



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 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.

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 s^{-1} (Carlberger, Biel and Stigh 2009).

2 Glass

2.1 Glass in general

In EN 1288-1:2000 (CEN, EN 1288-1:2000 Glass in building - Determination of the bending strength of glass - Part 1: Fundamentals of testing glass 2000) glass is described as given below.

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 1. Mechanical and thermal properties of glass (Saint-Gobain Sekurit - Glazing manual 2012, Larcher, et al. 2012).

Density (kg/m^3)	Knoop Hardness (HK)	Compression resistance (MPa)	Modulus of Elasticity (GPa)	Bending strength (MPa)	Poisson's ratio
2500	470	800 – 1000	70	45	0.22
Failure strain (%)	Failure Stress (MPa)	Softening temp. (°C)	Specific heat (J/gK)	Thermal cond. (W/mK)	Thermal exp. (K^{-1})
0.12	84	Approx. 600	0.8	0.8	9×10^{-6}

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 2. Mechanical properties of PVB layer (Xu, Sun, et al. 2011, Larcher, et al. 2012).

	PVB
Density (kg/m³)	870 – 1100
Elastic limit (MPa)	11
Modulus of Elasticity (GPa)	0.100 – 0.220
Failure stress (MPa)	28
Failure strain (%)	200
Poisson's ratio	0.48 – 0.495

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.

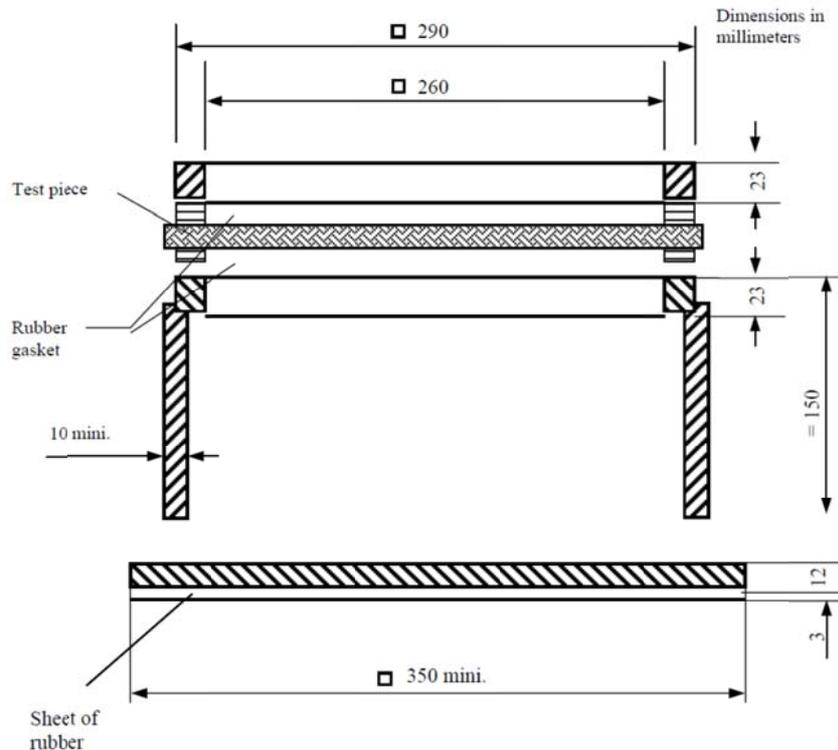
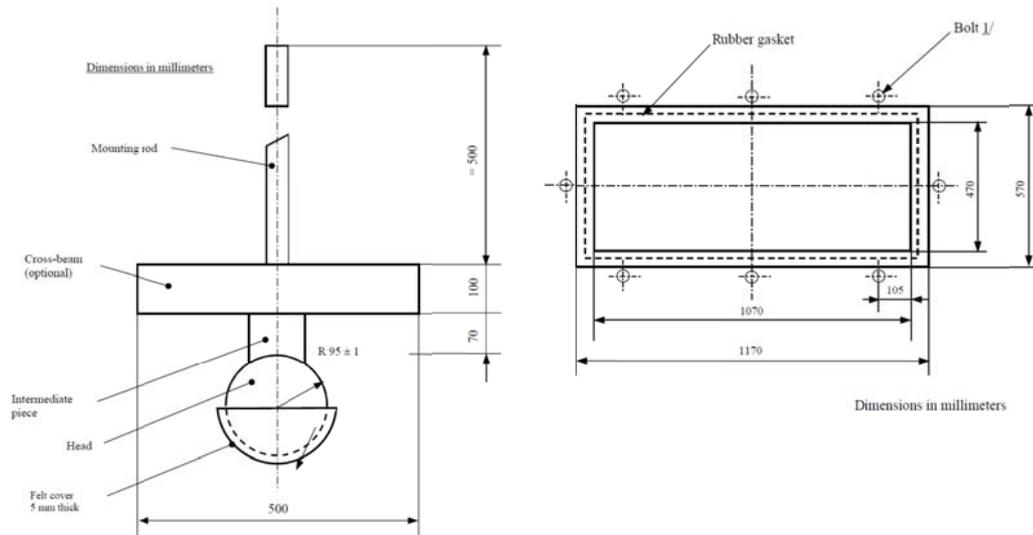


Figure 1. Support for ball tests (Regulation ECE R43 n.d.).



a) Headform weight.

b) Support for headform tests.

Figure 2. Headform test equipment (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.

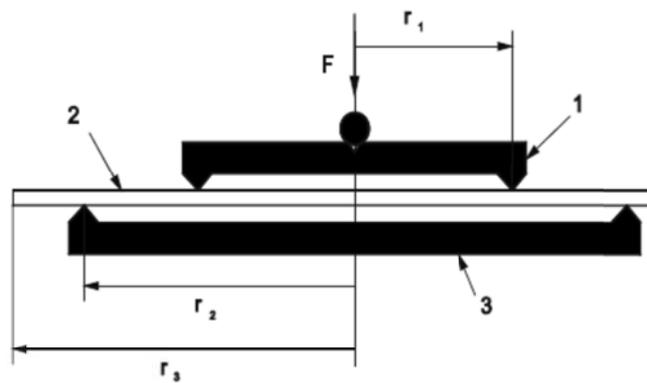
To test the material without the influence of the micro cracks a ring-on-ring test is sometimes used.

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.



- 1 Loading ring
- 2 Specimen
- 3 Supporting ring

Figure 3. Test arrangement for coaxial double ring test (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).

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.

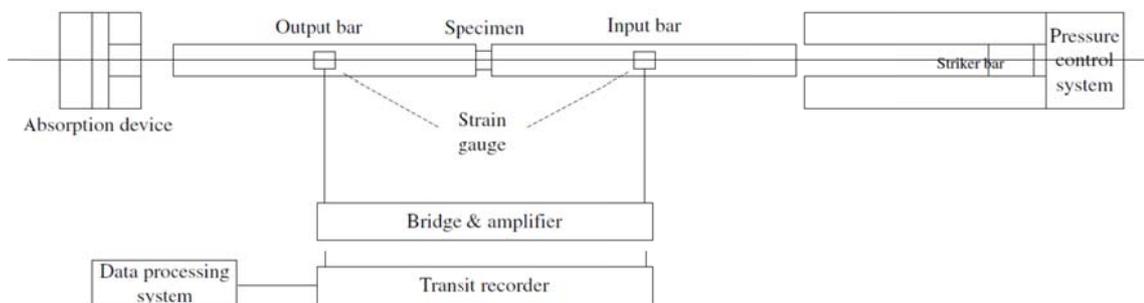


Figure 4. Schematic diagram of split Hopkinson pressure bar test (Xu, Li, et al. 2011).

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).

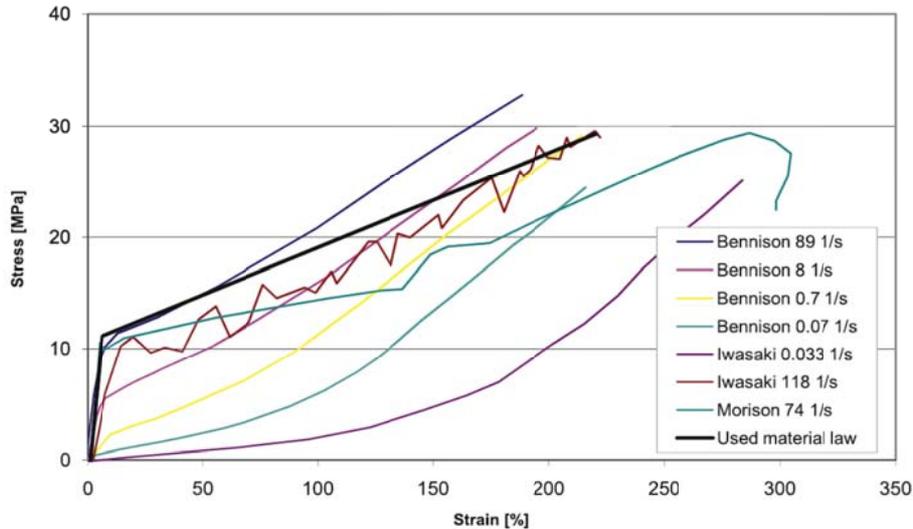


Figure 5. Behaviour of PVB at different strain rates (Larcher, et al. 2012, Morison, Zobec and Franceschet 2007, Iwasaki, Lataillade och Viot 2007, Bennison, et al. 2005).

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.

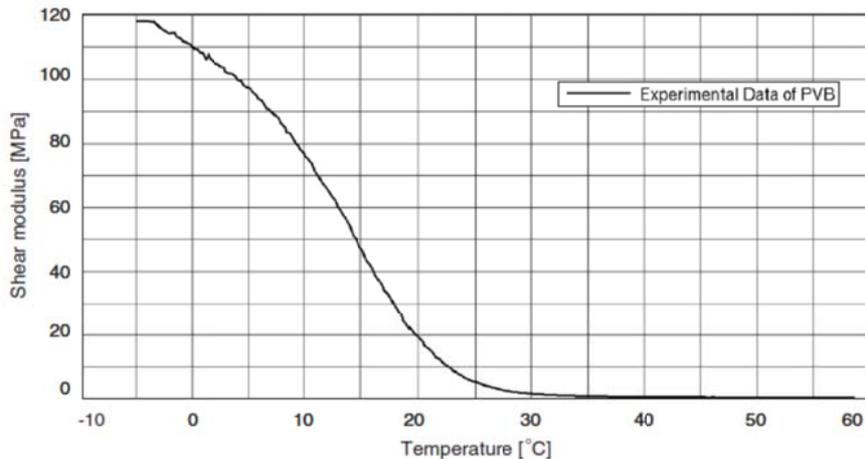


Figure 6. Shear modulus of PVB for different temperatures (Timmell, et al. 2007).

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.

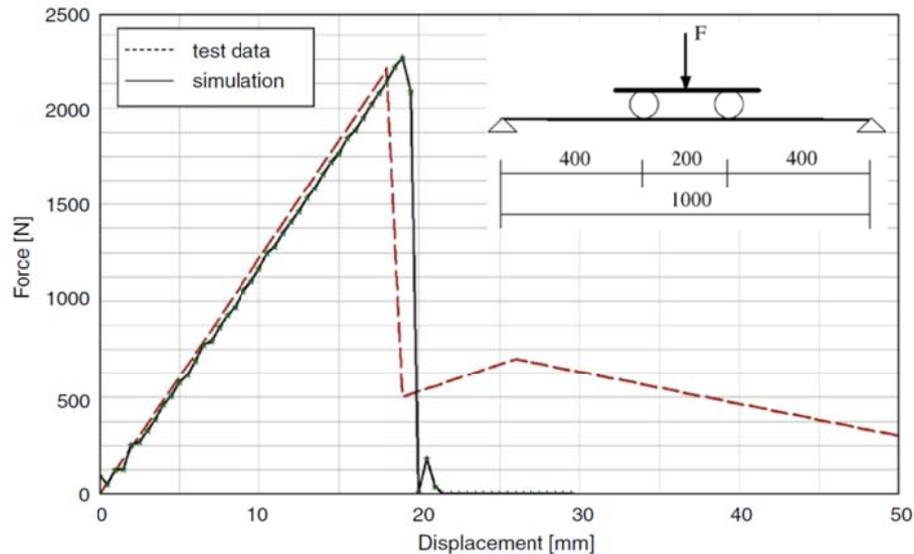


Figure 7. Testing of laminated safety glass in four-point bending (Timmell, et al. 2007).

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).

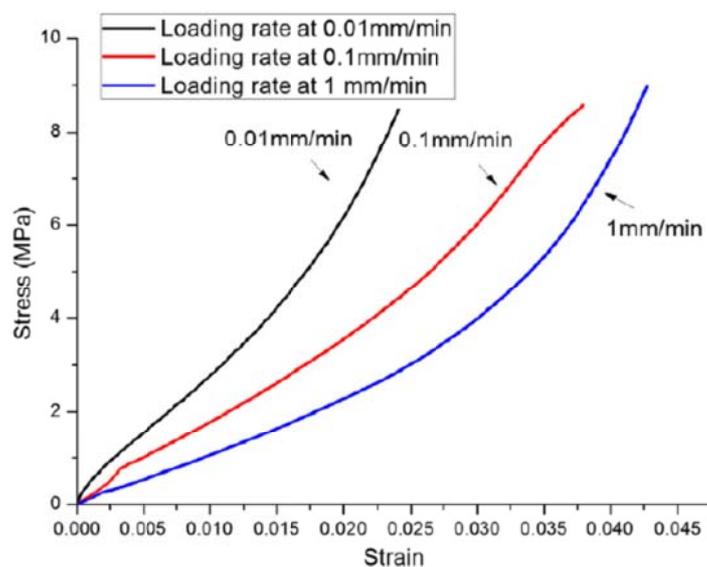


Figure 8. Stress-strain curves obtained from quasi-static compression experiments under three deformation rates (Xu, Li, et al. 2011).

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).

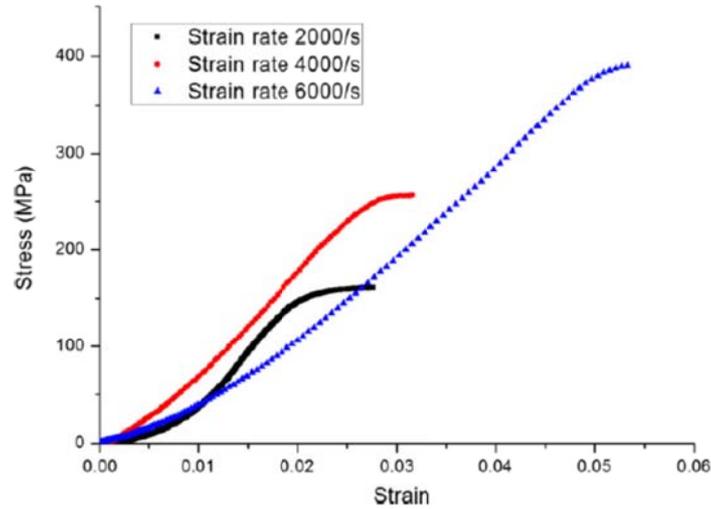


Figure 9. Stress-strain curves obtained from dynamic SHPB experiments at high strain rates (Xu, Li, et al. 2011).

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).

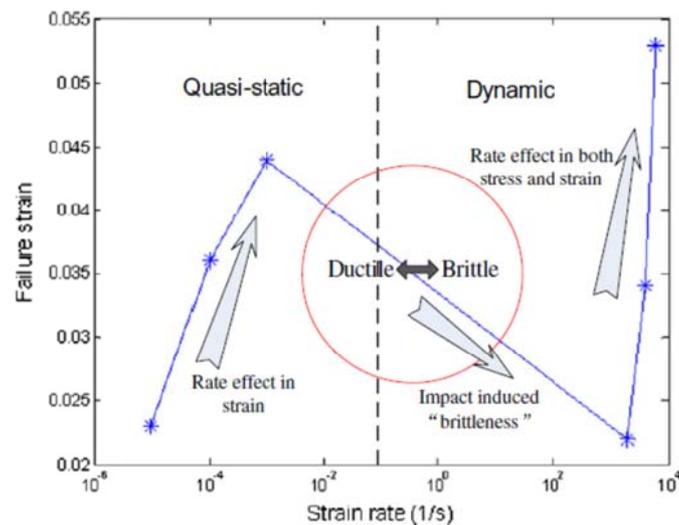


Figure 10. Mechanism of different failure strains at various strain rates of laminated safety glass (Xu, Li, et al. 2011).

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).

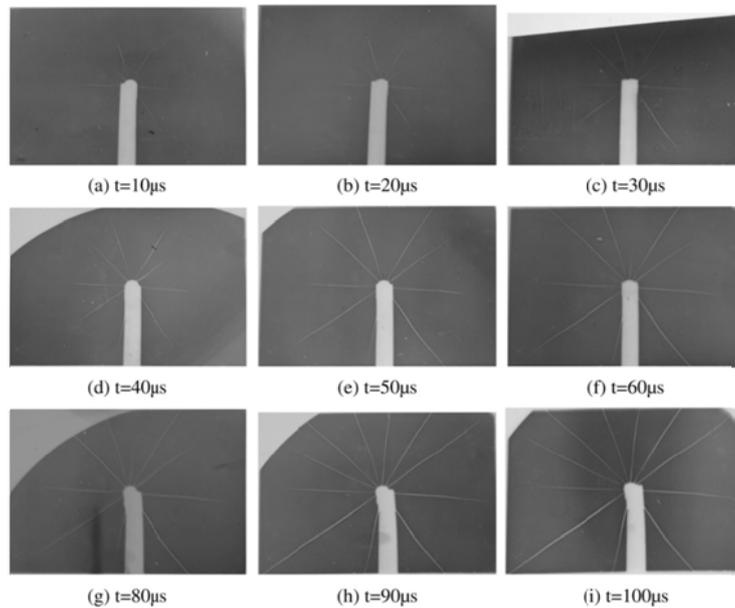


Figure 11. Snapshots of crack propagation with 1 kg of drop weight and 1 m of drop height (Xu, Sun, et al. 2011).

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).

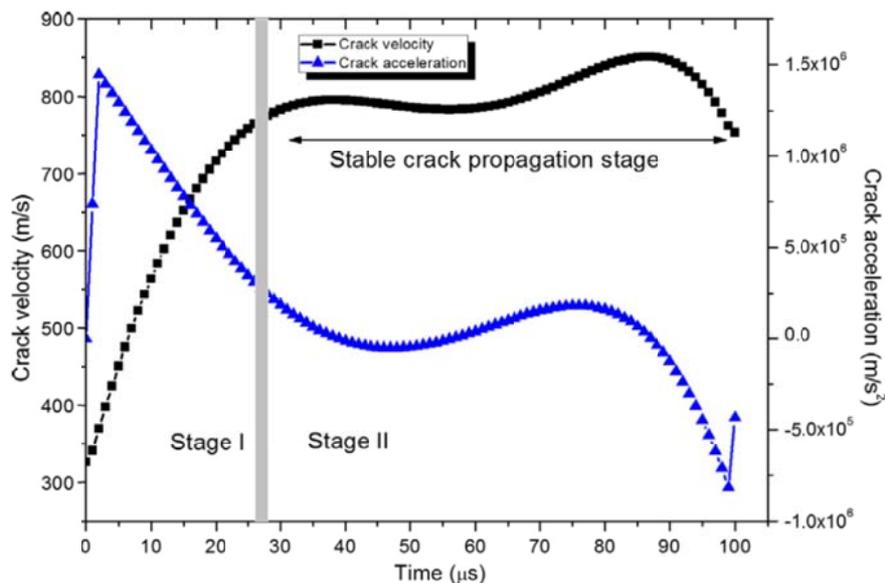


Figure 12. Averaged crack tip velocity and acceleration (Xu, Sun, et al. 2011).

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.

Manufacturer	System name	Adhesive type
Bostik	ISR (Industry Special Range)	Silyl Modified Polymer (SMP)
DOW	BETASEAL™	1 component polyurethane
Henkel	TEROSTAT	1 component polyurethane
Sika	SikaTack Sikaflex	1 component polyurethane

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.

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

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.

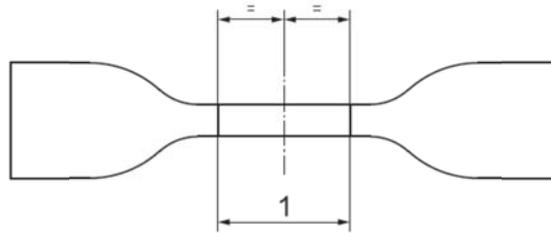


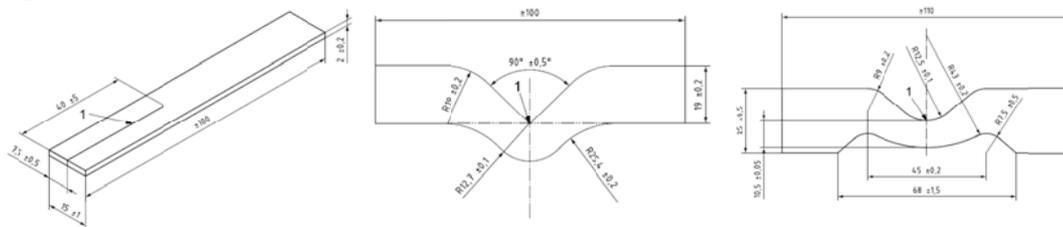
Figure 13. Shape of dumb-bell test pieces (SIS 2012).

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.



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

Figure 14. Specimen geometries according ISO 34. 1 indicates location of cut or nick.

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.

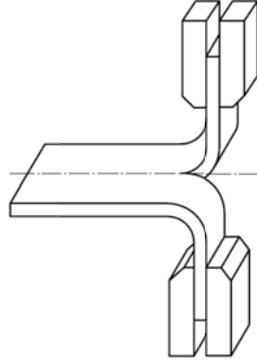


Figure 15. Positioning of trouser test piece in testing machine.

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 tensile lap shear strength is determined according to ISO 4587 (ISO, ISO 4587:2003 - Adhesives - Determination of tensile lap-shear strength of rigid-to-rigid bonded assemblies 2003).

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 $12.5 \text{ mm} \pm 0.25 \text{ mm}$. A typical adhesive thickness is 0.2 mm

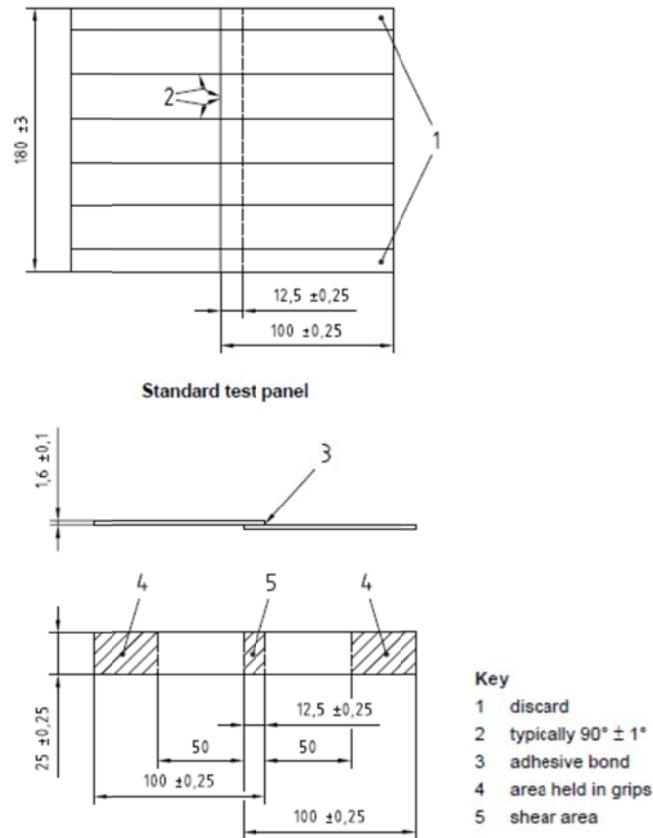


Figure 16. Shape and dimensions (in mm) of test panel and specimen.

Testing at constant speed shall result in fracture within a period of $65 \text{ s} \pm 20 \text{ s}$. 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'hacdec 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).

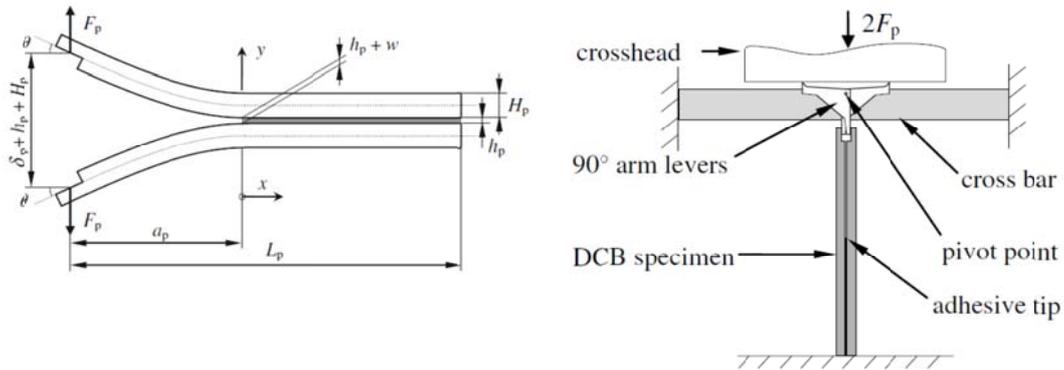
3.2.2 Other test methods

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. z a). In Fig. 17 b) an image showing how the loading might be achieved is depicted (Carlberger, Biel and Stigh 2009).



a) Double cantilever beam specimen. b) Loading of the DCB specimen.
Figure 17. Principle of double cantilever beam testing (Carlberger, Biel and Stigh 2009).

The crosshead speed in the experiments performed by Carlberger et al was 100 mm/s. This crosshead speed resulted in a strain rate around $1.5 \cdot 10^{-3} \text{ s}^{-1}$ 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_s in the figure), shear will take place at the end of the crack.

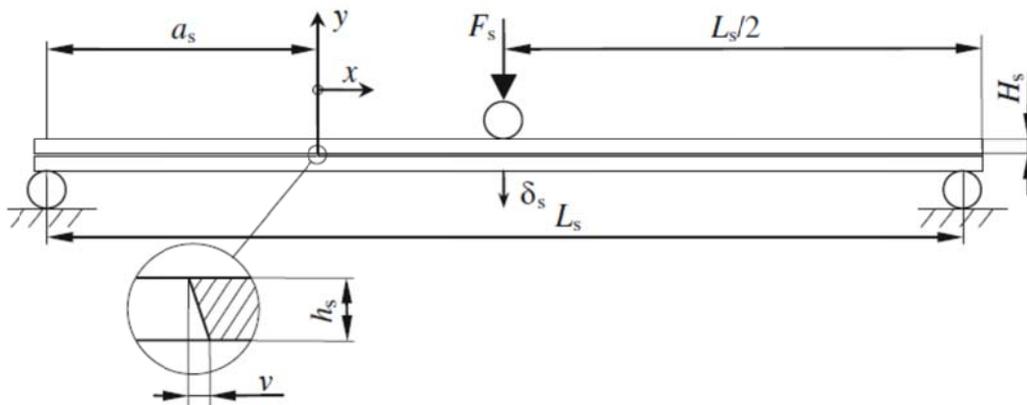


Figure 18. End notch flexure specimen (Carlberger, Biel and Stigh 2009).

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 ($100 - 5000 \text{ s}^{-1}$), split Hopkinson bar test was used with different setups for tension and compression as shown in Fig. 19. For low and intermediate strain rates ($0.1 - 53 \text{ s}^{-1}$) Morin et al. used a high speed hydraulic machine.

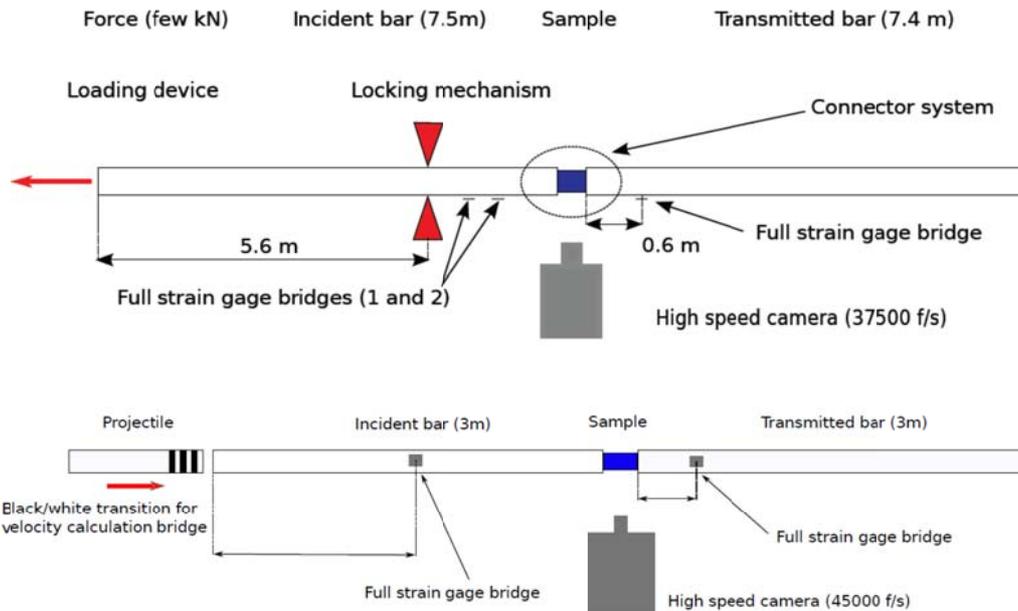


Figure 19. Split Hopkinson test in tension (upper) and compression (lower) (Morin, et al. 2011).

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.

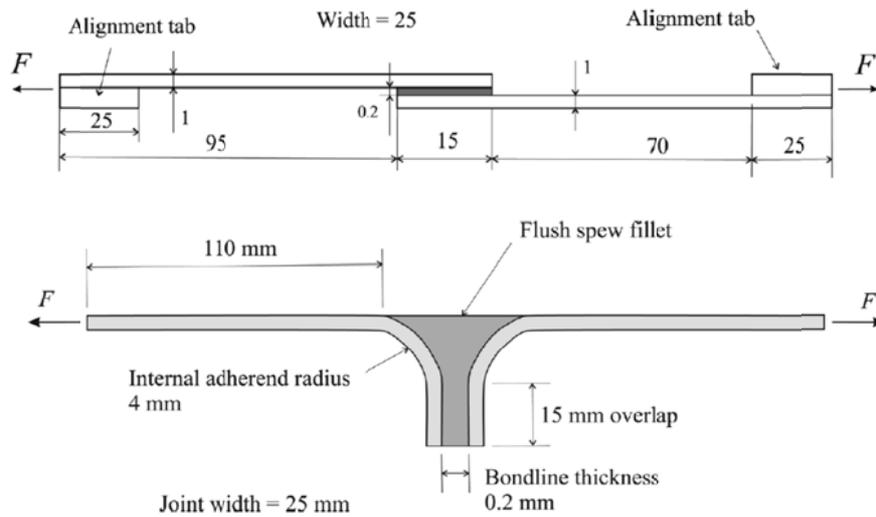


Figure 20. Single lap joint geometry (top) and T-peel joint geometry (bottom). Dimensions in mm.

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.

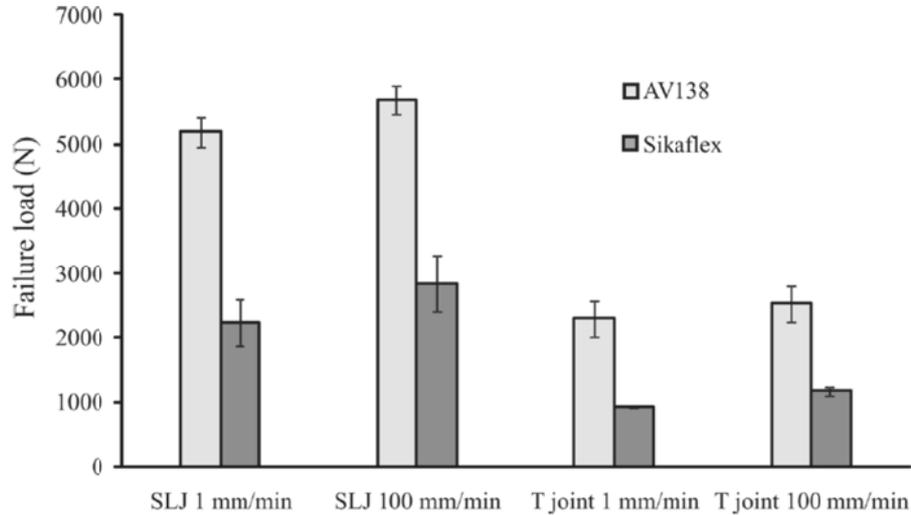


Figure 21. Failure loads for the epoxy adhesive (AV138) and the polyurethane (Sikaflex) in SLJs and T-joints tested in quasi-static conditions under 1 and 100 mm/min (Loureiro, et al. 2010).

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.

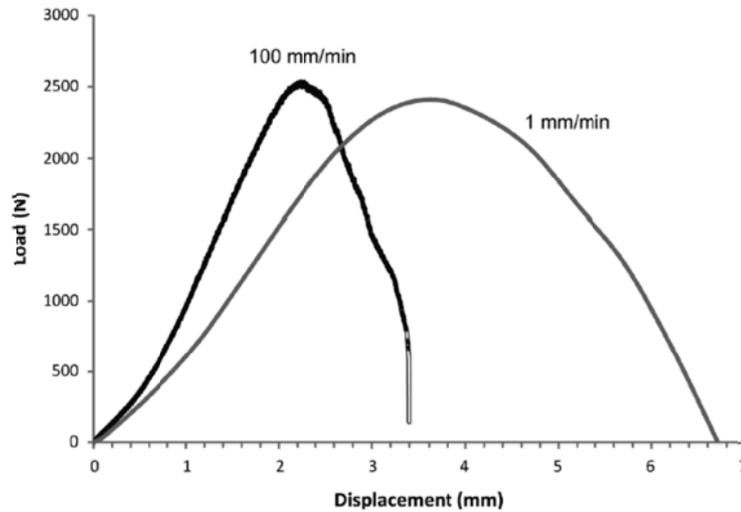


Figure 22. Load-displacement curves of SLJs for the polyurethane adhesive (Loureiro, et al. 2010).

Fatigue tests were also performed for the two joint geometries and adhesives. The fatigue data are shown in Figs. 23-24.

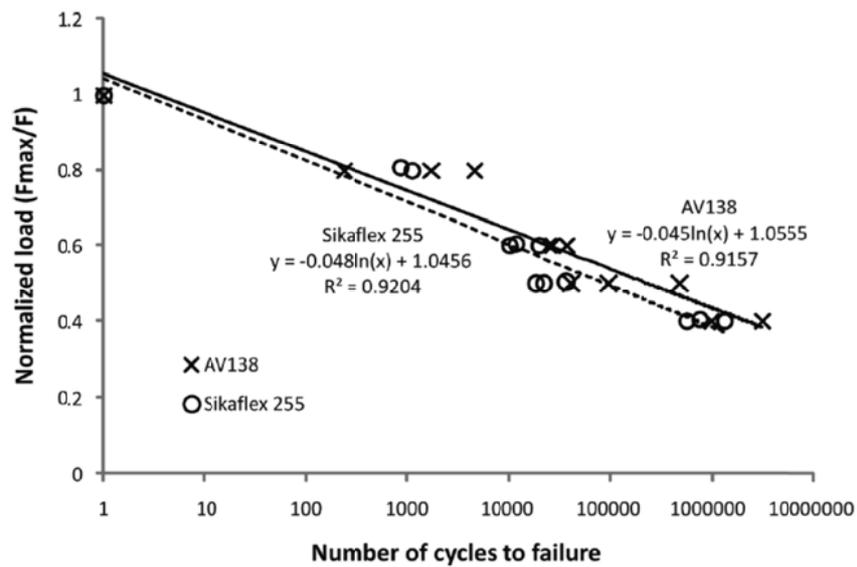


Figure 23. Fatigue life curve of SLJs (Loureiro, et al. 2010).

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.

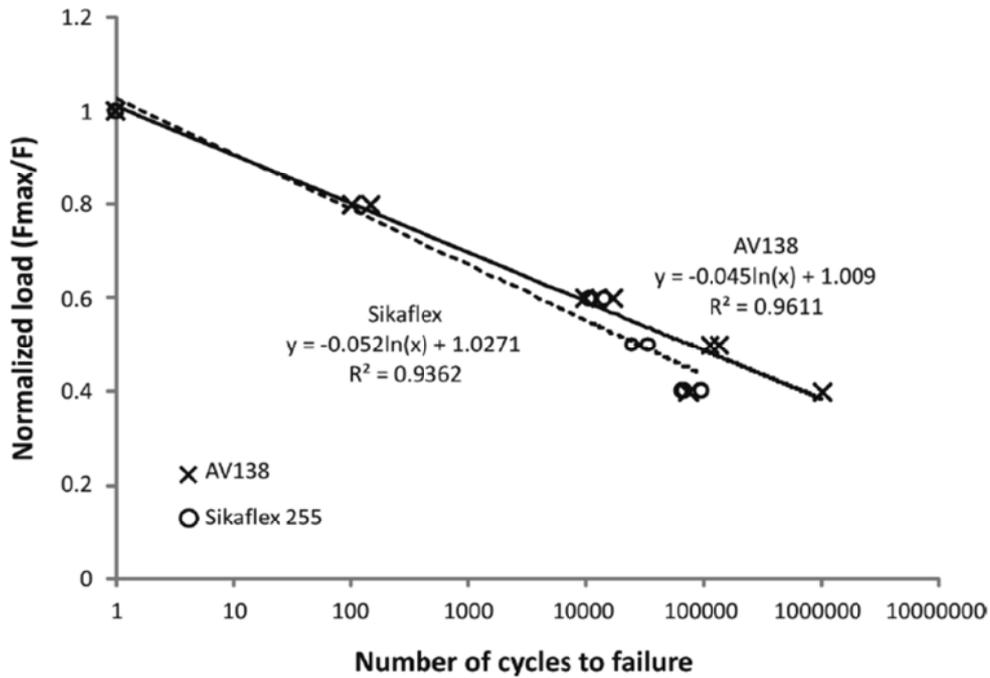


Figure 24. Fatigue life curves of T-joints (Loureiro, et al. 2010).

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

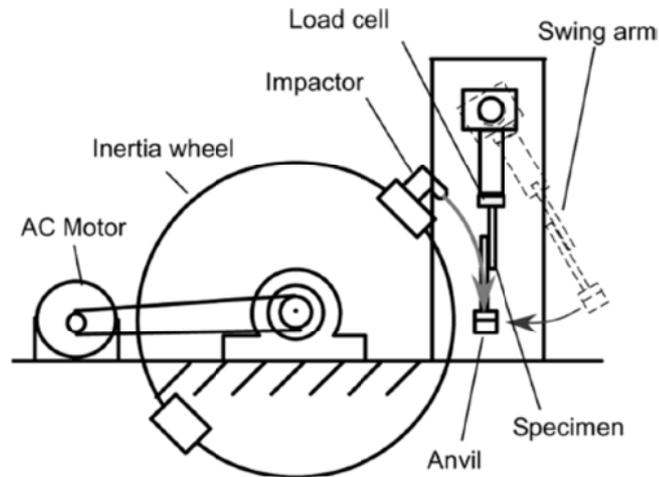


Figure 25. Inertial wheel impact testing equipment (Loureiro, et al. 2010).

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 $1.8 \cdot 10^5$ mm/min.

In Fig. 26 the static failure load at 1 mm/min are compared with the impact loads.

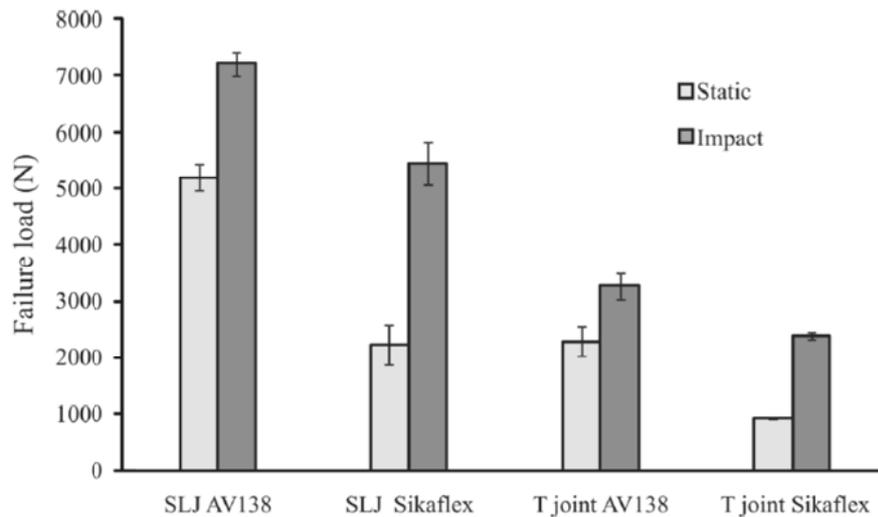


Figure 26. Failure loads of specimens under impact loads, along with static failure loads at 1 mm/min (Loureiro, et al. 2010).

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 $5.3 \cdot 10^{-3}$ and $53 s^{-1}$, respectively.

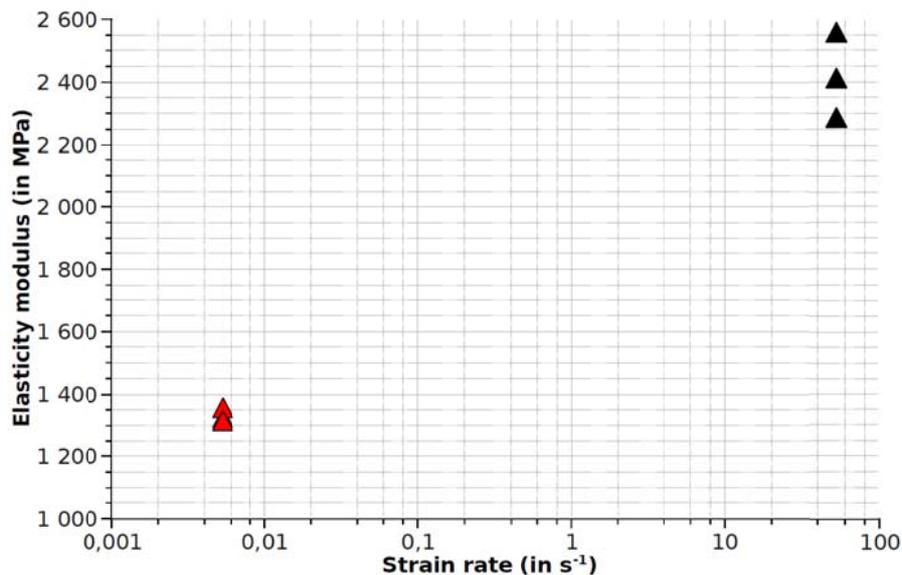


Figure 27. Evolution of the tensile elastic modulus as a function of the strain rate (Morin, et al. 2011).

Regarding plasticity for the tensile tests a visco-plastic behaviour was observed as shown in Fig. 28.

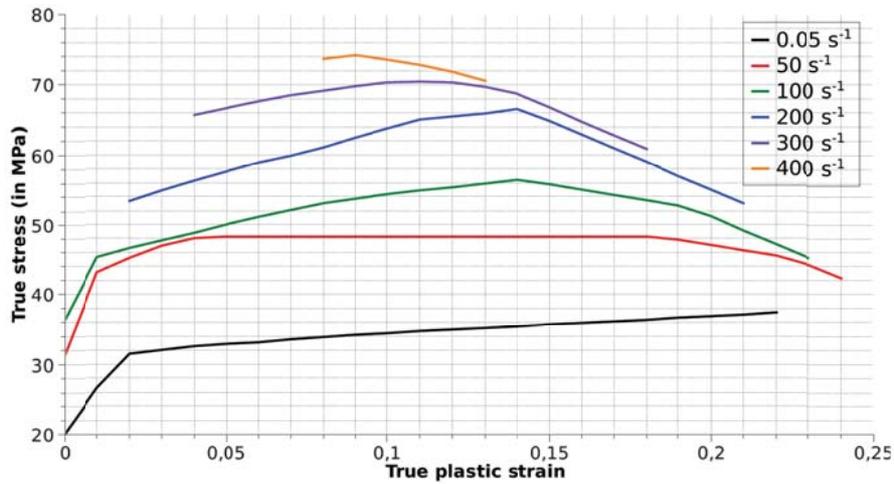


Figure 28. True tensile stress-true plastic strain curves at different strain rates (Morin, et al. 2011).

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.

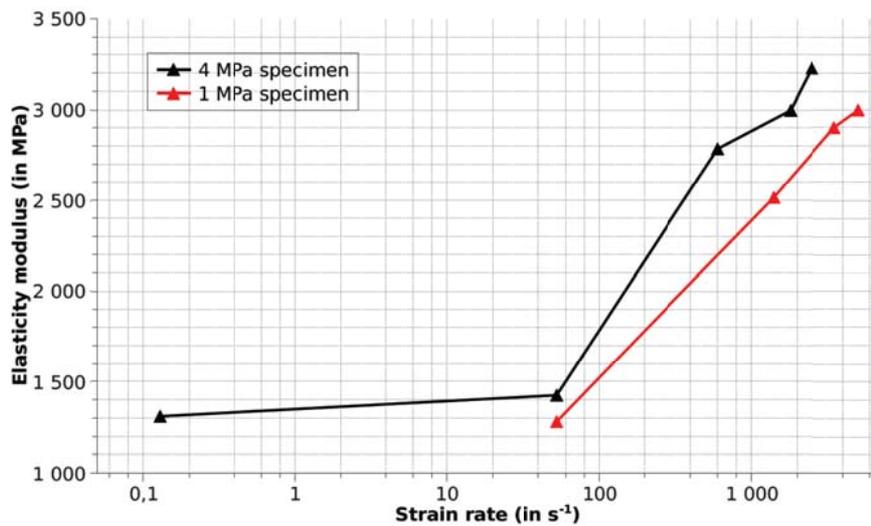


Figure 29. Evolution of the compressive elastic modulus as a function of the strain rate and curing pressure (Morin, et al. 2011).

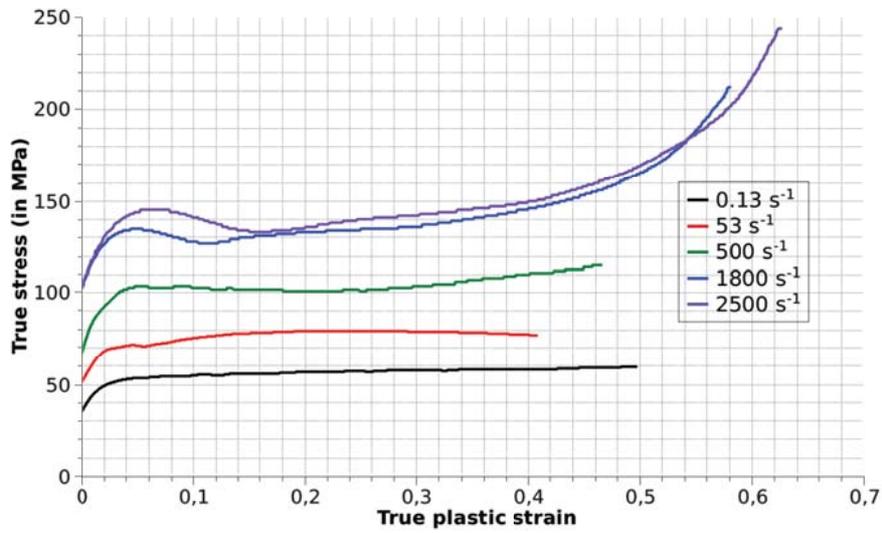


Figure 30. True compressive stress-true plastic strain curves at different strain rates. The curing pressure was 4 MPa (Morin, et al. 2011).

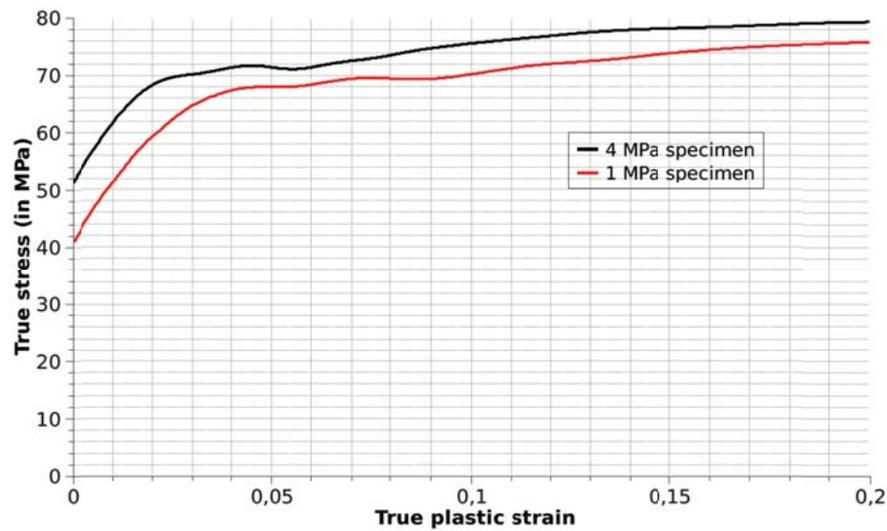


Figure 31. True compressive stress-true plastic strain curves at different curing pressures. The strain rate at testing was 53 s^{-1} (Morin, et al. 2011).

Fig. 31 shows the influence of curing pressure on the stress levels observed at compressive testing at a strain rate of 53 s^{-1} .

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5 Appendices

5.1 Appendix 1. Standard methods for mechanical testing of adhesives

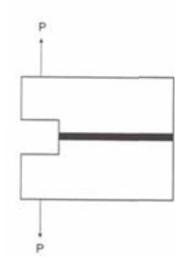
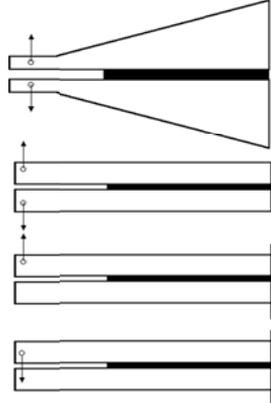
Table A1. Tensile and peel test methods for adhesives.

Test Method	Tensile Butt Joint	T-Peel	Climbing Drum	Floating Roller Method
Principle image of specimen or test set-up				
Mechanical Properties Obtained	Tensile strength/modulus	Peel strength	Peel strength/skin stiffness	Peel strength
Material Quantity Requirements per specimen	Low	Low	High	Low
Typical Specimen Dimensions (mm)	Diameter 15–25 Adherend thickness 12–15	Bond length 150 Width 25 Adherend thickness 0.5–1.0 Arm length 50	Long adherend 300 Short adherend 240 Width 25 Adherend thickness 0.5–5.0	Flexible adherend 250 Rigid adherend 200 Width 25 Adherend thickness 0.5–1.6
Materials Suitable for Testing	1–6	1, 4 and 6 Flexible-flexible adherend	Flexible-rigid adherend 1–6 + sandwich structures	Flexible-rigid adherend 1–6
Cost of Specimen Fabrication/Preparation	Low	Low-moderate	High	Low—moderate
Cost of Testing/Specimen	Low	Low	Low—moderate	Low—moderate
Specimen Fabrication Equipment Requirements	Surface preparation	Surface preparation Bonding+Bonding jig	Surface preparation Bonding+Bonding jig	Surface preparation Bonding+Bonding jig
Specimen Instrumentation Requirements	Extensometer	None	None	Extensometer (2 off)
Test Equipment and Fixture Requirements	Universal test machine + end grips	Universal test machine + end grips	Special test fixture Universal test machine + end grips	Special test fixture Universal test machine + end grips

Fatigue performance	Limited	Unsuitable	Unsuitable	Unsuitable
Creep Performance	Suitable	Possibly	Unsuitable	Unsuitable
Environmental suitability	Suitable	Suitable	Unsuitable	Unsuitable
Data Reduction	Straightforward	Straightforward	Straightforward	Straightforward
Accuracy (Estimated)	To be determined	Large uncertainty (> 30%)	To be determined	To be determined
Standard Test Methods Available	ASTM D 897 ASTM D 2095	ISO 8510: Part 2 ISO 11339 ASTM 1876	ASTM D 3167	ASTM D 3167

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

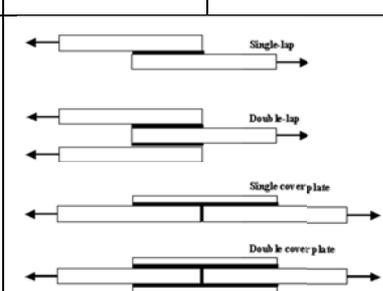
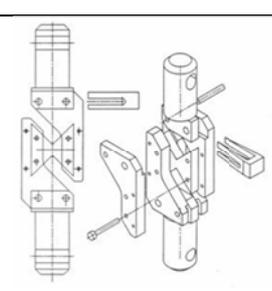
Table A2. Cleavage and Mode I fracture toughness test methods for adhesives.

Test Method	Wedge Cleavage	Compact Tension	DCB (Double Cantilever Beam)	TDCB (Tapered Double Cantilever Beam)
Principle image of specimen or test set-up				
Mechanical Properties Obtained	Fracture energy	Cleavage strength	Mode I fracture toughness	Mode I fracture toughness
Material Quantity Requirements/Specimen	Low	high	Low	High
Typical Specimen Dimensions (mm)	Length 200 Width 25 Adherend thickness 2	Length 25 Width 25 Adherend thickness 12	Length 356 Width 25 Adherend thickness 6.35	Length 240 Width 25 Adherend thickness 12.8
Materials Suitable for Testing	1	1—6	1—6	1 and 6
Cost of Specimen Fabrication/Preparation	Low	Low-moderate	Low	Moderate—High

Cost of Testing/Specimen	Low	Low-moderate	Low-moderate	Low-moderate
Specimen Fabrication Equipment Requirements	Surface preparation	Surface preparation Bonding + bonding jig	Surface preparation	Surface preparation Bonding + Bonding jig
Specimen Instrumentation Requirements	Travelling microscope or video camera	Extensometer for crack opening displacement	Travelling microscope or video camera	Travelling microscope or video camera
Test Equipment and Fixture Requirements	Self-stressed	Universal test machine + loading fixture	Universal test machine + loading fixture	Universal test machine + loading fixture
Fatigue performance	Not suitable	Suitable	Suitable	Suitable
Creep Performance	Possibly	Suitable	Unsuitable	Unsuitable
Environmental suitability	Suitable	Suitable	Suitable	Suitable
Data Reduction	Straightforward	Straightforward	Straightforward	Straightforward
Accuracy (Estimated)	To be determined	To be determined	Large uncertainty	Moderate
Standard Test Methods Available	ASTM D 3762	ASTM D 1062	ASTM D 3433	ASTM D 3433

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

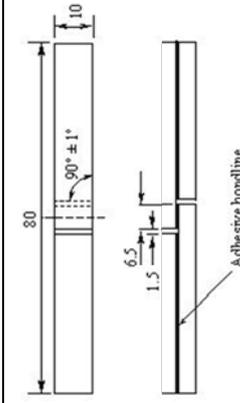
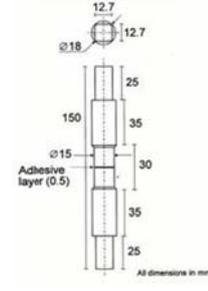
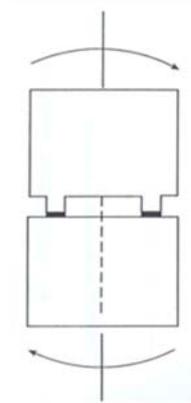
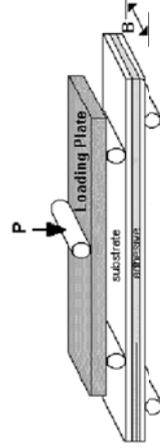
Table A3. Shear test methods for adhesives.

Test Method	Single-Lap	Double-Lap	V-Notched Beam	Arcan
Principle image of specimen or test set-up				
Mechanical Properties Obtained	Shear strength	Shear strength	Shear strength/modulus	Shear strength/modulus
Material Quantity Requirements/Specimen	Low	Low	Low	Low
Typical Specimen Dimensions (mm)	Length 100 Width 25 Adherend thickness 2 Overlap length 25	Length 100 Width 25 Adherend thickness 12 Overlap length 25	Length 76 Width 20 Notch width 12 Adherend thickness 5	Length 52 Width 40 Notch width 12 Adherend thickness 6

Materials Suitable for Testing	1, 3 and 6	1, 3 and 6	1, 3 and 6	1, 3 and 6
Cost of Specimen Fabrication/Preparation	Low	Low-moderate	Moderate	Moderate
Cost of Testing/Specimen	Low	Low-moderate	Low-moderate	Low-moderate
Specimen Fabrication Equipment Requirements	Surface preparation Bonding + bonding jig	Surface preparation Bonding + bonding jig	Surface preparation Bonding + Bonding jig	Surface preparation Bonding + Bonding jig
Specimen Instrumentation Requirements	None	None	Shear extensometer Strain gauges	Shear extensometer Strain gauges
Test Equipment and Fixture Requirements	Universal test machine + loading grips	Universal test machine + loading grips	Universal test machine + loading fixture	Universal test machine + loading fixture
Fatigue performance	Limited	Suitable	Unsuitable	Suitable
Creep Performance	Suitable	Possibly	Unsuitable	Unsuitable
Environmental suitability	Suitable	Suitable	Suitable	Suitable
Data Reduction	Straightforward	Straightforward	Straightforward	Straightforward
Accuracy (Estimated)	Moderate	Low (30%)	Low—moderate (10–20%)	Low—moderate (10–20%)
Standard Test Methods Available	BS 5350: Part C5 BS EN 1465 BS EN ISO 9664 ASTM D 1002 ASTM D 3166	BS 5350: Part C5 BS EN ISO 9664 ASTM D 1002 ASTM D 3166	No adhesive standard ASTM D 5379—PMCs	None

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

Table A4. Shear and Mode II fracture toughness test methods for adhesives.

Test Method	Thick Adherend	Torsion Butt Joint	Napkin Ring	ENF (End-Notched Flexure)
Principle image of specimen or test set-up				
Mechanical Properties Obtained	Shear strength/modulus	Shear strength/modulus	Shear strength	Mode II fracture energies
Material Quantity Requirements/Specimen	Low	Low	Low	Low
Typical Specimen Dimensions (mm)	Length 110 Width 5 Adherend thickness 26 Overlap length 5	Diameter 15–25 Adherend thickness 12–15	Length 356 Width 25 Adherend thickness 6	
Materials Suitable for Testing	1- 6	1—6	1—6	1 and 6
Cost of Specimen Fabrication/Preparation	Low-moderate	Low-moderate	Moderate-high	Low
Cost of Testing/Specimen	Low	Low-moderate	Low-moderate	Low-moderate
Specimen Fabrication Equipment Requirements	Surface preparation Bonding = bonding jig	Surface preparation Bonding + bonding jig	Surface preparation Bonding + bonding jig	Surface preparation Bonding + Bonding jig
Specimen Instrumentation Requirements	Extensometers (2 off)	Rotary extensometer	Rotary extensometer	Travelling microscope or video camera
Test Equipment and Fixture Requirements	Self-stressed	Universal test machine + loading fixture	Torsion test machine + loading fixture	Universal test machine + flexure fixture
Fatigue performance	Not suitable	Suitable	Suitable	Suitable
Creep Performance	Possibly	Suitable	Unsuitable	Unsuitable
Environmental suitability	Suitable	Suitable	Suitable	Suitable
Data Reduction	Straightforward	Straightforward	Straightforward	Straightforward
Accuracy (Estimated)	To be determined	To be determined	To be determined	Large uncertainty (30%)
Standard Test Methods Available	ISO 11003 ASTM D 3165	None	ASTM E 229	None

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

ISO 10354 (1992): Adhesives—Characterization of Durability of Structural-Adhesive-Bonded Assemblies—Wedge Rupture Test

ISO 11343 (2003): Adhesives—Determination of Dynamic Resistance to Cleavage of High-Strength Adhesive Bonds Under Impact Conditions—Wedge Impact Method Peel Tests

ISO 4578 (1997): Adhesives—Determination of Peel Resistance of High-Strength Adhesive Bonds—Floating-Roller Method

ISO 8510 Part 1 (1990) or BS EN 28510–1 (1993): Adhesives—Peel Test for a Flexible-Bonded-to-Rigid Test Specimen Assembly—Part 1: 90 Degree Peel

ISO 8510–2 (1990) or BS EN 28510–2 (1993): Adhesives—Peel Test for a Flexible-Bonded-to-Rigid Test Specimen Assembly—Part 2: 180 Degree Peel

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

ISO 14676 (1997): Evaluation of the effectiveness of surface treatment techniques for aluminium - Wet-peel test by floating-roller method

Shear Tests

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

ISO 6237 (2003): Adhesives—Wood-to-Wood Adhesive Bonds—Determination of Shear Strength by Tensile Loading

ISO 9653 (1998): Adhesives—Test method for Shear Impact Strength of Adhesive Bonds

ISO 10123 (1990): Adhesives—Determination of Shear Strength of Anaerobic Adhesives Using Pin-and-Collar Specimens

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

ISO 11003 Part 1 (2001): Adhesives—Determination of Shear Behaviour of Structural Adhesives—Part 1: Torsion Test Method Using Butt-Bonded Hollow Cylinders

ISO 11003 Part 2 (2001): Adhesives—Determination of Shear Behaviour of Structural Adhesives—Part 2: Tensile Test Method Using Thick Adherends

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

Tensile Tests

BS EN ISO 527 Part 1 (1993): Plastics—Determination of Tensile Properties—General Principles

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

BS EN ISO 9664 (1995): Adhesives—Test Methods for Fatigue Properties of Structural Adhesives in Tensile Shear

Mechanical Properties - Other Tests

BS EN ISO 178 (1998): Plastics—Determination of Flexural Properties

ISO 604 (2002): Plastics—Determination of Compressive Properties

BS EN ISO 11403 Part 1 (2000): Plastics—Acquisition and Presentation of Comparable Multipoint Data—Part 1: Mechanical Properties

5.2.2 ASTM STANDARDS - Number/Title

Cleavage Tests

ASTM D 1062–02: Standard Test Method for Cleavage Strength of Metal-to-Metal Adhesive Bonds

ASTM D 3433–99: Standard Test Method for Fracture Strength in Cleavage of Adhesives in Bonded Metal Joints

ASTM D 3807–98 (2004): Standard Test Method for Strength Properties of Adhesives in Cleavage Peel by Tension Loading (Engineering Plastics-to-Engineering Plastics)

ASTM D 5041–98 (2004): Standard Test Method for Fracture Strength in Cleavage of Adhesives in Bonded Joints

Peel Tests

ASTM D 1781–98 (2004): Standard Test Method for Climbing Drum Peel for Adhesives

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

ASTM D 3167–03a (2004): Standard Test Method for Floating Roller Peel Resistance of Adhesives

Shear Tests

ASTM D 905–03: Standard Test Method for Strength Properties of Adhesive Bonds in Shear by Compression Loading

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 3044–94 (2000): Standard Test Method for Shear Modulus of Wood-Based Structural Panels

ASTM D 3163–01: Standard Test Method for Determining Strength of Adhesively Bonded Rigid Plastic Lap-Shear Joints in Shear by Tension Loading

ASTM D 3164–03: Standard Test Method for Strength Properties of Adhesively Bonded Plastic Lap-Shear Sandwich Joints in Shear by Tension Loading

ASTM D 3165–00: Standard Test Method for Strength Properties of Adhesives in Shear by Tension Loading of Single-Lap-Joint Laminated Assemblies

ASTM D 3528–96 (2002): Standard Test Method for Strength Properties of Double Lap Shear Adhesive Joints by Tension Loading

ASTM D 3983–98(2004): Standard Test Method for Measuring Strength and Shear Modulus of Nonrigid Adhesives by the Thick-Adherend Tensile-Lap Specimen

ASTM D 4562–01: Standard Test Method for Shear Strength of Adhesives Using Pin-and-Collar Specimen

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

ASTM D 5379/D5379M-98: Standard Test Method for Shear Properties of Composite Materials by the V-Notched Beam Method

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

ASTM D 897–01: Standard Test Method for Tensile Properties of Adhesive Bonds

ASTM D 2095–96(2002): Standard Test Method for Tensile Strength of Adhesives by Means of Bar and Rod Specimens

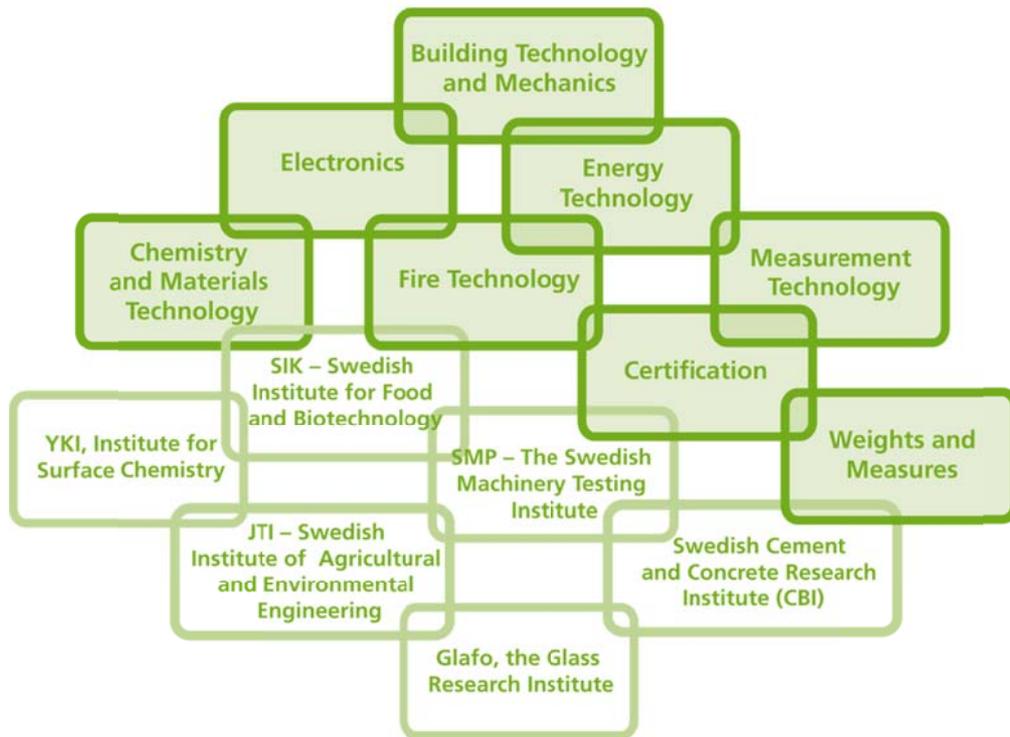
Mechanical Properties - Other Tests

ASTM D 695–02a: Standard Test Method for Compressive Properties of Rigid Plastics

ASTM D 1995–92 (2004): Standard Test Methods for Multi-Modal Strength Testing of Autohesives (Contact Adhesives)

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