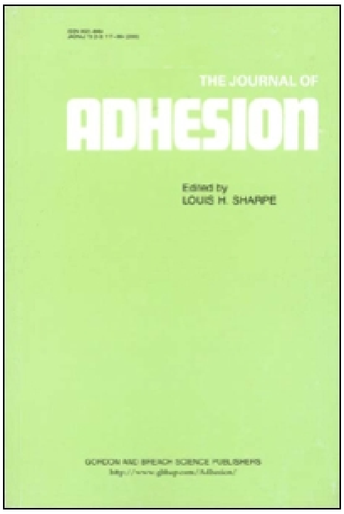


The Journal of Adhesion
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713453635

Mechanical Characterization of Flexible Adhesives

Mariana D. Banea[a]; Lucas F. M. da Silva[b]
[a] Instituto de Engenharia Mecânica (IDMEC), Porto, Portugal [b] Departamento de Engenharia Mecânica e Gestão Industrial, Faculdade de Engenharia, Universidade do Porto, Porto, Portugal

To cite this Article Banea, Mariana D. and da Silva, Lucas F. M.(2009) 'Mechanical Characterization of Flexible Adhesives', The Journal of Adhesion, 85: 4, 261 — 285
To link to this Article: DOI: 10.1080/00218460902881808
URL: http://dx.doi.org/10.1080/00218460902881808

The Journal of Adhesion, 85:261–285, 2009

ISSN: 0021-8464 print/1545-5823 online
DOI: 10.1080/00218460902881808

Mechanical Characterization of Flexible Adhesives

Mariana D. Banea[1] and Lucas F. M. da Silva[2]
[1]Instituto de Engenharia Mecânica (IDMEC), Porto, Portugal
[2]Departamento de Engenharia Mecânica e Gestão Industrial, Faculdade de Engenharia, Universidade do Porto, Porto, Portugal

In this paper, the performances of two different adhesive types—a polyurethane and a high temperature thixotropic adhesive sealant, room temperature vulcanizing (RTV) silicone rubber—were studied through adhesive joint tests. The standard Thick Adherend Shear Test (TAST) was performed in order to measure the shear properties of the adhesives. Single lap joints (SLJs) were fabricated and tested to assess the adhesive performance in a joint. The influence of temperature on the lap shear strength of the adhesives was investigated. It is shown that the lap shear strength of both adhesives is affected by variation of temperature. The effect of bondline thickness and overlap length on the lap shear strength of the adhesives was studied. The reduction of failure load with increasing the bondline thickness is a very common situation when dealing with structural adhesives. For the low strength flexible adhesive Sikaflex® 552 the failure load as well as the overall stiffness of the SLJs decreases as the bondline gets thicker, whereas for AS1805 RTV adhesive the failure loads increase as the bondline gets thicker. Also, in contrast to joints with brittle adhesives, the failure loads of joints with flexible adhesives increase almost proportionally with increasing overlap length. Fatigue tests were also performed and show a low variability in the results.

Keywords: Fatigue; Lap shear strength; Polyurethane adhesive; RTV adhesive; Temperature tests

1. INTRODUCTION

In modern bonding applications, flexible adhesives play an important role. Flexible adhesives have low elastic modulus but high extensions to failure. However, the advantageous properties of flexible adhesives

Received 31 October 2008; in final form 29 December 2008.
Presented in part at the 2nd International Conference on Advanced Computational Engineering and Experimenting (ACE-X 2008), Barcelona, Spain, 14–15 July, 2008
Address correspondence to Lucas F. M. da Silva, Departamento de Engenharia Mecânica e Gestão Industrial, Faculdade de Engenharia, Universidade do Porto, Rua Dr. Roberto Frias, 4200-465 Porto, Portugal. E-mail: lucas@fe.up.pt

in sustaining large strains and distributing peel forces more evenly on the bonded substrates lead to their use for structural joining applications in various industries. These adhesives are predominantly used when considerable expansion and contraction is expected in the joint, flexibility is required [as in the case when materials with different coefficients of thermal expansion (CTEs) such as different metals, fiber-reinforced plastics, or glass panels, have to be joined and thermal stresses must be avoided], or good gasket or sealing properties are necessary. Also, that they properly resist impact and vibration.

Relatively few data are available relative to the mechanical properties of flexible adhesives [1]. For example, the stress–strain characteristics of two moisture curing room temperature vulcanizing (RTV) elastomeric adhesives were investigated in the tensile mode and in the lap shear configuration by Geiss and Vogt [2]. They found that ageing in a humid atmosphere significantly influenced the mechanical properties of the adhesives.

Two different adhesive types—a polyurethane and a high temperature thixotropic adhesive sealant RTV silicone rubber—were studied through adhesive joint tests in this study. Polyurethane and RTV silicone rubber adhesives cure from the moisture in the air and form low strength structural joints. Polyurethanes have good low temperature properties, are easily processable, and bond well to many substrates. They find their greatest use in the automotive industry. On the other hand, silicone adhesives and sealants retain excellent properties over a temperature range from nearly 300°C down to the cryogenic range and are used where organic materials cannot withstand harsh environmental conditions, where superior reliability is required, or where the durability of silicone gives an economic advantage. Silicone joints are designed to utilize the good peel strength of the silicone elastomer rather than its tensile or lap shear properties [3].

In order to properly design a joint, the adhesive behavior has to be characterized. Thus, to determine the stresses and strains in adhesive joints in a variety of configurations, it is necessary to know the mechanical properties of the adhesive.

The influence of temperature on the lap shear strength of flexible adhesives is an important factor to consider as they are designed for use in adhesive joints that undergo significant dimensional changes during their service life. For example, one of the applications of RTV adhesives is to bond the ceramic tiles to the aluminium fuselage of the space shuttle. The most significant factors that determine the strength of an adhesive joint when used over a wide temperature range are the CTE (especially when compared with the CTE of the substrates) [4] and changes in the mechanical properties of the adhesive with temperature [5–7]. Studies that present experimental results of adhesive joints with structural adhesives (especially epoxies) as a function of temperature generally show a decrease in strength with increasing [8,9] and decreasing temperatures [5,10]. At high temperatures the cause is the low adhesive strength while at low temperatures it is the high thermal stresses. Adams *et al.* [8] studied the performance of single lap joints with epoxy adhesives at low and room temperatures. They investigated the effects of adherend mismatch, shrinkage, and adhesive properties on the stress state of lap joints. Owens and Lee-Sullivan [11] tested single lap joints with a rigid and a flexible epoxy adhesive at room temperature and at $-40^{\circ}C$ in quasi-static conditions. They studied stiffness loss due to crack growth in composite-to-aluminium joints. Results showed that the joint stiffness is more affected by the response of the adherends to the test temperature than by the modulus of the thin adhesive layer.

However, little is known in terms of temperature when it comes to flexible adhesives such as polyurethanes and silicones. The properties of adhesives and sealants over the range of service temperatures need to be studied for each type of application. For example, the polyurethanes find major uses in the automotive industry where the adhesive joints need to withstand temperatures between -40 and $80^{\circ}C$ [12], so that the adhesive joints must be characterized over these temperatures. On the other hand, silicone adhesives for aerospace applications need to withstand very high temperatures, typically in excess of $200^{\circ}C$.

A key parameter in the testing of adhesive joints is the glass transition temperature (T_g) of the adhesive. When the adhesively bonded joints are tested below this temperature, the adhesive will behave like a low-strain rigid material while above this temperature it will have a more rubber-like behaviour. For example, common epoxy adhesives have a T_g above room temperature and are, therefore, in the glassy state at room temperature while elastomeric adhesives (the case of polyurethane and RTV silicone studied here) have a T_g below $0^{\circ}C$ and are in a rubbery state at room temperature, so that they have different behaviour in adhesively bonded joints when tested as a function of temperature.

The effect of the bondline thickness on single lap joints is well documented in the literature. Most of the results are for typical structural adhesives and show that the lap joint strength decreases as the bondline increases [13–15]. There are many theories that attempt to explain this fact. Some researchers [15] explained that an increase in the bondline thickness increases the probability of having internal imperfection in the joint (voids and microcracks), which will lead to premature failure of the joints. Gleich *et al.* [16] showed with finite element analysis on single lap joints that increases in the interface stresses (peel and shear) as the bondline gets thicker causes the failure load of a bonded joint to decrease with increasing bondline thickness. They found that for the low bondline thickness range an optimum distribution of stresses along the joint interface exists for maximum joint strength. Crocombe [17] showed that thicker single lap joints have a lower strength determined by the plasticity of the adhesive. Grant *et al.* [18] found a reduction in joint strength with increased bondline thickness when testing SLJs for the automotive industry with an epoxy adhesive. The strength reduction was attributed to the higher bending moments for the lap joints with thick bondlines due to the increase in the loading offset. However, it is known that small variations in bondline thickness can result in significant changes in bond strength and that for comparative studies, careful consideration should be given to ensure that the stress and strain distributions (*i.e.*, maximum peel and shear stresses at the ends of the joint) for different systems are at least similar [19]. For example, Bryant [20] tested SLJs with elastomeric flexible adhesives and concluded that it was the applied strain rate that was responsible for the performance of joints with different bondline thicknesses. Crocombe [17] suggested that this is not the case for modern structural adhesives, which are not so strain rate dependent. More recently, Giannis [21] tested two flexible sealants, choosing the applied crosshead rate for each joint and concluded that the reduction in joint strength with increasing bondline thickness could not be explained by Bryant's suggestion.

The overlap length is another parameter that can affect the joint strength. Studies showed that, in contrast to joints with brittle adhesives, the joint strength of joints with ductile adhesive increases almost proportionally with increasing overlap length [22,23].

The fatigue behavior of adhesively bonded joints is also important and is influenced by many factors, such as the adhesive type, the adherends, joint geometry, environmental conditions, loading, and the quality of the joint fabrication process. For these reasons, fatigue tests can provide only comparative data and not design data. Although considerable research has been conducted to investigate the fatigue performance of adhesively bonded joints [24–27], the fatigue behavior has to be investigated for every particular adhesive/adherend system and application. As is known, generally, crack propagation resistance and fatigue resistance is greater with tough, flexible adhesives rather than with brittle adhesives. This is mainly due to both more uniform stress distributions and high internal energy damping with the more flexible adhesives.

The objective of the present study was to investigate the mechanical behavior of flexible adhesives to further develop ductile joints with these adhesives. The influence of adhesive thicknesses and the overlap on the lap shear strength was studied. Fatigue behavior of the RTV adhesive was also investigated.

2. EXPERIMENTAL

2.1. Adhesives Selected

Two adhesives were chosen for this study: Sikaflex 552, a one-component polyurethane hybrid adhesive supplied by Sika Portugal S.A. (Porto, Portugal) and AS1805, a high temperature thixotropic adhesive sealant, room temperature vulcanizing (RTV) silicone rubber supplied by ACC Silicones Ltd (Bridgwater, UK). The working temperature of Sikaflex 552 is from −40 to 90°C while for AS1805 RTV adhesive it is from −50 to 300°C.

2.2. Specimens Manufacture

2.2.1. TAST

For the Thick Adherend Shear Test (TAST), steel substrates of dimensions $110 \times 25 \times 12\,mm^3$ (see Fig. 1) were used. The joint surfaces were grit blasted and degreased with acetone prior to the application of the adhesive. The bondline thickness was nominally 0.7 mm and the length of the overlap test section was 5 mm. Two spacers (1.5 mm thick) were inserted in the gaps between the adherends after the application of the adhesive and prior to curing in order to provide the necessary spacing between the two adherends. These spacers were

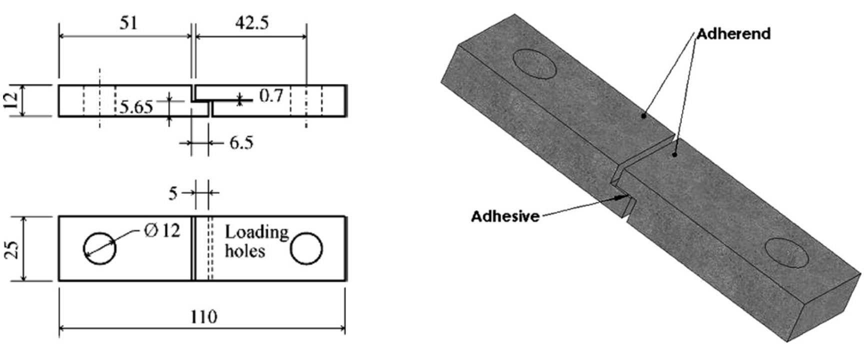


FIGURE 1 Standard TAST specimen (dimensions in mm).

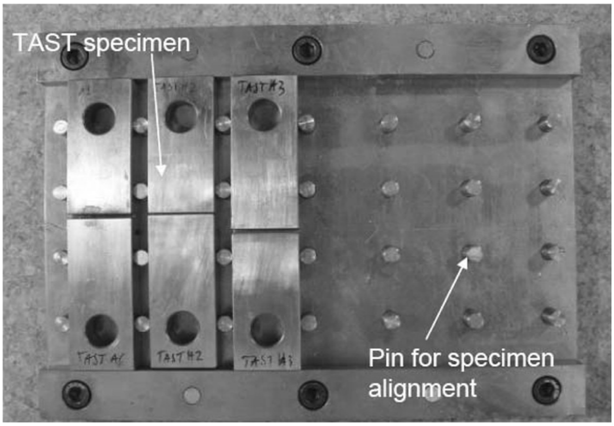


FIGURE 2 Mould for TAST specimen fabrication.

removed after the adhesive was cured. A specially designed mould with spacers for correct alignment of the specimens was used and is shown in Fig. 2. Sikaflex 552 and AS1805 adhesives were cured at room temperature for a week.

Prior to testing, each specimen must be dimensioned for use in calculations and to assure conformity to the standards dimension set out in ISO 11003-2:1993 [28]. Measurements for each specimen were taken and recorded for the width, length, and thickness of the bondline.

2.2.2. *Single Lap Joints*

For Sikaflex 552 adhesive, mild steel substrates (the adherends were considered to be almost infinitely rigid in comparison with the low modulus polyurethane adhesive) of 2 mm thickness and 25 mm width were used (see geometry in Fig. 3).

Aluminium alloy 6082-T651 (Al SiMgMn) substrates with a thickness of 3 mm and 25 mm width were used for SLJs made with AS1805 RTV adhesive.

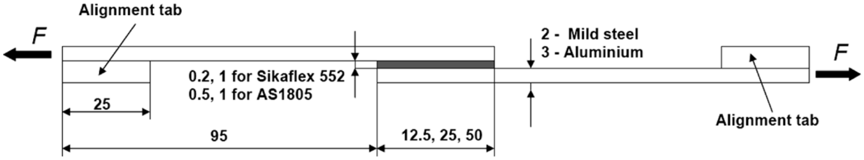


FIGURE 3 Single lap joint specimen geometry (dimensions in mm).

The length of the overlap was 12.5, 25, and 50 mm. The bond line thickness was 0.2 and 1 mm for Sikaflex 552 adhesive and 0.5 and 1 mm for AS1805 RTV adhesive. In order to achieve that, packing shims of different thicknesses were used in order to provide the necessary spacing between the two adherend halves. The geometry of the lap shear joint specimens used is shown in Fig. 3.

For Sikaflex 552 adhesive SLJs, the joint surfaces were grit blasted and degreased with acetone prior to the application of the adhesive.

For AS1805 RTV adhesive SLJs, in order to access the effect of the aluminium adherend surface treatment on the joint performance, two surface treatments were used: a) grit blasting (the bonding area was initially degreased with acetone, grit blasted, and again cleaned with acetone before the application of the adhesive) and b) chemical etching (etching in chromic acid solution for 10 min at 60°C, rinsing under tap then distilled water, and air drying). The mechanical and chemical treatment of the surface was performed just prior to the bonding process in order to avoid the formation of new oxide films.

A mold with spacers for correct alignment of the substrates was used (see Fig. 4). The substrates were bonded and then the joints were left under pressure for 24 hours at room temperature in a hydraulic press. They were then removed from the mold and left for another 10 days to fully cure the adhesive, following the manufacturer's suggested curing conditions (25°C and 50 RH for Sikaflex 552 adhesive, and 25°C and 65 RH for AS1805 RTV adhesive). After the end of the curing process, any excess adhesive was carefully removed.

2.3. Test Method

2.3.1. TAST

The TAST was performed at room temperature on a MTS servohydraulic machine (MTS, Eden Prairie, MN, USA), model 312.31, at

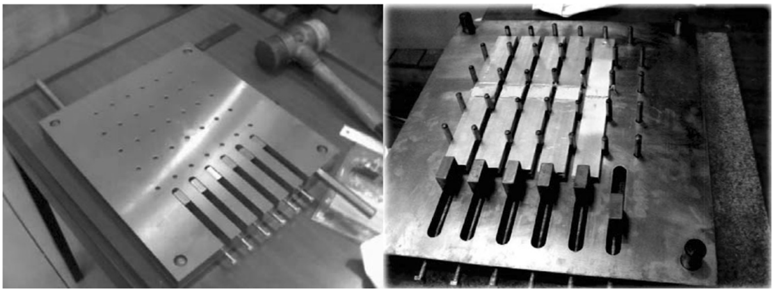


FIGURE 4 Mould for SLJ specimen fabrication.

a constant crosshead rate of 1 mm/min. For load measurements 10% of the capacity of the load cell (25 kN) was used. The displacement was measured with a 25 mm length MTS extensometer. As the extensometer is mounted on the metallic substrate, the extensometer measures not only the displacement of the adhesive, but also the displacement of the adherend. However, da Silva *et al.* [29] showed that the steel deformation can be neglected in the case of flexible adhesives, so that the adhesives displacement can be measured by the MTS extensometer method. Three joints were tested for each adhesive.

2.3.2. Single Lap Joint Tests

Testing was conducted at room temperature at a constant displacement rate of 1 mm/min using the MTS 312.31 servo-hydraulic machine. Loads and displacements to failure were recorded.

SLJs were tested at high temperature using a universal testing machine, Instron model 4208 (Instron Co., High Wycombe, UK), under a constant crosshead rate of 1 mm/min. A load cell of 5 kN was used. An Instron extensometer (50 mm gauge length) was used to record the adhesive displacement. For the high and low temperature tests the environmental chamber of the machine was used to reach the desired temperature: 80 and -40°C for Sikaflex 552 and 100, 200, and 300°C for AS1805 RTV adhesive. For low temperature testing at -40°C the Instron environmental chamber was cooled using solid carbon dioxide.

Three joints were tested to failure at each temperature. For each joint tested, load-displacement curves were produced.

2.3.3. Fatigue Tests

Fatigue was investigated by testing the AS1805 RTV adhesive SLJ specimens in force control with a sinusoidal waveform, load ratio (minimum to maximum load) of $R = 0.1$, and frequency of 5 Hz. The fatigue experiments were conducted at different load values from 30% up to 90% of the average quasi-static failure load of the SLJ specimens. The quasi-static failure load was calculated as the average of the maximum force reached by three specimens tested at a displacement rate of 1 mm/s. The specimen geometry (ISO 9664:1993 [30]) was a SLJ (Fig. 3) with a bondline thickness of 1 mm and an overlap of 25 mm.

The servo-hydraulic MTS 810 testing machine was used. Tests were performed in ambient laboratory conditions. During testing, thermocouples were placed at various points on the surfaces of the specimens in order to investigate any thermal (heat up) effects; however, no change in temperature was observed. The fatigue experiments were conducted up to failure or to a maximum of 1 million load cycles.

3. RESULTS AND DISCUSSION

3.1. TAST

Typical shear stress-strain curves for the Sikaflex 552 and AS1805 RTV adhesive tested at room temperature are shown in Fig. 5. From the shear stress-strain curve, the shear modulus and shear strength were calculated. In general, elastomeric materials exhibit non–linear stress–strain behavior and the definition of the modulus is very difficult. However, at small shear strains they obey Hooke's law and the modulus can be found. The values for shear modulus were calculated from the tangent to the shear stress-strain curve at the origin (a polynomial approximation of the curve was made).

The shear modulus, shear strength, and strain data for Sikaflex 552 and AS1805 RTV adhesives are presented in Table 1.

Typical failure modes of adhesives in TAST specimens are presented in Fig. 6. The failure was mainly cohesive. In some cases the failure was close to the interface (especially for Sikaflex 552) but after close inspection, it was evident that a thin layer of adhesive remained on the substrate.

3.2. SLJ Tests

3.2.1. Effect of Surface Treatment

The surface treatment is a parameter that can significantly affect the joint strength. Anyway, the results (average lap shear strength for grit blasted SLJs was 1.25 MPa, while for chemical etching AS1805 SLJs was 1.23 MPa) show no significant difference between the two surface treatment methods. However, because chemical surface treatment is expensive and toxic waste is generated, grit-blasting is by far the most

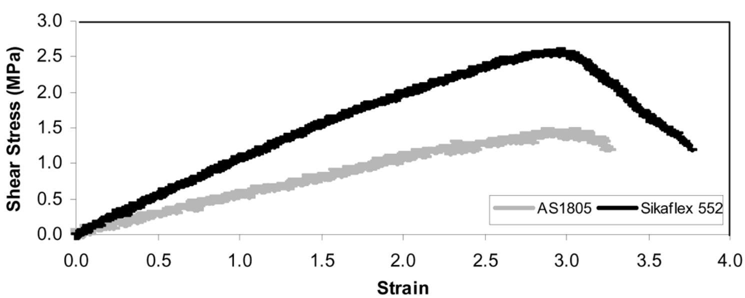


FIGURE 5 Typical TAST shear stress-strain curves of Sikaflex 552 and AS1805 RTV adhesives tested at room temperature.

TABLE 1 Shear Modulus and Strength Data of Sikaflex 552 and AS1805 Adhesives Obtained from the TAST

Adhesive	Shear modulus G (MPa)	Shear strength (MPa)	Shear strain (%)
Sikaflex 552	1.30 ± 0.12	2.39 ± 0.18	330 ± 16
AS1805	0.68 ± 0.03	1.47 ± 0.02	332 ± 17

practical method of the two, so it was chosen to be applied in all of the specimens used for testing in this work.

Failure surface for both surface treatments of AS1805 RTV adhesive joints can be seen in Fig. 7. The failure mode was mainly cohesive, with an apparent (visual inspection) adhesive failure at the interface in some areas, for both treatments. Therefore, it was concluded that the surface treatment has no influence on the failure mode of joints with this type of adhesive.

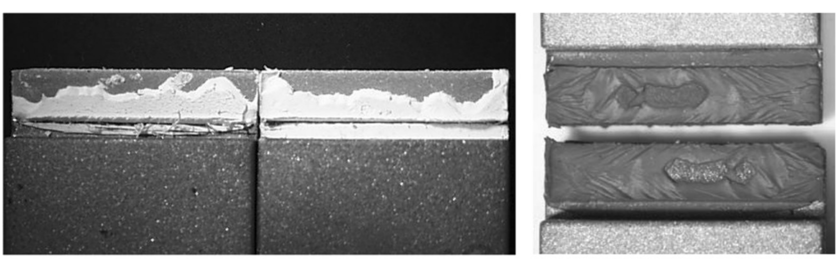
3.2.2. Effect of Temperature

Sikaflex 552 adhesive. The effect of temperature on the Sikaflex 552 lap shear joint strength was examined by testing SLJs (Fig. 3) with 0.2 mm adhesive thickness and 25 mm overlap. A summary of maximum load and average lap shear strength for Sikaflex 552 SLJs tested at room temperature (RT), −40 and 80°C is presented in Table 2. The average lap-shear strength (τ_{av}) is given by:

$$\tau_{av} = P/bL, \tag{1}$$

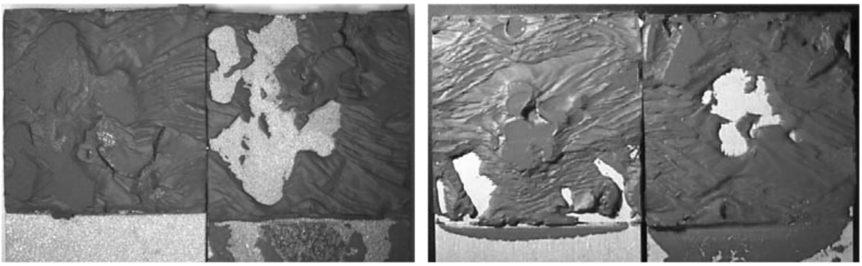
where P is the maximum load, b is the joint width, and L is the joint overlap length.

Representative load-displacement curves of Sikaflex 552 SLJs as a function of temperature are presented in Fig. 8. The steel adherends'



(a) (b)

FIGURE 6 Failure mode in TAST specimens: (a) Sikaflex 552 and (b) AS1805 RTV adhesive.



(a) (b)

FIGURE 7 Failure mode of AS1805 RTV adhesive SLJs with aluminium adherends as a function of surface treatment: (a) Grit blasted and (b) Chemical etching.

deformation is negligible in comparison with that of the adhesive. The nonlinear behavior observed from load-displacement curves is due to the adhesive deformation. The adhesive deformation to failure decreased with increasing temperature. Also, the overall stiffness of the SLJs varies with temperature, the joints being stiffer at −40°C than at RT.

With an increase of temperature, a slight decrease of the lap shear strength occurs because of the decrease in adhesive strength. The lap shear strength of the adhesive joints tested at 80°C is approximately 20% less than that of the specimens tested at room temperature. Data obtained from tests at −40°C showed an increase of the lap shear strength of the adhesive by approximately 115%, approximately twice as high as SLJs tested at RT. This is explained by the fact that polyurethane adhesives have low glass transition temperatures ($T_g = -60°C$ for Sikaflex 552, data provided by supplier). They remain ductile and their strength increases at low temperatures which leads to a higher joint strength.

AS1805 RTV adhesive. Representative load-displacement curves of AS1805 RTV adhesive SLJs (1 mm adhesive thickness) as a function of

TABLE 2 Maximum Load and Average Lap Shear Strength for Sikaflex 552 SLJs Tested at RT, −40°C, and 80°C

Temperature (°C)	Maximum load (kN)	Average lap shear strength (MPa)
RT	2.02 ± 0.19	3.23 ± 0.31
−40	4.36 ± 0.04	6.98 ± 0.06
80	1.58 ± 0.09	2.54 ± 0.15

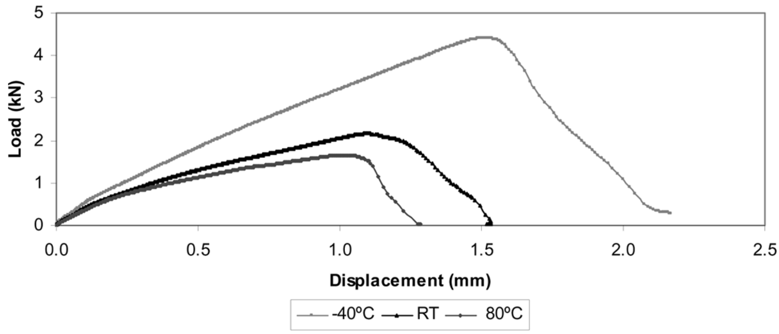


FIGURE 8 Representative Sikaflex 552 SLJ load-displacement curves as a function of temperature.

temperature are presented in Fig. 9. Nonlinear behavior of the adhesive can be observed. The displacement to failure decreased with increasing temperature. The AS1805 SLJs stiffness does not substantially vary with temperature until 200°C.

A summary of maximum loads and average lap shear strengths for AS1805 RTV adhesive SLJs tested at RT, 100, and 200°C is presented in Table 3.

Despite the fact that silicone systems can withstand exposure to temperatures of 200°C for long hours without degradation [31], the lap shear strength of the AS1805 RTV adhesive is affected by variation of temperature. The failure loads of the adhesive joints tested at 100°C fell to about one-half that at room temperature. Also, the failure loads of joints tested at 200°C decreased by approximately 66%. After visual examination, the adhesive seems not to physically degrade at 300°C

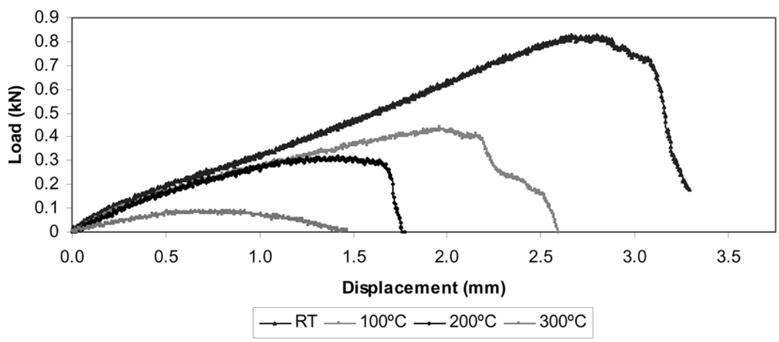


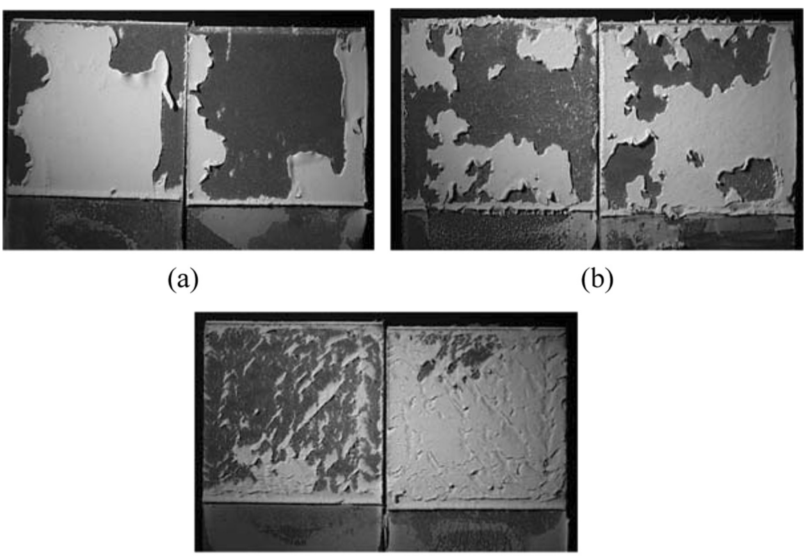
FIGURE 9 Representative load-displacement curves of AS1805 RTV adhesive SLJs as a function of temperature.

TABLE 3 Maximum Load and Average Lap Shear Strength for AS1805 RTV Adhesive SLJs as a Function of Temperature

Temperature (°C)	Maximum load (N)	Average lap shear strength (MPa)
RT	782.8 ± 51.81	1.25 ± 0.08
100	413.7 ± 22.22	0.66 ± 0.03
200	264.4 ± 73.11	0.42 ± 0.11
300	103.3 ± 16.54	0.16 ± 0.02

(maximum working temperature of the adhesive) but a dramatic fall in lap shear strength was observed.

Failure modes. After the tests, the failure modes of the specimens were evaluated visually. For Sikaflex 552 SLJs, the failure was cohesive within the adhesive in all cases, as can be seen in Fig. 10. Some areas of the failure surfaces shown in Fig. 10 seem to have failed adhesively, but a close inspection revealed a thin layer of adhesive on the substrate. The appearance of the failure bond surfaces varied with temperature. The failure surfaces



(a) (b) (c)

FIGURE 10 Failure mode of Sikaflex 552 SLJs tested at (a) −40°C; (b) RT and (c) 80°C.

at −40°C (Fig. 10a) show little adhesive deformation, indicating that the adhesive becomes less ductile, while the failure surface of the adhesives tested at 80°C (Fig. 10c) shows an increase of adhesive deformation which is a sign of more ductility.

The failure was a cohesive/adhesive mixed mode failure for AS1805 RTV adhesive (Fig. 11). Ridges can be observed in the fracture surface, which are oriented perpendicular to the loading direction. Pascal *et al.* [32] investigated the simple shear behavior of a rubber–like adhesive. They observed noticeable ridges that appeared on the fracture surface of the specimens, perpendicular to the loading direction. Using finite element analysis, they also found that the principal stresses acting in the rubber–like adhesive were practically oriented along the shearing direction. The appearance of the failure bond surfaces varies with temperature, as can be seen in Fig. 11. At 200°C, the drawings (ridges) from the failure surface disappeared. The failure surfaces at 200 and 300°C (Fig. 11b and c) suggest that the adhesive becomes less ductile at high temperature.

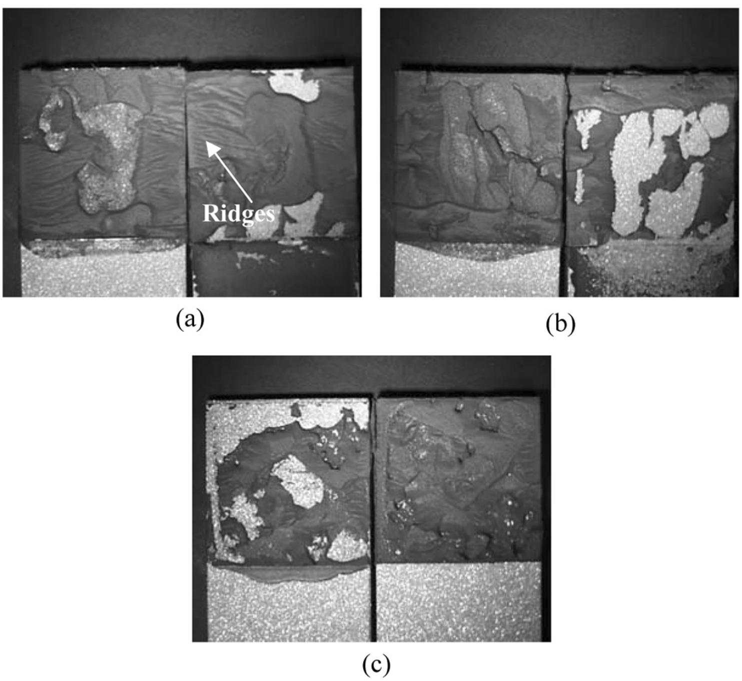


FIGURE 11 Failure mode of AS1805 RTV adhesive SLJs tested at (a) 100°C; (b) 200°C; and (c) 300°C.

3.2.3. Effect of Overlap Length and Bondline Thickness

The effect of the overlap length on the joint performance was examined by testing SLJs with 12.5, 25, and 50 mm overlap length. Typical Sikaflex 552 SLJ load displacement curves as a function of overlap length can be seen in Fig. 12. It can be seen that the slope of the curves increases as the overlap increases, which suggest that the joint is becoming more rigid.

Average failure loads and displacements for Sikaflex 552 SLJs as a function of the overlap length are presented in Fig. 13. As is known, ductile joints develop uniform load transfer over the joint length as compared with joints with brittle adhesives [22]. As expected, the failure loads of Sikaflex 552 SLJs increased almost proportionally with increasing the overlap length. Also, the SLJ's failure displacement increases slightly as the overlap increases, but in much less proportion (7% for 25 mm overlap and 16% for 50 mm overlap, respectively) than failure loads. The stiffness of the SLJs increased approximately proportionally with the overlap length showing an increase of the rigidity of the joints.

Sikaflex 552 SLJ load displacement curves as a function of adhesive thickness can be seen in Fig. 14. It can be noted that the slope decreases as the bondline increases, which means that the joint is becoming more flexible. The joint strength of the adhesive decreases and the failure displacement increases as the bondline gets thicker which is in accordance with the literature [21,33].

The behavior of the RTV adhesive in SLJs with various bondline thicknesses was also of interest. It is known that for structural adhesives, smaller than expected bondline thicknesses may produce insufficient wetting, while thicker than expected bonds may exhibit

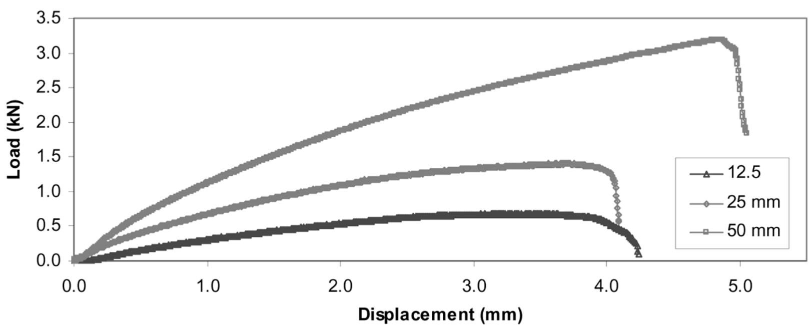


FIGURE 12 Load-displacement curves for Sikaflex 552 SLJs as a function of overlap length.

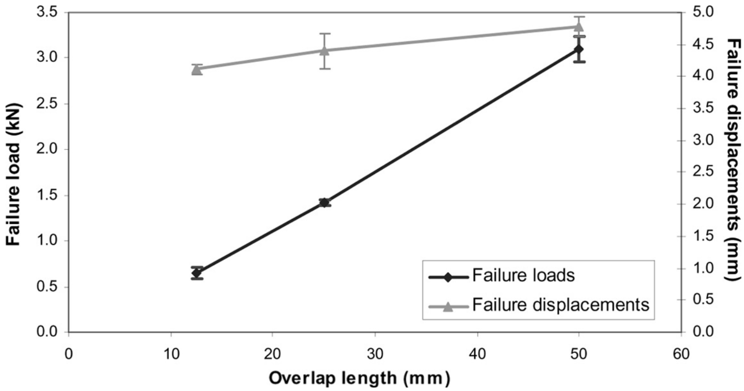


FIGURE 13 Average failure loads and displacements for Sikaflex 552 SLJs as a function of overlap length.

significant defects. Either case can lead to reduced joint performance. For AS1805 RTV adhesive, the optimum bondline thickness to be used in a joint, recommended by the producer, is from 1 to 2 mm. However, it was decided to experiment with a bondline thickness of 0.5 mm. Average failure loads of SLJs with AS1805 RTV adhesive as a function of bondline thicknesses and overlap length can be seen in Fig. 15. As for Sikaflex 552 SLJs, the failure loads of SLJs with AS1805 RTV adhesive increased almost linearly with increasing the overlap length. Figure 15 also shows that the average failure loads of the joint with AS1805 RTV adhesive increases as the bondline gets thicker from 0.5 to 1 mm, although for joints with 12.5 and 50 mm overlaps this trend is not obvious due to high experimental scatter. The failure surface of the joints with 0.5 mm thickness presents a high area of

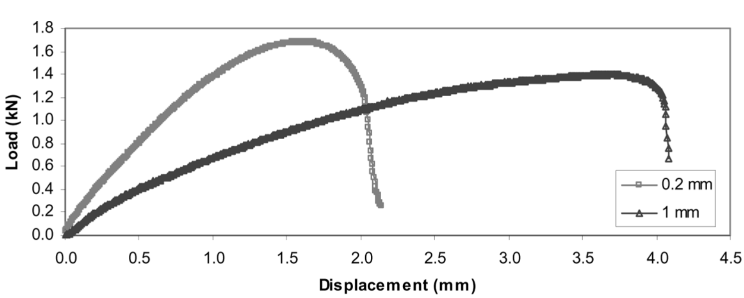


FIGURE 14 Load-displacement curves for the polyurethane adhesive Sikaflex 552 SLJs as a function of bondline thickness.

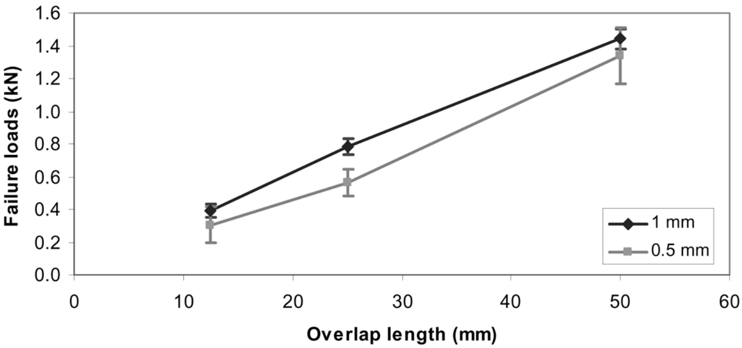
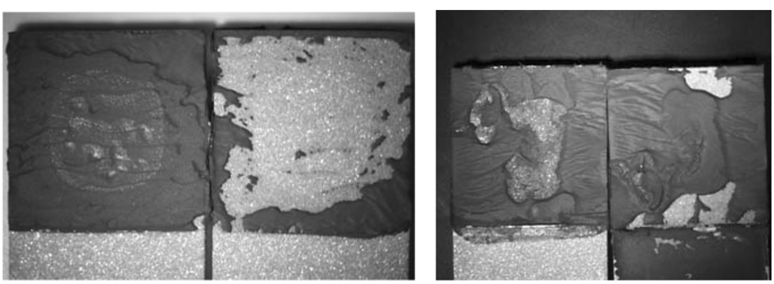


FIGURE 15 Failure loads of AS1805 RTV adhesive SLJs with various bondline thicknesses and overlap length.

adhesive failure (see Fig. 16a). Thus, it can be concluded that for the joints with 0.5 mm thickness, the interfacial (adhesive) strength was less than the cohesive strength of the adhesive, which resulted in adhesive failure. The failure mode, and subsequently the joint strength, changed when SLJs with 1 mm thickness were used. For this case, the failure took place inside the adhesive layer with a very few spots of adhesive failure (Fig. 16b). The explanation is probably not due to a joint mechanics argument as the other flexible adhesive studied has similar bulk properties and gave an increased joint strength for thinner bondlines. This phenomenon might be due to different cure and interfacial chemical reactions as the RTV adhesive thickness varies.

Failure load prediction. As referred to above, Pascal *et al.* [32] showed that flexible adhesives fail by tension, in the shear load



(a) (b)

FIGURE 16 Failure mode of AS1805 RTV adhesive SLJs (25 mm overlap) as a function of bondline thickness: (a) 0.5 mm and (b) 1 mm.

direction, due to the very high adhesive deformation. However, this approach needs a numerical tool and the adhesive tensile strength. In the present work, a simpler approach was used. The failure load of single lap joints can be predicted using the simple design methodology proposed by Adams *et al.* [34], based on the shear stress of the adhesive. The load corresponding to the total plastic deformation of the adhesive (global yielding) is given as

$$P = \tau_y \times b \times L, \tag{2}$$

where P is the failure load of the adhesive, τ_y is the shear yield strength of the adhesive, b is the joint width, and L is the overlap length. In Fig. 17b experimental and predicted failure loads (with values of shear stress obtained from TAST) of the single lap joints are shown. It can be seen that the simple criterion adopted for the joints gives failure loads that compare quite well with the experimental results. For comparison purposes, SLJs with a very brittle and stiff adhesive (Araldite® AV138/HV998, Huntsman, Salt Lake City, UT,

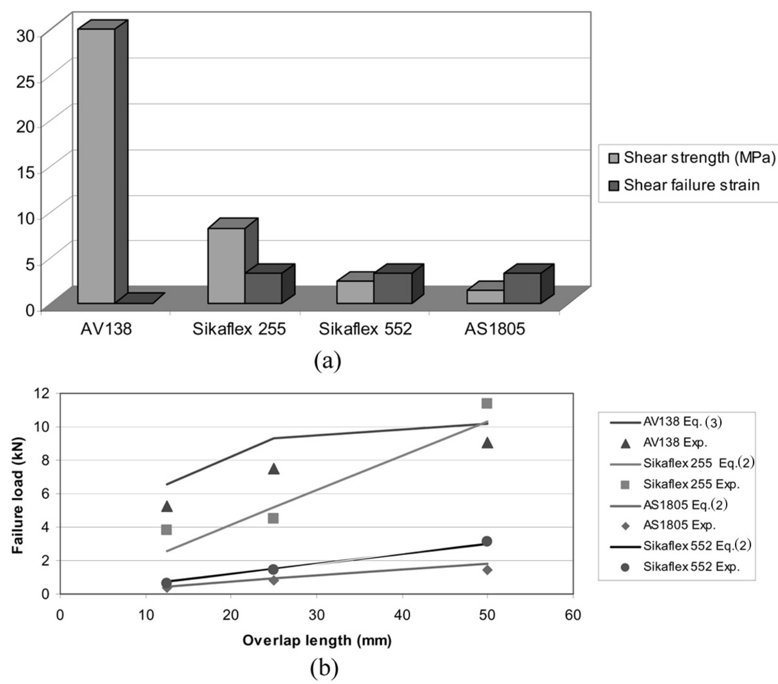


FIGURE 17 (a) Average shear strength and failure strain determined with TAST and (b) Experimental and predicted failure loads of SLJs.

USA) were tested. Also, a slightly stronger polyurethane Sikaflex 255 FC adhesive supplied by Sika (Porto, Portugal) was tested. Average shear strength and strain obtained with TAST for all the adhesives studied are presented in Fig. 17a.

For joints with ductile adhesives, the failure load is given by the load that causes adhesive global yielding along the overlap. This criterion works reasonably well provided the failure shear strain of the adhesive is more than 20%, which is the case for both adhesives used in the present study. However, for brittle adhesives (AV 138), this methodology is not applicable [35]. For joints with a brittle adhesive, Volkersen's model [36] is used and the failure occurs when the maximum shear stress at the ends of the overlap exceeds the shear strength of the adhesive. The following equation was used

$$P = \tau_r \frac{2bl \sinh(\lambda l)}{\lambda l[1 + \cosh(\lambda l)]}, \tag{3}$$

where

$$\lambda^2 = \frac{G}{t_a}\left(\frac{2}{Et_s}\right)$$

t_a is the adhesive thickness, G the adhesive shear modulus, and E the adherend Young's modulus. The Volkersen criterion works particularly well for the brittle adhesive AV138, as expected.

The bonded lap shear joints generate high localized stresses at the joint ends with very little stress carried in the central region of the overlap if a stiff, brittle adhesive is employed. Increasing the overlap length increases the joint strength to a point where a further increase in bond overlap length does not result in an increase in load carrying ability for the AV138 brittle adhesive. From Fig. 17 it can be seen that despite the very low strength (approximately one-third) of the polyurethane Sikaflex 255 adhesive compared with AV138 epoxy adhesive, the strength of the adhesives in a joint is similar for 50 mm overlaps, and the polyurethane adhesive has the advantage of being highly deformable. For overlaps longer than 50 mm, SLJs with Sikaflex 255 are expected to be much stronger than those with the apparently strong epoxy adhesive AV138.

For the joints with flexible adhesives tested in this work (Sikaflex 552 and AS1805), the average lap shear stress in the joints at failure in quasi-static loading is very similar to the measured shear strength with the TAST. (For Sikaflex 552 from TAST, τ_r=2.39 MPa and from SLJs τ_{av} = 2.27 MPa, whereas for AS1805 from TAST τ_r = 1.47 MPa and from SLJs τ_{av} = 1.25 MPa). This indicates that the shear stresses in the joints are essentially uniformly distributed whether a TAST or SLJ specimen is used. In other words, the SLJ can be used to determine the shear strength of flexible adhesives, contrarily to stiff and rigid adhesives like epoxies.

3.3. Fatigue Tests

The parameter combinations used in the AS1805 adhesive fatigue experiments are presented in Table 4. Specimens F1 to F9 were cyclically loaded up to failure. Experiments F10, F11, and F12 were stopped after 1.2×10^6, 1.01×10^6, and 3.1×10^6 cycles, respectively. The specimens that had not failed during fatigue loading were subsequently statically loaded up to failure in the same fashion as the statically tested specimens. The average lap shear strengths were 0.99, 1.04, and 1.28 MPa. This is approximately 80%, 84%, or 102.5% of the initial strength, indicating that very little damage had accumulated within the joint.

Figure 18 shows the resulting fatigue life curve of the AS1805 RTV adhesive SLJs in the typical logarithmic representation. Fatigue data were normalised with respect to the average static failure load, F_0. Load is used rather than stress amplitude because an average shear stress may be misleading, considering the non-uniform nature of the shear stresses and the existence of significant peel stresses, which most likely contribute to failure [37,38].

TABLE 4 Summary of the Experimental Fatigue Program and Results for SLJs with AS1805 RTV Adhesive

Experiment/ specimen	Load range F (N)	Amplitude ratio R	% of ultimate load	No. of cycles up to failure N_f
F01–F03	0–782.8 ± 51.81	–	100	1
F1	72–720	0.1	92	16
F2	55–550	0.1	70	973
F3	48–480	0.1	61	15175
F4	48–480	0.1	61	22500
F5	48–480	0.1	61	7197
F6	40–400	0.1	51	30235
F7	40–400	0.1	51	34549
F8	31–312	0.1	40	144915
F9	31–312	0.1	40	368415
F10	31–312	0.1	40	1236002
	619 statically			Not failed
F11	24–240	0.1	30	1012488
	652 statically			Not failed
F12	24–240	0.1	30	3189037
	801 statically			Not failed

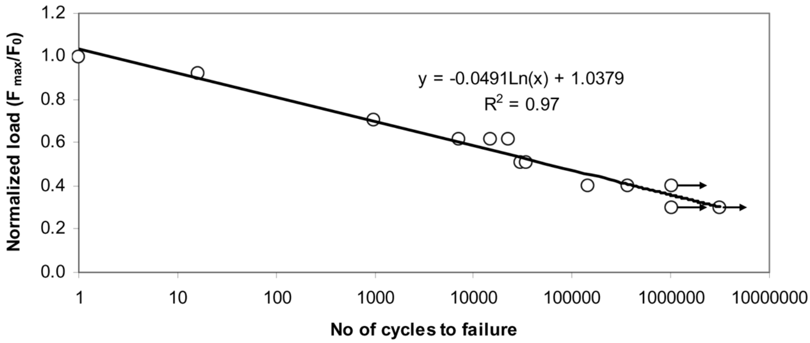


FIGURE 18 Fatigue life curve of AS1805 RTV adhesive SLJs.

A straight line was fitted to the measured values (a logarithmic approximation was made). The equation of the fitted straight line is

$$F_{max}/F_0 = 1.03 - 0.049\ln(N_f), \tag{4}$$

where F_{max} is the maximum load reached at regular intervals by the sinusoidal load applied to the specimen, F_0 is the average static failure load, and N_f is the number of cycles up to failure.

The correlation coefficient of the linear least-squares fit is $R^2 = 0.97$, which indicates a very good fit. This shows that there is a relatively little scatter in the results, indicating that the adhesive is not so sensitive to defects. Generally, the scatter associated with fatigue testing is large [39].

The experiments showed a fatigue limit at about 30% of the static failure load at $R = 0.1$ (the fatigue limit was defined as the highest maximum load at which a specimen could survive 10^6 cycles with no

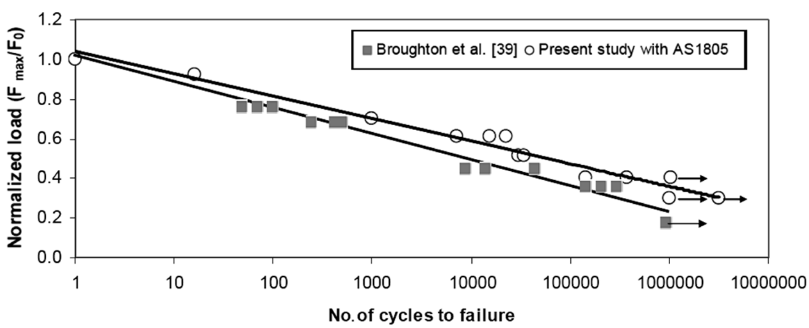


FIGURE 19 Comparison of the fatigue life curves with results from Broughton *et al.* [39].

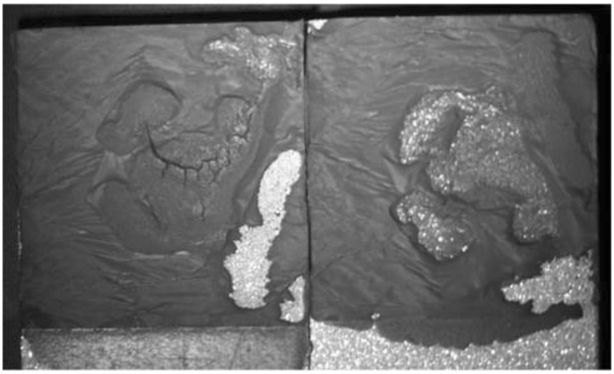


FIGURE 20 Failure mode of fatigue SLJ specimen F7 (failure after 34549 cycles).

visibly apparent damage). Damage initiation and progression before failure could not be detected with the chosen measurement setup (measurement of loads and displacements).

A comparison of the fatigue results with the results from [39], in which SLJs with a brittle and strong epoxy adhesive (AV119 from Huntsman) with steel adherends were tested show that the slope of the fatigue curve of the AS1805 bonded joints is flatter than that of the SLJs with the epoxy adhesive. This result indicates that the epoxy joints are more sensitive to changes in the applied maximum loads (see Fig. 19). The value of the fatigue life curve slope of SLJs with the AV119 epoxy adhesive is 0.09 while for AS1805 RTV adhesive it is 0.049, indicating that SLJs with the epoxy adhesive are more sensitive to fatigue loading than the SLJs with flexible adhesives.

In experiments F1–F9, the specimens failed during the cyclic loading. The fracture surfaces were evaluated visually and the failure modes were identical to the failure modes of the static SLJ experiments (see Fig. 20).

4. CONCLUSIONS

In this paper, two different flexible adhesives, a polyurethane and a RTV adhesive, were studied through adhesive joint tests. The following conclusions can be drawn:

1. SLJs of Sikaflex 552 adhesive were tested at RT, −40, and 80°C. Test results showed that the lap shear strength of the adhesive

is affected by variation of temperature. With increase of temperature, a slight decrease of the lap shear strength occurs because of the decrease in adhesive strength, whereas with decreasing the temperature the Sikaflex 552 SLJs became stronger and stiffer than at RT.

2. SLJs of RTV adhesive were tested at RT, 100, 200, and 300°C. The lap shear strength of the RTV adhesive was affected by variation of temperature. The adhesive did not physically degrade at 300°C (maximum working temperature of the adhesive) but a dramatic fall in lap shear strength was observed.
3. In contrast to joints with brittle adhesives, the joint strength of ductile joints with flexible adhesives increases almost linearly with increasing overlap length.
4. The reduction of failure load with increasing bondline thickness is a very common situation when dealing with structural adhesives. For the low strength flexible adhesive Sikaflex 552 the failure load as well as the overall stiffness of the SLJs decreases as the bondline gets thicker, whereas for AS1805 RTV adhesive the failure loads increase as the bondline gets thicker from 0.5 to 1 mm.
5. The fatigue tests on AS1805 SLJs showed a fatigue limit of approximately 30% of the static failure load. The slope of the fatigue curve of the AS1805 RTV adhesive bonded joints is flatter than that of the SLJs with an epoxy adhesive [39], therefore, indicating that the epoxy joints are more sensitive to changes in the applied maximum loads.

ACKNOWLEDGMENTS

The authors would like to thank the Portuguese Foundation for Science and Technology for supporting the work presented here through the research project PTDC/EME-PME/67022/2006, Sika Portugal for supplying the polyurethane adhesive, and ACC Silicones Ltd. for supplying the RTV adhesive.

REFERENCES

[1] Duncan, B. and Dean, G., *Int. J. Adhes. Adhes.* **23** (2), 141–149 (2003).
[2] Geiss, P. and Vogt, D., *J. Adhes. Sci. Technol.* **19** (15), 1291–1303 (2005).
[3] Petrie, E. M., *Handbook of Adhesives and Sealants*, 2nd ed., (McGraw-Hill, New York, 2007).
[4] da Silva, L. F. M. and Adams, R. D., *J. Adhes. Sci. Technol.* **20** (15), 1705–1726 (2006).
[5] Adams, R. D. and Mallick, V., *J. Adhesion* **43** (1–2), 17–33 (1993).

[6] da Silva, L. F. M. and Adams, R. D., *Int. J. Adhes. Adhes.* **27** (3), 216–226 (2007).
[7] Deb, A., Malvade, I., Biswas, P., and Schroeder, J., *Int. J. Adhes. Adhes.* **28**, 1–15 (2008).
[8] Adams, R. D., Coppendale, J., Mallick, V., and Al-Hamdan, H., *Int. J. Adhes. Adhes.* **12**, 185–190 (1992).
[9] Chiu, W. K., Chalkley, P. D., and Jones, R., *Computers and Structures* **53**, 483–489 (1994).
[10] Kang, S. G., Kim, M. G., and Kim, C. G., *Compos. Struct.* **78** (3), 440–446 (2007).
[11] Owens, J. F. P. and Lee-Sullivan, P., *Int. J. Adhes. Adhes.* **20**, 47–58 (2000).
[12] Cognard, P., *Collage des Composites, Secteurs Routier et Ferroviaire*, (Techniques de l'Ingênieur, Paris, 2003).
[13] Adnan, A. and Sun, C. T., *Journal of Adhesion* **84** (5), 401–420 (2008).
[14] da Silva, L. F. M., Rodrigues, T., Figueiredo, M. A. V., de Moura, M., and Chousal, J. A. G., *Journal of Adhesion* **82** (11), 1091–1115 (2006).
[15] Adams, R. D. and Peppiatt, N. A., *J. Strain Anal.* **9**, 185–196 (1974).
[16] Gleich, D. M., van Tooren, M. J. L., and Beukers, A., *J. Adhesis. Sci. Technol* **15**, 1091–1101 (2001).
[17] Crocombe, A. D., *Int. J. Adhes. Adhes.* **9**, 145–153 (1989).
[18] Grant, L. D. R., Adams, R. D., and da Silva, L. F. M., *Int. J. Adhes. Adhes.*, **29**, 405–413.
[19] Broughton, B. and Gower, M., Preparation and testing of adhesive joints, NPL Measurement Good Practice Guide No. 47 (NPL, Teddington, UK, 2001).
[20] Bryant, R. W., *Nature* **202**, 1087–1088 (1964).
[21] Giannis, S., "The mechanical and physical behaviour of aircraft fuel tank sealants," Ph.D. Thesis, University of Bristol (2005).
[22] de Castro, J. and Keller, T., *Composites Part B: Engineering* **39**(2), 271–281 (2008).
[23] da Silva, L. F. M., Ramos, J. E., Figueiredo, M. V., and Strohaecher, T. R., *Journal of Adhesion and Interface* **7** (4), 1–9 (2006).
[24] Gomatam, R.R. and Sancaktar, E., *J. Adhes. Sci. Technol.* **19** (8), 659–678 (2005).
[25] Erpolat, S., Ashcroft, I. A., Crocombe, A. D., and Abdel-Wahab, M. M., *International Journal of Fatigue* **26** (11), 1189–1196 (2004).
[26] Jones, R., Kotousov, A., and Marshall, I. H., *Fatigue & Fracture of Engineering Materials & Structures* **25** (2), 173–185 (2002).
[27] Abel, M. L., Adams, A. N. N., Kinloch, A. J., Shaw, S. J., and Watts, J. F., *Int. J. Adhes. Adhes.* **26** (1–2), 50–61 (2006).
[28] ISO 11003–2: 1993 (E), Adhesives – Determination of shear behaviour of structural bonds, Part 2: Thick-adherend tensile-test method (1993).
[29] da Silva, L.F.M., Silva, R. A. M. D., Chousal, J. A. G., and Pinto, A. M. G., *J. Adhesion Sci. Technol.* **22** (1), 15–29 (2008).
[30] ISO 9664: 1993, Adhesives – Test methods for fatigue properties of structural adhesives in tensile shear (1993).
[31] Parbhoo, B., in *Handbook of Adhesion*, 2nd ed., D. E. Packham (Ed.) (John Wiley & Sons, Ltd., Chichester, 2005), pp. 469–473.
[32] Pascal, J., Darque-Ceretti, E., Felder, E., and Pouchelon, A., *J. Adhes. Sci. Technol.* **8**, 553–573 (1994).
[33] da Silva, L. F. M., Carbas, R. J. C., Critchlow, G. W., Figueiredo, M. A. V., and Brown, K., *Int. J. Adhes. Adhes.* Submitted (2008).
[34] Adams, R. D., Comyn, J., and Wake, W. C., *Structural Adhesive Joints in Engineering*, 2nd ed., (Chapman & Hall, London, 1997).
[35] da Silva, L. F. M., Critchlow, G. W., and Figueiredo, M. A. V., *J. Adhes. Sci. Technol.* **22** (13), 1477–1494 (2008).

[36] Volkersen, O., *Luftfahrtforschung* **15**, 41–47 (1938).
[37] Abdel Wahab, M. M., Ashcroft, I. A., Crocombe, A. D., and Smith, P. A., *Composites Part A: Applied Science and Manufacturing* **35** (2), 213–222 (2004).
[38] Crocombe, A. D. and Richardson, G., *Int. J. Adhes. Adhes.* **19**, 19–27 (1999).
[39] Broughton, B., Mera, R. D., and Hinopoulos, G., *Cyclic fatigue testing of adhesive joints test method assessment*, Report No. 8, (National Physical Laboratory, Middlesex, UK, 1999).