MTS Adhesives Project 2: 
Failure Modes and Criteria

Report 3 Annex 5

Failure Modes in Adhesively Bonded Joints

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Abstract

A video technique has been used to assess the failure modes in two types of adhesively bonded joints. Thick adherend shear (TAST) and T-peel tests have been performed at three temperatures (-20°C, room temperature and 80°C) and the locus of failure and the crack paths monitored continuously. As the temperature increases failure becomes increasingly ductile and stress whitening is more apparent in the adhesive. At the highest test temperature failure was predominantly adhesive whereas cohesive failure becomes increasingly important as the test temperature is reduced.
1. Introduction

Failure modes in adhesively bonded joints are known to depend on the loading geometry, the adhesive type and the test temperature. In this study two different joint geometries have been investigated, thick adherend shear tests (TAST) where adherend deformation should play a minor role, and T-peel where adherend deformation is more significant, and tests have been performed at three temperatures -20°C, room temperature (22°C) and 80°C. Failure modes were monitored using a video recording system and confirmed with post facto examination.

This report was compiled as part of the activities undertaken within the MTS Adhesives Project 2 "Failure Modes and Criteria" and summarises the results of the experimental work undertaken.

2. Experimental Procedure

Joints were made using two adhesives widely used in industry representing a high stiffness and a compliant resin system:-

1) AV119 (Ciba Polymers, Duxford) a one-part toughened epoxide which cures at 120°C

2) F241 (Permabond Adhesives Ltd, Eastleigh), a compliant room temperature curing toughened acrylic.

Two bonded joint geometries were used based on thick adherend shear (TAST) and 180° T-peel tests and the test pieces used are shown in Figures 1 and 2. Surfaces to be bonded were grit blasted and treated with a primer (Permabond SIP) prior to joint assembly in purpose-built jigs. Tests were performed on a bench top Instron frame fitted with a calibrated load cell. Continuous monitoring of the test pieces was achieved using a modified zoom microscope stage fitted with a video camera and monitor; each test was recorded on video tape for subsequent analysis (Figure 3). In order to maximise the depth of field the aperture of the microscope was closed to its minimum setting and extra illumination was applied with lamps to improve visibility. Magnifications of up to 100x are achievable by this technique. Even so it is necessary that the edge of the joint is very flat and the adhesive and adherends are coplanar - this can be
best achieved by polishing but this introduces damage into the bond which influences subsequent behaviour. For this reason good bonds were selected from a larger number of possible samples to achieve the required visibility. Three bonds were tested for each joint type/test temperature/adhesive combination and the results averaged. There was considerable scatter in the results which is probably exacerbated by the need to select joints for good visibility.

Temperatures were controlled by the use of a liquid nitrogen cold finger and an industrial hot air blower working in isolation or in combination. By this method it was possible to control temperatures to ± 2°C during the test. The cold finger was held in contact with the rear face of the TAST and T-peel samples and thermally linked with a layer of heat sink compound. Temperatures were allowed to equilibrate for ten minutes prior to testing. The sample test temperature was monitored with a chromel/alumel thermocouple in contact with the front surface and was confirmed with regular checks using a hand-held spot contact thermocouple device on the other faces.

At -20°C test temperature some icing of the sample occurred prior to testing. This could be prevented by the use of the hot air blower at a low temperature rating. However, the resultant clouds of fog obscured the clarity of the video images and caused unacceptable fluctuations in temperature in a few cases.

Post facto examination was carried out with a low power reflected light microscope to confirm the failure modes and estimate the percentage of adhesive/cohesive failure. A good correlation between the results of the video assessment and post facto analysis was maintained throughout.

3. Results and Discussions

3.1 Failure Loads

For both adhesives it was possible to generate results using the same T-peel sample test geometry. Failure loads are summarised in Table 1. For the F241 tests there is a decrease in failure load as the test temperature increases whereas for the AV119 the failure load is constant above room temperature but appears to be reduced at lower temperatures. However, the scatter is large at this temperature and testing a second batch of samples at room temperature produced similar low T-peel strengths so this may be an effect of increased scatter
at the lower test temperatures or ageing affecting the joint strength (see later). The shapes of the load/time curves are shown schematically in Figure 4. In all cases there is an increase to an initial peak which occurs at the onset of cracking. At low test temperatures failure occurs quickly after this in a brittle fashion whereas at room temperature and above some slower crack propagation can occur at a lower test load. This is accompanied by a progressive rounding-off of the initial load peak and a transition to a more ductile failure mode.

TAST samples in the original specimen geometry proved highly variable for the F241 and a second set were produced with a much thicker adherend (10mm as opposed to 2mm). For both AV119 and F241 the TAST strength is reduced as the temperature increases which agrees well with previous results. In most cases fracture is catastrophic with a crack propagating completely across the adhesive in a fraction of a second at failure. However, at 80°C test temperature a small amount of residual bond strength remains, particularly for the F241 adhesive. The failure mode becomes increasingly ductile as the temperature increases.

The large scatter in the test results at a given temperature is probably due to the quality of the bonded samples rather than due to any problems with the test procedure. The need to select flat samples with visible glue lines may well have artificially introduced variability into the test since these samples do not always give the strongest bonds probably due to the presence of edge defects. Variations in surface preparation and the time delay before testing are also important.

3.2 Observations of Failure

3.2.1 AV119 peel

For the reduced temperature tests (-20°C) the initial failure mode was brittle adhesive failure followed by cohesive failure ranging from 10 to 60% of the original bond length. The crack started at the surface of the fillet and propagated rapidly to the interface (Figure 5a). The initial failure was followed by two or three secondary crack propagation events with visible crack arrests after testing. Failure was cohesive in this region. Regions of the failed adhesive had pits and bubbles visible and appeared to be porous. No signs of stress whitening were observed during the test or on video replay.
For tests at ambient temperature the failure was more ductile. In this case the initial failure was cohesive in the bulk of the fillet changing to adhesive failure as the crack propagation progressed and then back to cohesive at the end of the test (Figure 5b). The majority of the adhesive (90 to 95%) remained on one side of the failed joint. No obvious signs of stress whitening were observed during the test but some evidence is available from video images (Figure 6).

For the tests at 80°C the failure mode was much more ductile and the rate of crack propagation was reduced. Initial failure in the fillet was adhesive with cracks propagating to the surface and along the interface (Figure 5c). This was followed by a short region of cohesive failure (10-20%) and then a long region of adhesive failure (Figure 7). Some crack arrests were visible and again the adhesive appeared to be porous or to have undergone some pull-out. Stress whitening was clearly visible during the test.

3.2.2 AV119 TAST

For tests at low temperature the failure mode was brittle and predominantly adhesive (~90% / Figure 6a). Metallic flakes appeared on the failed surface of the adhesive implying that at least some crack propagation occurred through the metal. No stress whitening was observed during the test.

At ambient temperature the failure was less brittle but still predominantly adhesive. More than 95% of the adhesive remained on one half of the joint (Figure 8b). Again metallic particles were observed on 2 to 5% of the surface of the adhesive. No signs of stress whitening were observed.

At 80°C the failure mode was more ductile than at lower temperatures but still adhesive with 95% of the failed adhesive remaining on one half of the joint (Figure 8c). Yet again metallic flakes were visible on the surface of this adhesive. Stress whitening was observed on the video replay of one test.

3.2.3 AV119 general observations

In all tests crack propagation was very rapid with a crack running to arrest in less than the time between single video frames. The preponderance of interfacial failure during the test is apparent - crack propagation along the interface can occur through the tops of the highest metal asperities leading to some metal
transfer onto the failed adhesive surface. These may have been left weak after
the grit blasting process. Crack initiation in the TAST test occurs at the
adhesive/adherend interface reliably for all tests. For the T-peel test failure is
initially cohesive in the fillet but changes to be adhesive as the crack propagates
along the T.

As the temperature increases the increased ductility of the adhesive leads to
some reduction in stress concentrations and a very slight reduction in crack
propagation velocity. However, the failure mode becomes predominantly
adhesive as the temperature increases.

3.2.4 F241 peel

The more flexible F241 adhesive leads to much slower crack propagation and
more ductile failure. For tests at -20°C the failure was cohesive (Figure 9a) and
some stress whitening of the adhesive in the fillet region occurred which
remained visible after testing. As the temperature increases the crack propagates
closer to one of the adherends, but in all cases a complete film of adhesive
remained on each half of the joint after failure. This can be easily identified by
the use of a coating thickness measurement gauge based on magnetic induction
(e.g. a Fisher Permascope).

Tests at ambient temperature showed similar behaviour with a long period of
slow crack growth prior to joint failure. Again failure was 100% cohesive
(Figure 7b) and some stress whitening of the fillet region was visible after testing.

At 80°C the failure was more ductile with the failure load falling off to about 50%
of its initial value and a slow crack propagation to final failure. The failure was
again cohesive (Figure 9c) with stress whitening clearly visible at the crack tip
during testing (Figure 10) and on the two halves of the joint after testing.

3.2.5 F241 TAST

For both reduced and ambient temperature tests the failure was brittle and the
joint failed in a catastrophic single crack propagation. Failure was mixed
adhesive/cohesive with no obvious trend for a given test temperature
(Figure 11). Some stress whitening of the adhesive in the fillet region was
observed after testing.
At 80°C a similar pattern of failure was observed although no stress whitening was visible after testing.

3.2.6 F241 general observations

Unlike the AV119 the flexible F241 adhesive shows cohesive failure behaviour in the T-peel test with well defined stress whitening at the crack tip becoming increasingly common as the temperature increases. However, in the TAST test adhesive failure is far more common. In the T-peel test crack initiation occurs in or close to the fillet region - in many cases interfacial failure and cohesive cracking occur at the same time independently and crack bridging between the two only occurs later in the test (Figure 12). For the TAST test failure occurs so rapidly that it is difficult to determine where initiation occurs. A crack runs through about 60% of the bond area allowing the adherends to move apart to accommodate the strain. Adhesive bridges are then pulled apart giving the joint some residual strength.

4. Discussion

Video imaging technique can give some useful information on failure modes provided that the edges of the adherends and adhesive on the surface to be viewed are well aligned to prevent problems with depth-of-field. Furthermore the illumination needs to be adjusted to maximise visibility which can lead to slight heating of the bond if care is not taken. Both cracking and stress whitening are apparent in the test and are often more visible on subsequent examination of the video.

The strengths of the AV119 bonds tested here are broadly comparable with previous work using the same adhesive. Brittle cohesive failure at low temperatures has been observed followed by more adhesive ductile failure as the temperature increases previously which is similar to what has been reported here. However, a somewhat greater tendency for interfacial failure was observed in this study. This may well reflect the time between the making of the joints and their testing which amounted to several months. Indeed the second batch of AV119 T-peel samples which were tested soon after joint manufacture showed much higher strengths and an increased likelihood of cohesive failure. It thus
appears that pretreatment and joint ageing cannot be ignored in any complete study of failure modes.

5. Conclusions

A video microscopy in combination with a bench-top mechanical testing machine can be used to determine the locus of failure in adhesively bonded joints. However, following crack trajectories is often difficult if the speed of crack propagation is faster than the conventional video sampling rate (50 frames/second). For the T-peel test geometry crack nucleation and propagation is much easier to assess as failure is not so rapid. The speed of failure is reduced and the usefulness of the test increases as the test temperature increases.

The failure modes of adhesively bonded joints depend on surface preparation and the time before testing as well as on the loading configuration and the test temperature. Interfacial failure becomes increasingly common as joints age, even if no evidence for bondline corrosion is immediately apparent. Interfacial porosity, localised variations in wettability and surface defects, such as friable regions introduced by grit blasting, become increasingly important as the joint area is reduced. This leads to variability in joint performance and failure modes.
<table>
<thead>
<tr>
<th>Test Temperature</th>
<th>TAST Failure load (N)</th>
<th>T-Peel Failure load (N)</th>
<th>TAST Failure Load (N)</th>
<th>T-Peel Failure Load (N)</th>
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<tr>
<td>-20°C</td>
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<td>153±73</td>
<td>2567±603</td>
<td>160±87</td>
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<tr>
<td>+2°C</td>
<td>265±187</td>
<td>153±33</td>
<td>1490±276</td>
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<td>22°C</td>
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<tr>
<td>±2°C</td>
<td>167±69</td>
<td>258±20</td>
<td>550±89</td>
<td>103±25</td>
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</tbody>
</table>

**Geometry**

- Glueline thickness: 1mm 0.12mm 1mm 0.12mm
- Sample width: 2mm 4mm 10mm 4mm

* batch 1: Tested some time after manufacture
  batch 2: Tested immediately
FIGURE CAPTIONS

Figure 1: T-peel Joint Geometry

Figure 2: Thick Adherend Shear Test (TAST) Geometry

Figure 3: Schematic of mechanical test arrangements

Figure 4: Schematic load/displacement curves for the adhesive /joint/ temperature combinations tested

Figure 5: Typical failure path for AV119 peel tests (a) -20°C, (b) 22°C and (c) 80°C

Figure 6: AV119 T-peel at increased temperature (80°C) showing (A) fillet before test (B) stress whitening during test (C) crack propagation and interfacial separation

Figure 7: AV119 T-peel at ambient temperature showing (A) fillet before test (B) crack initiation and branched crack path (C,D) stress whitening at tip of propagating crack

Figure 8: Typical failure paths for AV119 TASTs (a) -20°C, (b) 22°C and (c) 80°C

Figure 9: Typical failure paths for F241 TASTs (a) -20°C, (b) 22°C and (c) 80°C

Figure 10: F241 T-peel at increased temperature (80°C) showing (A,B) fillet before test and (C,D) stress whitening during test

Figure 11: Typical failure paths for F241 TAST samples

Figure 12: Initiation stages of F241 T-peel tests
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