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Tests for Strength of Adhesion

Summary

The design of adhesively bonded structures requires accurate material properties data. These data are often best obtained from bulk test specimens. Bulk specimen data can give an indication of the cohesive strength of the materials but designers also need to consider the strengths of the interfaces between the adhesive and substrates. Information on interface strengths is normally obtained from adhesives joint tests, such as lap shear or T-peel. However, the complexity of stress distributions in such joints leads to difficulties in obtaining quantitative interface strengths that are applicable to other loading configurations.

As part of a DTI funded Measurements for Materials Systems project, Interfacial Adhesion Strength, a number of alternative test methods for adhesion strength – pull-off, profiled butt joint, pull-out and 3-point bend – have been studied as alternative methods for quantifying adhesion strength. These test methods have been evaluated, in experimental and Finite Element (FE) studies, for their ability to quantify the strength of adhesion between adhesive and adherend.

Most of the tests are superficially easy to perform and interpret using analytical formulations. The results presented indicate that the methods are able to distinguish between 'good' and 'bad' adhesion. However, care must be taken when evaluating results. The failure stresses calculated through analytical methods are often considerably lower than those predicted in the FE stress analyses. The calculated adhesion strengths will be conservative and likely to be far lower than the bulk material strengths. Only in the profiled butt joint test are the analytical average stresses and the FE predicted peak stresses comparable. However, this test is time consuming in both specimen preparation and performance, and also requires special alignment fixtures for bonding and testing specimens.

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Introduction

The design of adhesively bonded structures requires accurate material properties data. These data are often best obtained from bulk test specimens [1]. Bulk specimen data can give an indication of the cohesive strength of the adhesive but designers also need to consider the strengths of the interfaces between the adhesive and substrates. Information on interface strengths is normally obtained from adhesives joint tests, such as lap shear. However, the complexity of stress distributions in such joints leads to difficulties in obtaining quantitative interface strengths that are applicable to other loading configurations. In many of these joint tests, observed failure modes tend to be mixed – both interfacial and cohesive – and it can be difficult to detect the point of first failure.

A review of available test methods for coatings and adhesives [2] found a large number of techniques that are used for characterising adhesion. Many of these are qualitative, assessing adhesion from the appearance of the fracture surface. Some types of joint test such as the tensile butt joint or thick adherend shear can provide good quality engineering data on adhesives but the stress concentrations near the joint ends make interpretation of local failure stress and strain values a problem.

Adhesives

Two structural epoxy adhesives formulated for the automotive industry were used in these studies:

- Adhesive A - a single-part, heat cured toughened epoxy with high impact resistance. Bulk tensile strength is approximately 58 MPa and bulk shear strength is approximately 31 MPa.
- Adhesive B - a two part toughened epoxy that is formulated to be tolerant of oiled surfaces. 5027 cures at room temperature but is formulated to undergo a high temperature post cure, which is claimed to improve the adhesion strength. Bulk tensile strength is approximately 22 MPa and bulk shear strength is approximately 13 MPa.

Tensile Pull-Off and Butt Joint Tests

The pull off test is used widely to test the adhesion of coatings to substrates and is also used to assess adhesives. This test is attractive as it is quick and simple to perform, requires low cost equipment and produces a quantified measure of the adhesive strength from the maximum force applied to the sample.

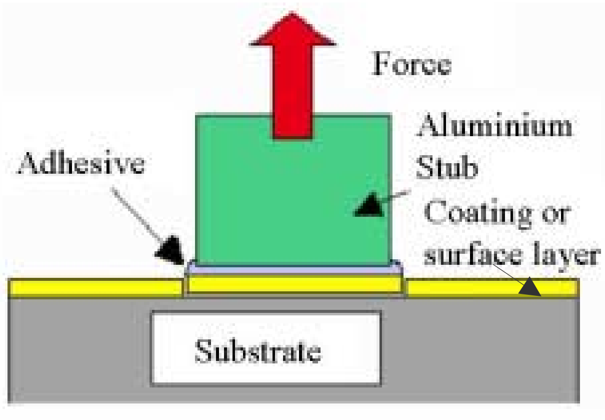

Figure 1: Pull-Off Test

The main features of the test are shown in Figure 1, the coating indicated could be a surface oxide layer on a metal adherend. An aluminium test specimen is shown in Figure 2. It is critical in using the test that failure does not occur at the interface between the aluminium stub and the adhesive. The sample shown in Figure 2 failed cohesively in the adhesive indicating that the adhesion strength exceeded the cohesive strength of the adhesive. The grit blasted and solvent cleaned surfaces of the aluminium stub provide a good surface for bonding and the high rigidity of the pull stub relative to the thinner adherend biases the failure away from the stub.

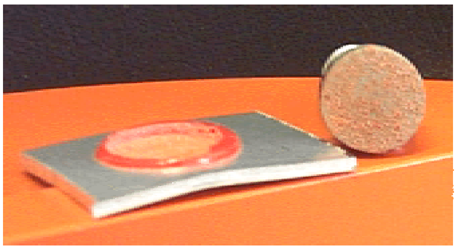

Figure 2: Pull-Off Specimen

The pull-off test was used to assess the strength of bonding to a number of surfaces and the results shown in Table 1 indicate a reasonable degree of repeatability amongst the measurements.

Table 1: Pull-Off Test Results

Surface and Treatment	Pull-Off Strength (MPa)
Adhesive A and Aluminium	
no treatment	10.7 ± 0.8
light clean	13.0 ± 0.5
light clean + anodise	14.0 ± 0.5
full clean	14.8 ± 0.4
full clean + anodise	14.3 ± 0.7
full clean + over anodise	13.3 ± 1.3
Adhesive B and Galvanised Steel	
no treatment	13.8 ± 0.9
light oil	5.0 ± 0.9
heavy oil	3.4 ± 1.1

The results show reasonable distinction between good and bad surfaces for bonding. In the case of good surfaces the failure tended to be cohesive in the adhesive with much greater levels of adhesion failure seen with the poorer surfaces. At no time was failure observed at the interface between the pull stub and the adhesive.

It is interesting to note that, in this test, the ultimate failure strengths of the two Adhesives A and B appear similar, around 14 MPa. However, bulk specimen tests indicate tensile strengths of approximately 58 MPa and 22 MPa, respectively. These results suggest that the pull-off test produces substantial stress concentrations within the adhesive layer. As Figure 2 indicates, there is substantial deformation of the adherend sample in the test that would lead to high cleavage stresses. The simplicity of the test and specimen preparation means that there is little control over alignment of the specimen.

The more tightly controlled tensile butt joint produced strength values that were much closer to the bulk strengths of each of the adhesives. The alignment of the butt joint specimen is closely controlled during manufacture using a precision manufactured assembly jig, Figure 3. Prior to testing the load train of the test machine is carefully adjusted using an alignment fixture and a dummy test specimen until all bending forces are eliminated. The sample is then clamped in rigid collets to prevent slippage.

This test arrangement, although time consuming for sample preparation and testing, produces very reliable results. The method was used to evaluate models for plastic yield functions of adhesives under high hydrostatic stress levels, generated in the adhesive layer. FE predictions showed a stress concentration near the outer rim of the adhesive layer, even when the edges were profiled. The size of the stress concentration peak varied with the materials model chosen. This made it difficult to use the test to evaluate failure criteria. Some more extensive profiling options were investigated with FEA. The most promising profile was when the butted adherend ends were interlocking concave and convex hemispheres with 50 mm radii of curvature – producing a bowl shaped adhesive layer.

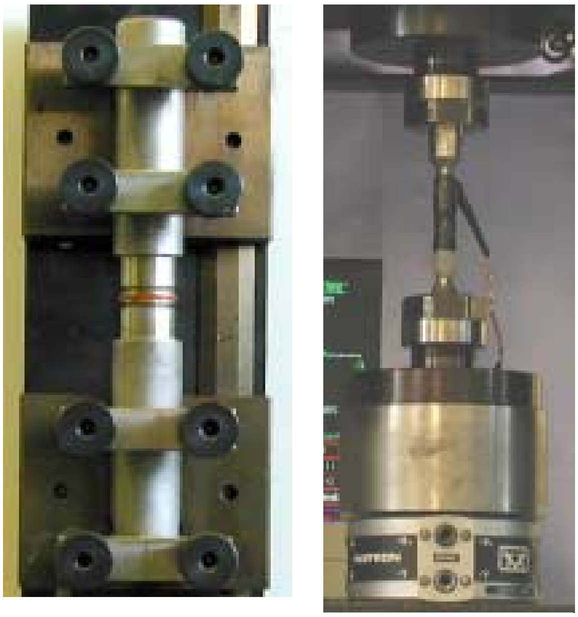

Figure 3: Butt Joint Sample and Test Alignment

Figure 4 shows tensile stress distributions in the profiled butt joint specimen. It is noticeable that the region of highest stress extends from the centre of the specimen over the majority of the adhesive layer. Stress values near the rim are lower. The highest stress predicted is only a few percent greater than the average stress (calculated from the force divided by the bonded area). The modified butt joint test specimen appears to offer a method for determining reliable values for stress at failure from measured loads.

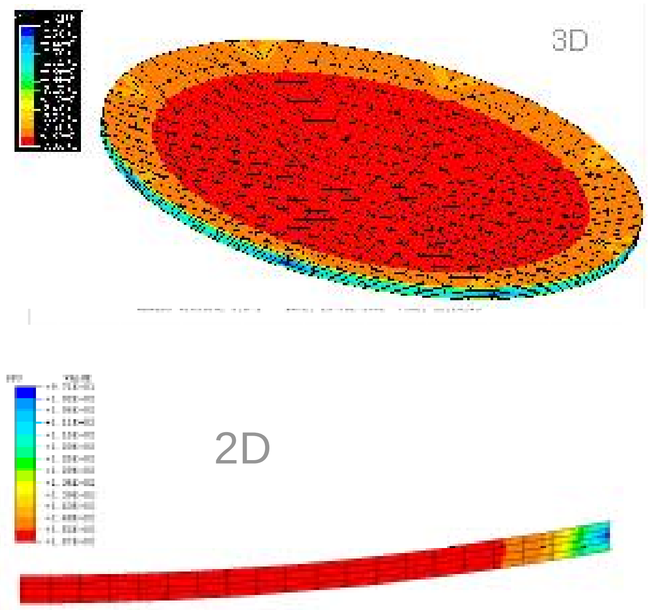

Figure 4: Stress distribution in modified butt joint

Test data for this modified specimen bonded with Adhesive A are shown together with data for unmodified specimens in Figure 5. There is little difference between the maximum levels of load achieved in each type of specimen as the maximum load is limited by the plastic yield of this tough adhesive. Average failure stresses determined by this test were 53 MPa and 28 MPa for Adhesive A and Adhesive B, respectively, close to the bulk tensile strengths.

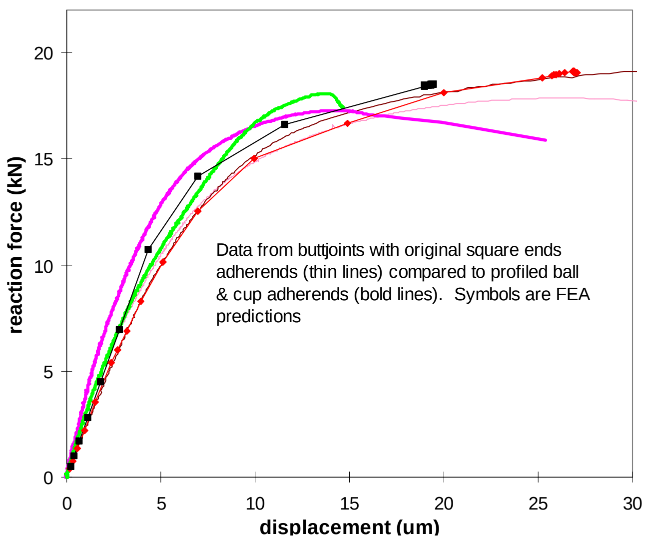

Figure 5: Butt joint test results

Tensile Pull Out Test

In composites technology the adhesion of the fibres to the resin is a key determinant of the strength of the system. Tests were developed to assess this adhesion from the force required to pull embedded fibres from the resin. Analytical routines, based on elastic shear lag analyses, were developed to calculate stresses along the interfaces [3].

At first glance, this test seems promising for adhesives. The specimen is relatively easy to prepare and testing is straightforward with a simple fixture. The largest stresses are at the interface, biasing the test towards interfacial failure and this point is easily identified from the sharp fall in load.

The adherend samples must be thin, as a sufficient thickness of adhesive is required around the embedded sample. As the thickness of the adhesive increases, preparation of the adhesive block becomes more difficult. A sample suitable for adherends up to 2 mm thick is shown in Figure 6.

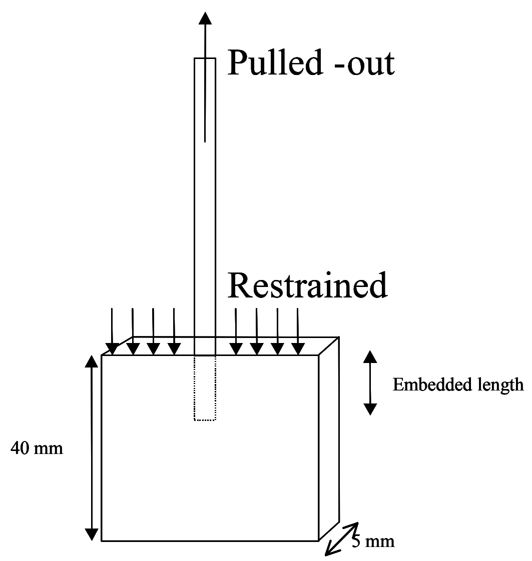

Figure 6: Pull-out specimen

Specimens were prepared from different 1 mm thick shim material (including steel, aluminium and brass) and the two epoxy adhesives. Both of these adhesives bond well to the metals and in tests with an original embedded length of 15 mm many of the adherends broke before adhesion failure occurred. Therefore, test specimens were prepared with shorter embedded lengths (typically

3 or 5 mm) to reduce tensile forces on the adherend. These shorter lengths increase relative uncertainties in the result. In general there is a fairly high degree of scatter in pull out test results.

Figure 7 shows a typical result for Adhesive A. It is obvious that the force-extension response is dominated by the shim although the failure of the interface is clear from the dramatic drop in load. The results show that the behaviour of the adherend is clearly inelastic at the point of interface failure.

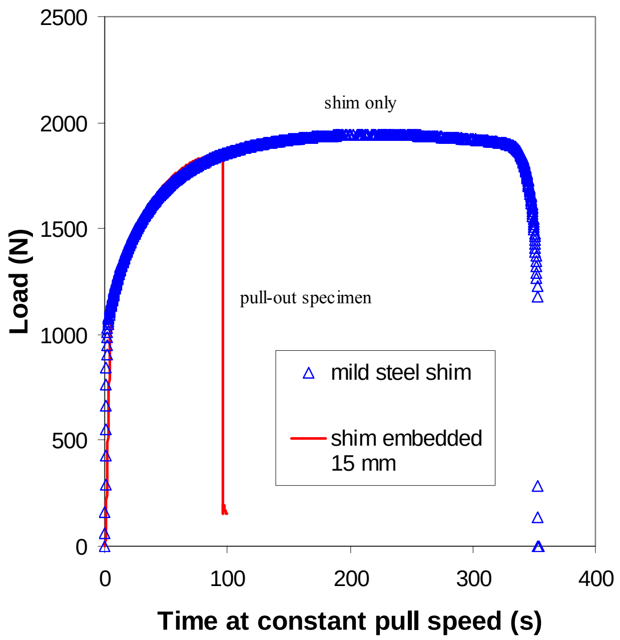

Figure 7: Pull-out test result

Shear lag calculations of interfacial shear stress (IFSS) values were made using the following formula:

$$\tau_i = n\frac{F}{wh}\frac{\cosh[n(L_e - x)/r]}{2\sinh(nL_e/r)},$$

$$n^2 = \frac{E_a}{E_s(1+\nu_a)\ln\left(\frac{R}{r}\right)}$$

where L_e is embedded length, E tensile modulus, ν Poisson's ratio, x distance along shim, w the shim width and h the shim thickness. R and r are equivalent radius values for the matrix and shim respectively. The subscripts s and a represent shim and adhesive, respectively.

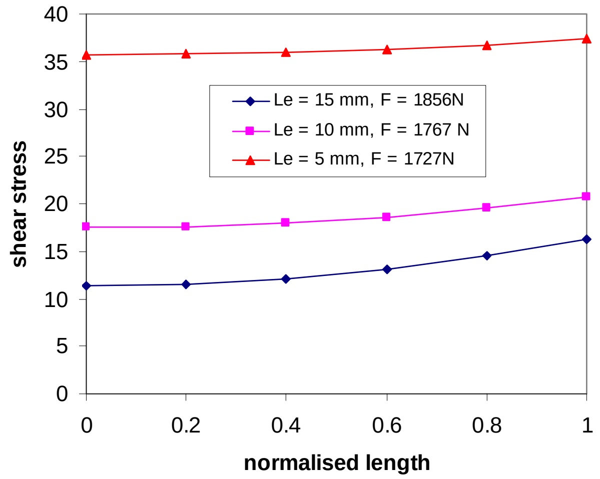

Figure 8: IFSS values calculated for different embedded lengths

Although the inelastic behaviour of the shim likely invalidates the elastic assumptions of the model, stress calculations were made for various embedded lengths of 5x1 mm cross-section mild steel adherends in Adhesive A. The average loads at failure for 5, 10 and 15 mm embedded lengths were 1726, 1767 and 1856 N respectively – far from proportionate to the embedded length as would be expected if average shear stress was the failure criteria. The IFSS values calculated at the failure loads are shown in Figure 8.

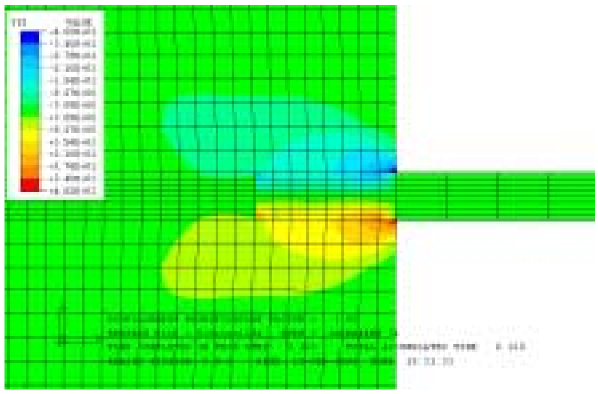

Figure 9: Shear stress in pull-out specimen

Quite clearly, shear stress at the interface does not seem to correlate with failure. An FE prediction of stress, such as Figure 9, shows intense stress concentrations near the exit point of the shim. Stress near the exit of the adherend is approximately three times the stress along the centre of the adherend, a higher concentration factor than predicted analytically.

Adhesive B specimens were tested with two different cure conditions - with only a room temperature cure or after a high temperature post-cure. The results indicated a dramatic increase in adhesion strength following post-cure. The post-cured specimens were so well bonded that the samples failed either in the adherend or in the adhesive block below the end of the shim.

The extensive inelastic deformation of the adherend and the resulting complexity of the interfacial stress distribution lead to problems in the interpretation of results from this test.

Adhesion by Three Point Bend

Bend tests are common techniques for assessing adhesion of coatings and paints to surfaces. The four-point bend test is often used for thin, brittle coatings. Failure of coatings can be detected by visual inspection or acoustic emission. There is a three-point bend test for adhesives ISO 14679 [4].

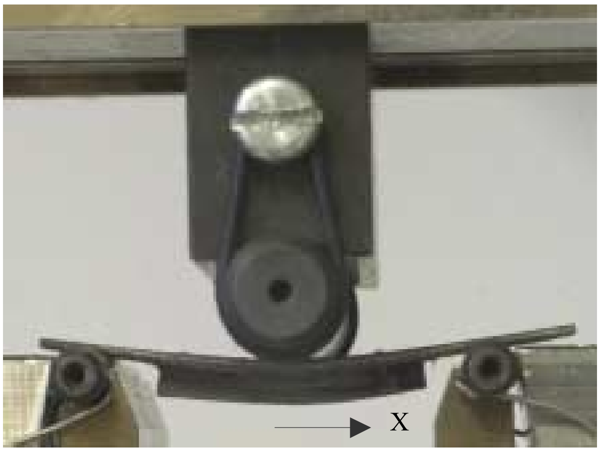

Figure 10: 3 Point Bend Test

ISO 14679 samples were prepared by applying a 25 mm by 5 mm by 3.8 mm thick ‘rib’ of adhesive to the underside of a thin rectangular adherend (50 mm by 10 mm). Moulds prepared from silicone rubber enable control of the position and shape of the rib. The samples are then cured according to normal cure schedules. It may not be possible to test all types of adhesives using this method, as the adhesive layer in this test is relatively thick. It is virtually impossible to make representative samples this thick with certain types of adhesive, for example anaerobics. Furthermore, if the adhesive cures rapidly large temperature rises may occur in the sample, damaging the material.

The samples are tested in a standard three point bend arrangement with supporting rollers at a 33 mm separation, as shown in Figure 10. The crosshead is driven at a constant speed and the test concludes when failure occurs. The adhesive rib imparts considerable stiffening to the beam and a rapid drop in load accompanies the start of failure in the adhesive layer. The technique is suitable for mass screenings as tests can be performed very rapidly. The ‘open’ nature of the adhesive block provides the potential for accelerated ageing of the interface in chemical environments.

Acoustic emission techniques were used in an attempt to identify the point of first failure. However, the signal picked up noise from the friction between the supports and the sample that made identification of the point of failure difficult. What measurements were made tended to confirm that the drop in load coincides with the initiation of a crack.

Analytical calculations [5] have been developed for the shear stress τ at the interface based on the bending moments and elastic properties of the beam and rib.

$$\tau = \frac{FE_2 b(h - h_1)}{2b_1(G_2 - \frac{G_1^2}{G_0})}\left[\frac{h+h_1}{2} - \frac{G_1}{G_0}\right]$$

$$G_0 = E_1 b_1 h_1 + E_2 b(h - h_1)$$

$$G_1 = \frac{E_1 b_1 h_1^2}{2} + \frac{E_2 b(h^2 - h_1^2)}{2}$$

$$G_2 = \frac{E_1 b_1 h_1^3}{3} + \frac{E_2 b(h^3 - h_1^3)}{3}$$

The subscripts 1 and 2 refer to the rib and adherend, respectively. F is the force, b width, h depth and E is tensile modulus. This formulation predicts shear stress at the interface in the centre of the beam. This seems unrealistic, as stress is likely to vary along the x-direction (defined in Figure 10).

The test was used to evaluate bonding between the adhesives and the six aluminium surface treatments described earlier. The post cured Adhesive B proved to be too flexible and too well bonded to initiate failure at the interface – failure occurred at the centre of the adhesive rib, on the underside. For the Adhesive A and room

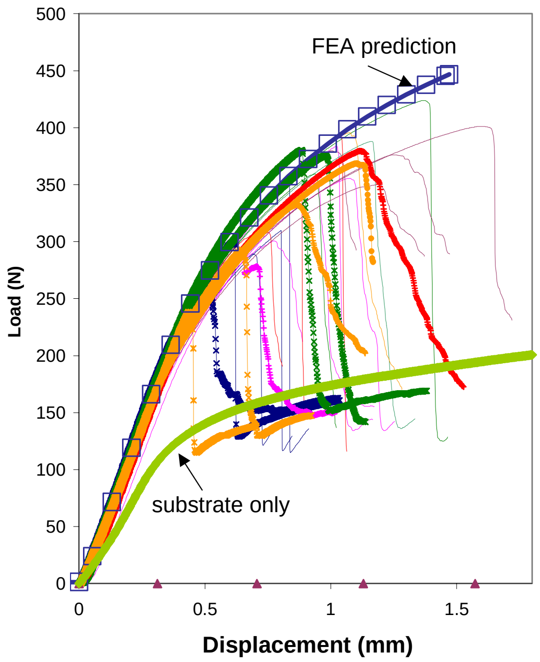

Figure 11: 3 Point bend results for Adhesive A-aluminium samples

temperature cured Adhesive B failure occurred, as desired, at the end of the rib at the interface between the beam and the rib.

A modification to the test, bonding a stiffener strip to the outer edge of the adhesive rib was considered in order to increase the peel stresses at the end on the rib and thus bias failure towards the interface. A series of tests were performed using a 2.9 mm thick rib of Adhesive B and a 0.9 mm thick steel stiffener. Failure was observed to initiate at the aluminium- rib interface as desired. The calculation for shear stress was modified to account for the presence of the stiffener.

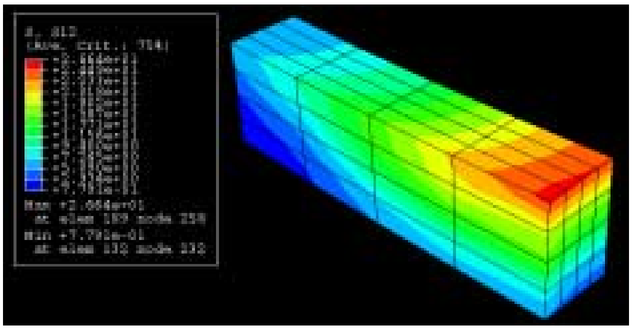

Figure 12: Shear stress distribution in the adhesive rib. The top of the rib is at the interface between the adhesive and the adherend.

Results for basic 3-point bend tests on the Adhesive A bonded to aluminium adherends are shown in Figure 11. Comparison with the results for the substrate only shows the stiffening effect of the adhesive rib. The shear stresses at failure that were calculated for various test systems are shown in Table 2. The mean failure stress values, around 5 MPa for Adhesive A and 8 MPa for Adhesive B, seem low. Finite element predictions were made for the test systems and the predicted force-extension curve shown in Figure 11 (symbols) is in good agreement with the experimental measurements.

The FE predicted shear stress distribution in the Adhesive A specimen near failure is shown in Figure 12. The maximum stress at the interface was 27 MPa, far greater than the analytical value. The minimum interface stress is approximately 5 MPa at the centre of the rib, consistent with the analytical formulation. The modified, stiffened specimen shows less of a variation in shear stress along the interface but peel stresses are increased substantially. The test results show similar patterns to the pull-off test results – i.e. able to distinguish poor surfaces from good surfaces but not differences between the good surfaces.

Table 2: Bend Test Results

Surface	Shear Stress (MPa)
Adhesive A and Aluminium	
no treatment	4.3 ± 0.4
light clean	4.9 ± 0.6
light clean + anodise	4.9 ± 1.0
full clean	5.8 ± 0.4
full clean + anodise	5.3 ± 0.4
full clean + over anodise	4.8 ± 0.8
Adhesive B and Aluminium (+ backing strip)	
no treatment	6.5 ± 0.3
light clean	7.7 ± 0.3
light clean + anodise	7.5 ± 1.2
full clean	8.1 ± 0.4
full clean + anodise	8.0 ± 0.3
full clean + over anodise	8.4 ± 0.1

Concluding remarks

Pull and bend test methods have been evaluated for their ability to quantify the strength of adhesion between adhesive and adherend. Both experimental and FE studies have been undertaken. Most of the tests are superficially easy to perform and interpret using analytical formulations. The results presented indicate that the methods are able to distinguish between ‘good’ and ‘bad’ adhesion but insensitive to small differences. However, care must be taken when evaluating results. The failure stresses calculated through analytical methods are often considerably lower than those predicted in the FE stress predictions. The calculated adhesion strengths will be conservative and likely to be far lower than the bulk material strengths.

Only in the profiled butt joint test are the analytical average stresses and the FE predicted peak stresses comparable. However, this test is time consuming in both specimen preparation and performance, and requires special alignment fixtures for bonding and testing specimens.

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