly limited to low strain rates.

Key words: Windscreen adhesives, polyurethane adhesive, laminated safety glass, experimental methods, experimental data, crash, high strain rate
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Summary

This literature survey presents some of the available information in the literature regarding experimental methods and experimental data for mechanical testing of laminated safety glass and windscreen adhesives for the automotive industry.

When testing laminated safety glass several methods exist that involve dynamic loading. The evaluation of these tests does not normally involve acquisition of loads or displacements but do only include evaluation of the fracture appearance. Some methods, like split Hopkinson bar test, do include acquisition and analysis of loads and strains.

The literature survey indicates that there is a lack in windscreen adhesive data for higher strain rates. Articles describing testing at high strain rates exist regarding structural adhesives. Methods for high strain rate testing, both in compression and tension, have thus been used for testing of structural adhesives. Data from adhesive suppliers are mainly limited to low strain rates.
1 Introduction
This literature survey aims to present available information in the literature regarding experimental methods and experimental data for mechanical testing of laminated safety glass and windscreen adhesives for the automotive industry.

1.1 Delimitations
The literature survey will focus on laminated safety glass and windscreen adhesives in crash, i.e., experimental data and methods at high strain rates. The literature survey does not present how adhesives should be applied and used for best performance but focuses on mechanical properties and test methods of adhesives and laminated safety glass. Although the focus is on high strain rates corresponding to crash, static and quasi-static test methods will be presented together with methods covering higher strain rates. Typical strain rates in the base material at crash simulations are about $300 \text{s}^{-1}$ (Carlberger, Biel and Stigh 2009).

2 Glass
2.1 Glass in general

Glass is a homogeneous isotropic material having almost perfect linear-elastic behaviour over its tensile strength range.

Glass has a very high compressive strength and theoretically a very high tensile strength, but the surface of the glass has many irregularities which act as weaknesses when glass is subjected to tensile stress. These irregularities are caused by attack from moisture and by contact with hard materials (e.g. grit) and are continually modified by moisture which is always present in the air.

Tensile strengths of around 10 000 MPa can be predicted from the molecular structure, but bulk glass normally fails at stresses considerably below 100 MPa.

The presence of the irregularities and their modification by moisture contributes to the properties of glass which need consideration when performing tests of strength. Because of the very high compressive strength, glass always fails under tensile stress.

Further information on glass properties can be found in manufacturer’s manuals where e.g. Saint-Gobain Sekurit presents some of the data given in Table 1.

<table>
<thead>
<tr>
<th>Density (kg/m$^3$)</th>
<th>Knoop Hardness (HK)</th>
<th>Compression resistance (MPa)</th>
<th>Modulus of Elasticity (GPa)</th>
<th>Bending strength (MPa)</th>
<th>Poisson’s ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>2500</td>
<td>470</td>
<td>800 – 1000</td>
<td>70</td>
<td>45</td>
<td>0.22</td>
</tr>
<tr>
<td>Failure strain (%)</td>
<td>Failure Stress (MPa)</td>
<td>Softening temp. (°C)</td>
<td>Specific heat (J/gK)</td>
<td>Thermal cond. (W/mK)</td>
<td>Thermal exp. (K$^{-1}$)</td>
</tr>
<tr>
<td>0.12</td>
<td>84</td>
<td>Approx. 600</td>
<td>0.8</td>
<td>0.8</td>
<td>$9 \times 10^6$</td>
</tr>
</tbody>
</table>
The compression strength/resistance defines the ability of a material to resist a load applied vertically to its surface. The bending strength given in Table 1 is determined from ring-on-ring test (described in section 2.3.2).

### 2.2 Laminated safety glass

According to Pilkington and Saint-Gobain Sekurit the surface area of glazing per vehicle has increased vastly during the last 35-40 years. Pilkington says that the glazing area has increased by 50% in 35 years and Saint-Gobain Sekurit says that it has been doubled in the last 40 years. Partly these glazed areas consist of laminated safety glass.

Laminated safety glass is basically two panes of glass joined by an interlayer of polyvinyl butyral (PVB). During an accident, the glass will crack but it will stick together due to the PVB layer. Apart from keeping the windscreen intact the PVB layer also decreases the noise and improves the acoustic comfort inside the car. Saint-Gobain Sekurit has an acoustic PVB, dBCONTROL®, which consists of three layers; two outer layers of normal PVB and an inner layer made of a material with high damping properties. How this inner layer affects the mechanical properties and modelling of the laminated safety glass is not presented on Saint-Gobain Sekurit’s web page apart from stating that the PVB layers provide the mechanical properties of the laminated glass as requested by existing norms such as R43 (Saint-Gobain Sekurit - Glazing manual 2012).

A typical laminated safety glass has a thickness of 5 mm of which the two glass panes and the PVB interlayer have thicknesses of 2.1 mm and 0.76 mm, respectively.

Typical properties of PVB layer are given in Table 2.

<table>
<thead>
<tr>
<th>Property</th>
<th>PVB</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (kg/m³)</td>
<td>870 – 1100</td>
</tr>
<tr>
<td>Elastic limit (MPa)</td>
<td>11</td>
</tr>
<tr>
<td>Modulus of Elasticity (GPa)</td>
<td>0.100 – 0.220</td>
</tr>
<tr>
<td>Failure stress (MPa)</td>
<td>28</td>
</tr>
<tr>
<td>Failure strain (%)</td>
<td>200</td>
</tr>
<tr>
<td>Poisson’s ratio</td>
<td>0.48 – 0.495</td>
</tr>
</tbody>
</table>

### 2.3 Experimental methods

When performing mechanical testing of glass there exist methods that involve dynamic loading. The evaluation of these tests does not normally involve acquisition of loads or displacements but do only include evaluation of the fracture. Other methods involve acquiring of load and deformation. Some of these methods are described in the sections below.

#### 2.3.1 Mandatory test methods for laminated safety glass

The mandatory mechanical test methods for laminated safety glass for vehicles are described in a UN regulation as given in this section.

**Mechanical tests according to ECE R43**

The performance of laminated safety glass is regulated in United Nations ECE R43, Rev. 2 (Regulation ECE R43 n.d.) and presents mainly two different tests for mechanical strength; ball impact test and headform test.
The ball impact tests consist of two forms of tests with different weight of the steel ball dropped on a glass specimen. One of these tests uses a 227 g ball and one uses a 2260 g ball.

The purpose of the 227 g ball test is to assess the adhesion of the interlayer of laminated glass and the purpose of the 2260 g ball test is to assess ball-penetration resistance of laminated glass.

The purpose of the headform test is to verify the compliance of glazing with the requirements relating to the limitation of injury in the event of impact of the head against the windscreen.

For the ball impact tests a ball of the specified weight is dropped to a 300x300 mm² glass specimen which is fixed in between two frames. The 2260 g ball is dropped from 4 m and the 227 g ball is dropped from different heights depending on the thickness of the glass to be tested. For a typical 5 mm thick windscreen glass the drop height is 10 m.

The 2260 g ball tests are deemed to have given a satisfactory result if the ball does not pass through the glazing within five seconds after the moment of impact. For the 227 g ball test the weight of fragments are evaluated and should not exceed a specified value which for a 5 mm thick glass is maximum 15 g.

The test equipment at ball and headform tests is shown in Figs. 1-2.

Figure 1. Support for ball tests (Regulation ECE R43 n.d.).
2.3.2 Other test methods

Four point bending

In a paper (Timmell, et al. 2007), standard 4-point bending has been used to determine the deformation behaviour of laminated safety glass and to verify the model data used. Four point bending test is also described in EN 1288-3, be it for glass in buildings (CEN, EN 1288-3:2000 Glass in building - Determination of the bending strength of glass - Part 3: Test with specimen supported at two points (four point bending) 2000).

One drawback in using 4-point bending is that the micro cracks on the cut surface of the edges will initiate fracture and the actual materials characteristics of the glass is not actually tested.

To test the material without the influence of the micro cracks a ring-on-ring test is sometimes used.

Ring-on-ring test

By using a ring-on-ring test, also called coaxial double ring test, it is possible to determine the bending strength of the actual glass without the influence of the micro cracks of the cut edges. The test is performed using two rings in between which the glass is positioned. By loading the upper ring bending is introduced in the glass and ultimately the bending strength of the glass can be determined. The test procedure is described in EN 1288-5 (CEN, EN 1288-5:2000 E - Glass in building - Determination of the bending strength of glass - Part 5: Coaxial double ring test on flat specimens with small test surface areas 2000).

The test arrangement is illustrated in Fig. 3.

The ring-on-ring test should, according to EN 1288-1, not be used for laminated safety glass and should be used as a method of evaluating the comparative bending strength of flat glass.
Adding high-speed photography to standard test methods

To assess the behaviour of the glass fracture there are examples in the literature where high-speed photography is used in combination with the methods described above (Xu, Sun, et al. 2011, Mattiasson 2012). By using high-speed photography the crack growth can be studied and, in combination with acquisition of load and displacement, a better understanding of the fracture in glass is achieved. The monitoring of crack growth also gives the possibility to verify calculations regarding crack pattern and growth.

Using high-speed photography is of interest for e.g. the methods described in ECE R43 (Regulation ECE R43 n.d.) for which only the appearance of the glass after the impact is analysed.

High strain-rate testing

In the literature split Hopkinson pressure bar tests (SHPB) are performed on laminated safety glass (Xu, Li, et al. 2011). By using SHPB strain rates of up to 6000 s^{-1} could be achieved. A schematic image of the SHPB test is shown in Figure 4.
2.4 Experimental data

2.4.1 PVB experimental data

In Fig. 5 data for the PVB layer presented by Larcher et al (2012) is shown. The curves are determined from standard tensile tests (the data are most likely engineering values). All test data shown indicates that the behaviour of PVB under small strain rates is viscoelastic. This behaviour changes when loading the PVB at higher strain rates (about 10 s⁻¹). The material becomes more and more elastic with hardening, and the Young’s modulus increases dramatically. The hardening parameter corresponds to the Young’s modulus for small strain rates. The strain limit appears to be similar to the static one (Larcher, et al. 2012).

![Graph showing stress vs. strain for different strain rates](image)


As depicted in Fig. 6 the shear modulus of PVB is temperature dependent and increases as the temperature decreases. At temperatures exceeding 30°C the shear modulus is close to zero.

![Graph showing shear modulus vs. temperature](image)

Figure 6. Shear modulus of PVB for different temperatures (Timmell, et al. 2007).

As stated by e.g. Timmell et al (2007) a polymer behaves qualitatively the same if we increase the strain rate or if we decrease the temperature. The response of the PVB-interlayer varies from rubbery elastic at low strain rates to glass like linear elastic for high strain rates (Timmell, et al. 2007).
2.4.2 Laminated safety glass data

Fig. 7 shows test data from a four-point bending test in which the displacement was increased slowly (Timmell, et al. 2007). The tested specimen had a length, $l=1100$ mm, a width, $w=600$ mm and a thickness, $t=6.72$ mm of which the PVB layer was 0.72 mm. The results are most likely affected by the micro cracks of the cut edges.

![Figure 7. Testing of laminated safety glass in four-point bending (Timmell, et al. 2007).](image)

When modelling the four-point bending test (as also shown in Fig. 7) the authors used a Young’s modulus of $E=70$ GPa, a Poisson ratio of 0.23 and a failure strain of 0.15 %.

Further mechanical data for laminated safety glass is shown in Figs. 8-9. Both figures indicated the strain-rate dependency of laminated safety glass in compression where Fig. 8 shows low strain rate data and Fig. 9 shows high strain rate data. The paper does not state how many tests that were performed at each strain rate. The stress-strain curves in Fig. 9 are determined by using split Hopkinson pressure bar test (SHPB).

![Figure 8. Stress-strain curves obtained from quasi-static compression experiments under three deformation rates (Xu, Li, et al. 2011).](image)
The stress–strain curves in Fig. 8 show a nonlinear characteristic in mechanical behaviour of the laminated safety glass and this nonlinear phenomenon is probably caused by both nonlinear mechanical response of the PVB interlayer and progressive micro-crack growth in the tested sample. As the loading rate increases, the major failure onset (MFO) strain increases while the MFO stress remains nearly the same. The major responsible reasons are: (i) in extremely low strain rate (quasi-static) situation, the outer glass panel plays a critical role in mechanical response and (ii) glass is a rate-independent material whereas PVB is a rate dependent one (Xu, Li, et al. 2011).

![Figure 9. Stress-strain curves obtained from dynamic SHPB experiments at high strain rates (Xu, Li, et al. 2011).](image)

From the test data shown in Figs. 8-9 the authors state that, when changing the strain rate at loading of laminated safety glass the failure strain will be discontinuous as shown in Fig. 10 (Xu, Li, et al. 2011). The MFO strain was defined as the strain at which the sample starts to have major cracks and a drop in load is observed.

The stress and strain in Fig. 8 is engineering values. No information regarding the number of tests for each strain and deformation rate is given in the paper. In Fig. 9 the stress-strain data are calculated from wave propagation theories in solids. Thus, the data in Fig. 9 is dynamic stress and dynamic strain (Xu, Li, et al. 2011).

![Figure 10. Mechanism of different failure strains at various strain rates of laminated safety glass (Xu, Li, et al. 2011).](image)
Fig. 10 shows the strain variation during different strain rates. In both quasi-static and dynamic scenarios, strain rate effect will cause the ultimate strain to increase before unloading due to major cracks (MFO strain). On the contrary, in the domain where quasi-static load changes into dynamic load, material behaviour becomes more brittle, shown by the decrease in MFO strain induced by the quick load of the impact. This phenomenon is also observed and concluded by Nemat and Deng [referred to by Xu, Li et al], and Huang et al. [referred to by Xu, Li et al]. Therefore, the ultimate strain’s discontinuity at continuous strain rates is inevitable (Xu, Li, et al. 2011).

Based on high-speed photography images of crack growth, as shown in Fig. 11, the crack tip velocity and acceleration can be calculated as depicted in Fig. 12. The steady-state cracking speed in stage II is about 811 m/s. The stable crack growth occurs due to retardment effects from microcracks impeding the crack propagation (Xu, Sun, et al. 2011).

![Figure 11. Snapshots of crack propagation with 1 kg of drop weight and 1 m of drop height (Xu, Sun, et al. 2011).](image)

![Figure 12. Averaged crack tip velocity and acceleration (Xu, Sun, et al. 2011).](image)
3 Adhesives

3.1 Adhesives for automotive glass

There are several adhesive manufacturers worldwide who produce automotive direct-glazing adhesive systems. The manufacturers often differentiate between adhesives for OEM (original equipment manufacturer) and after-market adhesive system although both are polyurethane based adhesives. Some manufacturers and their adhesive systems for direct-glazing are given in Table 3 below.

Table 3. Some important adhesive manufacturer and examples of their systems for direct-glazing.

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>System name</th>
<th>Adhesive type</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bostik</td>
<td>ISR (Industry Special Range)</td>
<td>Silyl Modified Polymer (SMP)</td>
</tr>
<tr>
<td>DOW</td>
<td>BETASEAL™</td>
<td>1 component polyurethane</td>
</tr>
<tr>
<td>Henkel</td>
<td>TEROSTAT</td>
<td>1 component polyurethane</td>
</tr>
<tr>
<td>Sika</td>
<td>SikaTack</td>
<td>1 component polyurethane</td>
</tr>
<tr>
<td></td>
<td>Sikaflex</td>
<td></td>
</tr>
</tbody>
</table>

As seen in Table 3 the adhesives for direct-glazing are mostly polyurethanes but other adhesive types exist. Polyurethanes are used for most materials but is especially good for plastics and metals.

These adhesives are often abbreviated to PU or PUR, and are chemically reactive formulations that may be one-component or two-component systems and can be fast curing. A fast cure usually necessitates applying the adhesives by machine. They are often used with primers.

The single-component formulations that are available, are partially polymerised and stable until cure is initiated by the action of absorbed atmospheric moisture. Their reaction rate is slower because it takes time to absorb the necessary water. Polyurethanes can be supplied as reactive chemicals, solvent solutions, pastes or hot melts. The single-component polyurethanes provide strong, resilient joints which are impact resistant and have good low-temperature strength compared with many other adhesives. Polyurethanes find major uses in the bonding of glass fibre reinforced plastics (GRP), direct-glazing of automobiles and lamination of both insulation panels and flexible packaging materials (Adhesive Toolkit 2012).

Presented in Table 4 below are adhesive data as given by the manufacturers.
Table 4. Mechanical data for some adhesive manufacturers adhesive systems.

<table>
<thead>
<tr>
<th></th>
<th>Bostik ISR 70-08 AP</th>
<th>DOW BETASEAL 1943</th>
<th>Henkel TEROSTAT</th>
<th>Sika SikaFlex-252</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (kg/m³)</td>
<td>1.5</td>
<td>1.25 – 1.30</td>
<td>1.27</td>
<td>1.20</td>
</tr>
<tr>
<td>Tensile strength (MPa)</td>
<td>2.9 (DIN 53504/ISO 37)</td>
<td>&gt; 5 (DIN 53504)</td>
<td>8 (DIN 53504)</td>
<td>4 (ISO 37)</td>
</tr>
<tr>
<td>Stress at 100% (MPa)</td>
<td>2.3 (DIN 53504/ISO 37)</td>
<td>Not given</td>
<td>2 (DIN 53504)</td>
<td>Not given</td>
</tr>
<tr>
<td>Elongation at break (%)</td>
<td>250 (DIN 53504/ISO 37)</td>
<td>ca. 300</td>
<td>400 (DIN 53504)</td>
<td>&gt;300</td>
</tr>
<tr>
<td>Shear strength (MPa)</td>
<td>2.4 (Alu-Alu, 2 mm, 50 mm/min) (DIN 53283/ASTM D1002)</td>
<td>Min. 5 (after 7 d 23°C/50% rh, 2 mm adhesive height) (EN 1465)</td>
<td>1.5 (@ 24 h, DIN EN 1465)</td>
<td>2.5 (ISO 4587)</td>
</tr>
<tr>
<td>Tear strength (N/mm)</td>
<td>16 (Type C, 500 mm/min) (DIN 53515/ISO 34)</td>
<td>Not given</td>
<td>Not given</td>
<td>9 (ISO 34)</td>
</tr>
<tr>
<td>Modulus of elasticity (10%) (MPa)</td>
<td>5.5 (DIN 53504/ISO 37)</td>
<td>Not given</td>
<td>Not given</td>
<td>Not given</td>
</tr>
</tbody>
</table>

Based on the shear strength of an adhesive the adhesives can be divided into; structural, semi-structural and sealant. Structural adhesives have a shear strength higher than 15 MPa, semi-structural 8 – 15 MPa and sealant less than 8 MPa (Albinsson 2012). Based on this, the windscreen adhesives should be considered as sealants.

### 3.2 Experimental methods

As an overview of test methods the division of methods found on Adhesive Toolkit webpage (Adhesive Toolkit 2012) is given in Appendices 1-2. The test methods are given in Tables A1 – A4 presented in Appendix 1. Each of the test methods is specified in different standards. The name of the ISO and ASTM standards is given in the tables as well as listed with their full title in Appendix 2. The tables in Appendix 1 also specify relative costs associated with the different methods and other useful information regarding the methods. All tables are adopted from Adhesive Toolkit (2012).

Due to the focus on windscreen adhesives and crash in this literature survey all of the methods given in the appendices should be seen as a general overview of adhesive test methods.

In the main part of the report the methods presented are thus methods used for testing of windscreen adhesives or methods used for high strain rate testing. For the higher strain rate testing other adhesives might be reviewed due to a lack of high strain rate testing of non-structural adhesives.
3.2.1 Test methods used by the adhesive manufacturers

As given in Table 3 the adhesives manufacturers test the mechanical properties according to some given ISO, DIN or ASTM standards. These methods are focused on in this section.

Tensile strength
To determine the tensile properties ISO 37 is applied (SIS 2012). From this test tensile strength, elongation at break and stress at 100% elongation (or stress at some other strain level) is determined. Modulus of elasticity is given in Table 3 as being determined by Bostik using ISO 37. ISO 37 does not specify how to derive the modulus of elasticity.

When testing an adhesive according to ISO 37 often a dumb-bell test piece is used as shown in Fig. 13. The test pieces are formed using a die.

![Figure 13. Shape of dumb-bell test pieces (SIS 2012).](Image)

Different types of test pieces are described in the standard. Typically the narrow portion of the test piece shall be 2.0 mm with a test length (indicated by 1 in the figure) of 25 mm.

The tensile test machine used at testing should be capable of operating at rates of traverse of 100 mm/min, 200 mm/min and 500 mm/min. Having the specified 25 mm specimen test length these different rates of traverse give rise to strain rates of the order of $10^{-1}$ s$^{-1}$, i.e., far from the desired strain rate at crash.

Tear strength
To determine the tear strength the standard ISO 34 describes the test method (ISO 2010). Three different alternative specimen geometries are defined for this kind of test; trouser test piece, angle test piece and crescent test piece. The different test specimens are referred to as Method A, B and C, respectively. The different geometries are shown in Fig. 14.

![Figure 14. Specimen geometries according ISO 34. 1 indicates location of cut or nick.](Image)

a) Trouser test piece die.  b) Angle test piece die.  c) Crescent test piece die.

The test consists in measuring the force required to tear a specified test piece, in continuation of the cut or nick already produced in the test piece or, in the case of method B, procedure (a), completely across the width of the test piece.

The tearing force is applied by means of a tensile testing machine, operated without interruption at a constant rate of traverse until the test piece breaks. Dependent upon the
method employed, the maximum (used for angled and crescent test piece) or median force (used when determining the trouser tear strength) achieved is used to calculate the tear strength.

No correlation between data obtained by the alternative test pieces is implied.

When testing the trouser type specimen the positioning in the test machine is done according to Fig. 15.

![Figure 15. Positioning of trouser test piece in testing machine.](image)

For the trouser test a deformation rate of 100 mm/min is used. For angle and crescent test piece a deformation rate of 500 mm/min is used. Thus, these rates are low in comparison to crash situations.

**Tensile lap shear strength**


The adhesive lap-shear strength is determined by stressing a single-overlap joint between rigid adherends in shear by the application of a tensile force parallel to the bond area and to the major axis of the specimen. The specimen is shown in Fig. 16 and should have a length of overlap of 12.5 mm ± 0.25 mm. A typical adhesive thickness is 0.2 mm.
Testing at constant speed shall result in fracture within a period of 65 s ± 20 s. If a machine working at constant rate of loading is used, apply the shear load at a rate of 8.3 MPa to 9.8 MPa per minute.

The results from testing should be expressed as the mean of the breaking force, in newtons, or the breaking stress, in megapascals, of the valid specimen. The lap shear strength, in megapascals, is calculated by dividing the breaking force, in newtons, by the shear area, in square millimetres.

A variant of the tensile lap shear test is the **thick adherend shear test (TAST)**. The TAST can be seen as an optimized single lap shear test, since thick substrates and a small overlap are used in order to limit the influence of stress singularities (Cognard, Créac’hcadec and Sohier 2011). This kind of test method is also suitable to use for elastomeric adhesives (Carlberger, Private communication 2012). The TAST is described in ISO 11003-2 (ISO, ISO 11003-2:2002 - Adhesives - Determination of shear behaviour of structural adhesives - Part 2: Tensile test method using thick adherends 2002).

### 3.2.2 Other test methods

In the literature a large amount of methods are described when testing adhesives. The methods mentioned in section 3.2.1 are the ones used by the manufacturers. In the present section methods used in research, involving other adhesives than windscreen adhesives, and higher strain rates are presented. Some test methods are also mentioned in the section presenting experimental data.
**Peel Strength and Fracture Energy**

To achieve pure peel deformation the double cantilever beam (DCB) can be used (Carlberger, Biel and Stigh 2009). From this kind of test the peel strength and fracture energy can thus be determined. The method is described in ASTM D 3433 (ASTM 1999).

The principle of loading and the test specimen are shown in Fig. 17 a). In Fig. 17 b) an image showing how the loading might be achieved is depicted (Carlberger, Biel and Stigh 2009).

![Figure 17. Principle of double cantilever beam testing (Carlberger, Biel and Stigh 2009).](image)

The crosshead speed in the experiments performed by Carlberger et al was 100 mm/s. This crosshead speed resulted in a strain rate around $1.5 \times 10^{-3}$ s$^{-1}$ when testing at room temperature. The strain rate varies during the test due to softening of the adhesive. The given strain rate was determined when half of the fracture energy was consumed.

From the DCB test the strain energy release rate ($J$, given in J/m$^2$) can be determined.

**Shear strength**

Pure shear can be achieved by using end notched flexure (ENF) specimens (Carlberger, Biel and Stigh 2009). As shown in Fig. 18 the specimen is subjected to three-point bending and due to the occurrence of the unbounded part of the specimen, which can be considered as a crack ($a_0$ in the figure), shear will take place at the end of the crack.

![Figure 18. End notched flexure specimen (Carlberger, Biel and Stigh 2009).](image)

From the ENF test the strain energy release rate ($J$, given in J/m$^2$) can be determined.
High speed testing
There are examples in the literature on high strain rate testing of adhesives (Morin, et al. 2011). These tests are performed on a structural adhesive BETAMATE 1496V which is a one component epoxy. To achieve the high strain rates (100 – 5000 s⁻¹), split Hopkinson bar test was used with different setups for tension and compression as shown in Fig. 19. For low and intermediate strain rates (0.1 – 53 s⁻¹) Morin et al. used a high speed hydraulic machine.

![Split Hopkinson test setup](image)

Figure 19. Split Hopkinson test in tension (upper) and compression (lower) (Morin, et al. 2011).

As seen in Fig. 19 the tests were filmed with high speed cameras. The acquired images were used for 3D digital image correlation analysis to achieve a full strain field of the specimen tested.

3.3 Experimental data
3.3.1 Sikaflex-256 FC experimental data
In a study (Loureiro, et al. 2010) Sikaflex-256 FC was tested (a one-component polyurethane) and compared with an epoxy adhesive. According to the datasheet for the polyurethane adhesive it is chemically identical to Sikaflex-252 but has a somewhat higher strength and elongation at break. The tensile strength is approximately 7 MPa, elongation at break 400 % and tensile lap shear strength 5 MPa. The Sikaflex-256 grade is an aftermarket grade for automotive glass replacement business.

According to Lennart Nystedt at Sika, Sweden (Nystedt 2012) the thickness of the adhesive joint (0.2 mm) is suitable for the epoxy adhesive but not the Sikaflex adhesive. A thicker adhesive thickness is suitable for the elastomeric type of adhesive. Nystedt states that a thicker elastomeric adhesive joint would improve the measured Sikaflex properties.

Several interesting data are presented regarding the mechanical properties of this polyurethane adhesive. In the study the authors used T-peel joints and single lap joints and loaded them statically, in fatigue and with an impact. The specimens are shown in Fig. 20.
In a single lap shear joint (SLJ) the major stress component is shear and in the T-peel joint the loading is directly through the adhesive although a bending moment and rotation is introduced (Loureiro, et al. 2010).

From the static (quasi-static) tests it is seen that the SLJ is non-linear from the start (and has a much lower stiffness compared to the epoxy SLJ). All joints failed cohesively. A summary of the failure loads in the static tests are shown in Fig. 21.

As seen in Fig. 21 a slight increase in failure load is observed as the deformation rate increases. A comparison of the load-displacement curves for the SLJ test at different deformation rates are shown in Fig. 22.
Fatigue tests were also performed for the two joint geometries and adhesives. The fatigue data are shown in Figs. 23-24.

The fatigue load in Figs. 23-24 are normalized to the average static failure load (failure loads determined for 1 mm/min deformation rate). According to the authors the fatigue results are somewhat surprising since elastomeric materials are known for their improved fatigue resistance. A possible cause for the results the adhesive heating during fatigue testing which might have stronger influence on the elastomeric adhesive in comparison to the epoxy (Loureiro, et al. 2010). As was the case for the static tests, all specimens failed cohesively in the fatigue tests. The fatigue tests were performed with a load ratio $R=0.1$ at a frequency of 10 Hz.
Figure 24. Fatigue life curves of T-joints (Loureiro, et al. 2010).

The two geometries were also impact tested with the test set up shown in Fig. 25.

Figure 25. Inertial wheel impact testing equipment (Loureiro, et al. 2010).

At impact testing an impactor on the inertial wheel hits an anvil at the end of the specimen (T-joint or SLJ type). The impact velocity was 3 m/s which is equivalent to a deformation rate of $1.8 \cdot 10^2$ mm/min.

In Fig. 26 the static failure load at 1 mm/min are compared with the impact loads.
Interestingly it can be observed that the differences between the two adhesives become less pronounced under impact loading conditions compared to static loading. The study also concluded that the joint strengths were much higher under impact loading than under static loading, especially for the polyurethane adhesive (Loureiro, et al. 2010).

### 3.3.2 High strain rate test of structural adhesive

In a study (Morin, et al. 2011) a structural adhesive, BETAMATE 1496V, was tested at strain rates ranging from 0.1 to 5000 s\(^{-1}\) using a high speed hydraulic machine and a split Hopkinson bar test equipment. The adhesive was tested in both tension and compression and when manufacturing the test specimens these were cured at different pressures (1 and 4 MPa, respectively) to study the influence of both strain rate and curing pressure.

The tensile tests performed at low and intermediate strain rates showed a non-negligible visco-elastic phenomenon as depicted in Fig. 27. The strain rates were 5.3 \(\times 10^{-3}\) and 53 s\(^{-1}\), respectively.

Figure 26. Failure loads of specimens under impact loads, along with static failure loads at 1 mm/min (Loureiro, et al. 2010).

Figure 27. Evolution of the tensile elastic modulus as a function of the strain rate (Morin, et al. 2011).
Regarding plasticity for the tensile tests a visco-plastic behaviour was observed as shown in Fig. 28.

![Figure 28](image)

Figure 28. True tensile stress-true plastic strain curves at different strain rates (Morin, et al. 2011).

Similarly, Figs. 29-30 show the elastic modulus and the stress-strain curves at different strain rates and thus the visco-elastic and visco-plastic behaviour observed in compression.

![Figure 29](image)

Figure 29. Evolution of the compressive elastic modulus as a function of the strain rate and curing pressure (Morin, et al. 2011).
Figure 30. True compressive stress-true plastic strain curves at different strain rates. The curing pressure was 4 MPa (Morin, et al. 2011).

Figure 31. True compressive stress-true plastic strain curves at different curing pressures. The strain rate at testing was 53 s⁻¹ (Morin, et al. 2011).

Fig. 31 shows the influence of curing pressure on the stress levels observed at compressive testing at a strain rate of 53 s⁻¹.
4 References


Albinsson, Ola, interview by Torsten Sjögren. Discussion on adhesives (March 16, 2012).


—. "EN 1288-3:2000 Glass in building - Determination of the bending strength of glass - Part 3: Test with specimen supported at two points (four point bending)." European Committee for Standardization, 2000.


5 Appendices

5.1 Appendix 1. Standard methods for mechanical testing of adhesives

Table A1. Tensile and peel test methods for adhesives.

<table>
<thead>
<tr>
<th>Test Method</th>
<th>Tensile But Joint</th>
<th>T-Peel</th>
<th>Climbing Drum</th>
<th>Floating Roller Method</th>
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<tbody>
<tr>
<td>Principle image of specimen or test set-up</td>
<td><img src="image1.png" alt="Image" /></td>
<td><img src="image2.png" alt="Image" /></td>
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<td></td>
</tr>
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<td>Mechanical Properties Obtained</td>
<td>Tensile strength/modulus</td>
<td>Peel strength</td>
<td>Peel strength/skin stiffness</td>
<td>Peel strength</td>
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<td>Material Quantity Requirements per specimen</td>
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<td>Low</td>
<td>High</td>
<td>Low</td>
</tr>
<tr>
<td>Typical Specimen Dimensions (mm)</td>
<td>Diameter 15–25</td>
<td>Bond length 150</td>
<td>Long adherend 300</td>
<td>Flexible adherend 250</td>
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<tr>
<td></td>
<td>Adherend thickness 12–15</td>
<td>Width 25</td>
<td>Short adherend 240</td>
<td>Rigid adherend 200</td>
</tr>
<tr>
<td></td>
<td>Adherend thickness 0.5–1.0</td>
<td>Width 25</td>
<td>Adherend thickness 0.5–5.0</td>
<td>Width 25</td>
</tr>
<tr>
<td></td>
<td>Arm length 50</td>
<td>Adherend thickness 0.5–1.6</td>
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</tr>
<tr>
<td>Materials Suitable for Testing</td>
<td>1—6</td>
<td>1, 4 and 6</td>
<td>Flexible-rigid adherend</td>
<td>Flexible-rigid adherend</td>
</tr>
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<td>Flexible-flexible adherend</td>
<td>1—6 + sandwich structures</td>
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<td>1—6</td>
</tr>
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<td>Low-moderate</td>
<td>High</td>
<td>Low—moderate</td>
</tr>
<tr>
<td>Cost of Testing/Specimen</td>
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<td>Low</td>
<td>Low—moderate</td>
<td>Low—moderate</td>
</tr>
<tr>
<td>Specimen Fabrication Equipment Requirements</td>
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<td>Surface preparation Bonding+Bonding jig</td>
<td>Surface preparation Bonding+Bonding jig</td>
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<td>None</td>
<td>Extensometer (2 off)</td>
</tr>
<tr>
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<td>Universal test machine + end grips</td>
<td>Universal test machine + end grips</td>
<td>Special test fixture Universal test machine + end grips</td>
<td>Special test fixture Universal test machine + end grips</td>
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<td>Unsuitable</td>
<td>Unsuitable</td>
</tr>
<tr>
<td>---------------------</td>
<td>---------</td>
<td>------------</td>
<td>------------</td>
<td>------------</td>
</tr>
<tr>
<td>Creep Performance</td>
<td>Suitable</td>
<td>Possibly</td>
<td>Unsuitable</td>
<td>Unsuitable</td>
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<td>Unsuitable</td>
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<td>Straightforward</td>
<td>Straightforward</td>
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<td>To be determined</td>
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<td>ASTM D 2095</td>
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<td>ASTM 1876</td>
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</table>

1 = metal-metal; 2 = metal-plastic; 3 = metal-composite; 4 = plastic-plastic; 5 = plastic-composite; 6 = composite-composite.

Table A2. Cleavage and Mode I fracture toughness test methods for adhesives.

<table>
<thead>
<tr>
<th>Test Method</th>
<th>Wedge Cleavage</th>
<th>Compact Tension</th>
<th>DCB (Double Cantilever Beam)</th>
<th>TDCB (Tapered Double Cantilever Beam)</th>
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<td><img src="image3.png" alt="Image" /></td>
<td><img src="image4.png" alt="Image" /></td>
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<td>Mechanical Properties Obtained</td>
<td>Fracture energy</td>
<td>Cleavage strength</td>
<td>Mode I fracture toughness</td>
<td>Mode I fracture toughness</td>
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<td>Material Quantity Requirements/Specimen</td>
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<td>high</td>
<td>Low</td>
<td>High</td>
</tr>
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<td>Materials Suitable for Testing</td>
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<td>1—6</td>
<td>1—6</td>
<td>1 and 6</td>
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<td>Low</td>
<td>Moderate—High</td>
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<td>Low-moderate</td>
<td>Low-moderate</td>
</tr>
<tr>
<td>--------------------------</td>
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<td>--------------</td>
<td>--------------</td>
</tr>
<tr>
<td>Specimen Fabrication Equipment Requirements</td>
<td>Surface preparation</td>
<td>Surface preparation Bonding + bonding jig</td>
<td>Surface preparation</td>
<td>Surface preparation Bonding + Bonding jig</td>
</tr>
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<td>Specimen Instrumentation Requirements</td>
<td>Travelling microscope or video camera</td>
<td>Extensometer for crack opening displacement</td>
<td>Travelling microscope or video camera</td>
<td>Travelling microscope or video camera</td>
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<td>Test Equipment and Fixture Requirements</td>
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<td>Universal test machine + loading fixture</td>
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<td>Suitable</td>
<td>Suitable</td>
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1 = metal-metal; 2 = metal-plastic; 3 = metal-composite; 4 = plastic-plastic; 5 = plastic-composite; 6 = composite-composite.

Table A3. Shear test methods for adhesives.

<table>
<thead>
<tr>
<th>Test Method</th>
<th>Single-Lap</th>
<th>Double-Lap</th>
<th>V-Notched Beam</th>
<th>Arcan</th>
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<td>Shear strength</td>
<td>Shear strength/modulus</td>
<td>Shear strength/modulus</td>
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<td>Length 76</td>
<td>Length 52</td>
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<td>Width 25</td>
<td>Width 25</td>
<td>Width 20</td>
<td>Width 40</td>
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<td>Adherend thickness 2</td>
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<td>Adherend thickness 6</td>
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<td>1, 3 and 6</td>
<td>1, 3 and 6</td>
<td>1, 3 and 6</td>
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<td>Moderate</td>
<td>Moderate</td>
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<td>Surface preparation</td>
<td>Surface preparation</td>
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<td>Bonding + bonding jig</td>
<td>Bonding + Bonding jig</td>
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<td>Strain gauges</td>
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<td>Suitable</td>
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<td>Creep Performance</td>
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<td>Unsuitable</td>
<td>Unsuitable</td>
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<td>Low—moderate (10–20%)</td>
<td>Low—moderate (10–20%)</td>
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1 = metal-metal; 2 = metal-plastic; 3 = metal-composite; 4 = plastic-plastic; 5 = plastic-composite; 6 = composite-composite.
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<tr>
<th>Test Method</th>
<th>Thick Adherent</th>
<th>Torsion Butt Joint</th>
<th>Napkin Ring</th>
<th>ENF (End-Notched Flexure)</th>
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<td><img src="image2.png" alt="Image" /></td>
<td><img src="image3.png" alt="Image" /></td>
<td><img src="image4.png" alt="Image" /></td>
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<tr>
<td>Mechanical Properties Obtained</td>
<td>Shear strength/modulus</td>
<td>Shear strength/modulus</td>
<td>Shear strength</td>
<td>Mode II fracture energies</td>
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<td>1–6</td>
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<td>1 and 6</td>
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<td>Surface preparation Bonding + bonding jig</td>
<td>Surface preparation Bonding + bonding jig</td>
<td>Surface preparation Bonding + Bonding jig</td>
</tr>
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<td>Rotary extensometer</td>
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<tr>
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1 = metal-metal; 2 = metal-plastic; 3 = metal-composite; 4 = plastic-plastic; 5 = plastic-composite; 6 = composite-composite.
5.2 Appendix 2. Standards for mechanical testing of adhesives

5.2.1 ISO STANDARDS - Number/Title

Cleavage Tests


Peel Tests


ISO 11339 (2003): Adhesives - T-Peel Test for Flexible-to-Flexible Bonded Assemblies


Shear Tests
ISO 4587 (2003): Adhesives—Determination of Tensile Lap-Shear Strength of Rigid-to-Rigid Bonded Assemblies


ISO 10964 (1993): Adhesives—Determination of Torque Strength of Anaerobic Adhesives on Threaded Fasteners


ISO 13445 (2003): Adhesives—Determination of Shear Strength of Adhesive Bonds between Rigid Substrates by the Block-Shear Method

Tensile Tests
ISO 6922 (1987): Adhesives—Determination of Tensile Strength of Butt Joints


**Mechanical Properties - Other Tests**


### 5.2.2 ASTM STANDARDS - Number/Title

**Cleavage Tests**


**Peel Tests**


ASTM D 1876–01: Standard Test Method for Peel Resistance of Adhesives (T-Peel Test)


**Shear Tests**


ASTM D 1002–01: Standard Test Method for Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading (Metal-to-Metal)


ASTM D 4896–01: Standard Guide for Use of Adhesive-Bonded Single Lap-Joint Specimen Test Results

ASTM D 5648–01: Standard Test Method for Torque-Tension Relationship of Adhesives Used on Threaded Fasteners (Lubricity)

ASTM D 5656–04: Standard Test Method for Thick-Adherend Metal Lap-Shear Joints for Determination of the Stress-Strain Behaviour of Adhesives in Shear by Tension Loading

ASTM D 5649–01: Standard Test Method for Torque Strength of Adhesives Used on Threaded Fasteners

**Tensile Tests**


**Mechanical Properties - Other Tests**

SP Technical Research Institute of Sweden

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