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**Creep Testing of Adhesive Joints
T-Peel Test**

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Creep Testing of Adhesive Joints Environmental Effects

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ABSTRACT

The T-(or 180°) peel test is the most widely used method by industry for determining the relative peel resistance of adhesive bonds between flexible adherends. This test geometry has been adopted by most standards bodies and is widely used by industry to evaluate environmental durability of adhesively bonded systems. The simplicity and low costs associated with specimen manufacture, testing and data analysis has contributed to the widespread use of this method for assessing environmental and creep resistance. A series of tests have been conducted on bonded T-peel joints exposed simultaneously to heat, humidity and load.

This report evaluates the test method for producing in-situ peel strength data on adhesively bonded joints. The test method is evaluated in terms of fitness for purpose in assessing environmental performance and provides a guide to specimen geometry, manufacture and testing. The report also includes an experimental evaluation of a self-stress (spring) loading fixture used for creep testing in hot/humid environments. A number of tools have been employed in data interpretation. These include finite element analysis, statistical techniques, analytical modelling, fractographic analysis, dynamic mechanical thermal analysis and mechanical testing.

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Approved on behalf of Managing Director, NPL, by Dr C Lea,
Head of Centre for Materials Measurement and Technology.

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1. INTRODUCTION

Since the inception of structural bonding, peel tests have been an integral part of the adhesive performance specifications, and have played an important role in the development of adhesives. The emphasis has been on the development of higher peel strengths, often at the expense of environmental resistance. Peel resistance being defined as the average force per unit test specimen width, measured along the bond line that is required to separate progressively two adherend members of a bonded joint. The incessant demand for higher peel stresses has provided an impetus for the development of suitable peel tests. These test methods are normally used to compare adhesives where the peel force is a measure of fracture energy. The data generated has little use in the stress analysis of a bonded joint.

The T- (or 180°) peel test is the most widely used method for determining the relative peel resistance of adhesive joints manufactured from flexible metallic adherends (e.g. thin steel or aluminium alloy sheet). The term flexible refers to the ability of the adherend to bend through 90° without breaking or cracking. The simplicity and low costs associated with specimen manufacture, testing and data analysis has contributed to the widespread use of this method for assessing environmental and fatigue resistance. This test method has been adopted by most standards bodies and is widely used by industry to evaluate environmental durability of adhesively bonded systems [1-4].

This report evaluates the T-peel test in terms of fitness for purpose in assessing environmental performance and provides guidance on specimen geometry, manufacture and testing. Finite element analysis (FEA) has been used to determine joint stiffness and stress distributions within the joint. The evaluation includes data from T-peel joints that have been exposed simultaneously to heat, humidity and load. **Throughout this report, statements of particular importance or relevance are highlighted in bold type.**

The research discussed in this report forms part of the Engineering Industries Directorate of the United Kingdom Department of Trade and Industry project on “Performance of Adhesive Joints - Combined Cyclic Loading and Hostile Environments”, which aims to develop and validate test methods and environmental conditioning procedures that can be used to measure parameters required for long-term performance predictions. This project is one of three technical projects forming the programme on “Performance of Adhesive Joints - A Programme in Support of Test Methods”.

2 T-PEEL TEST

This section discusses the basic test geometry used for generating peel strength data and provides a number of recommendations based on experimental results and numerical analysis.

2.1 SPECIMEN GEOMETRY

The T-peel test is suited for use with metal adherends, but other flexible adherends (e.g. fibre-reinforced plastics) may also be used. Specimens are typically 25 mm wide, have a

minimum bonded length of 150 mm, and 50 mm long arms [2]. The recommended thickness is 0.5 mm for steel and 0.7 mm for aluminium. Adhesive layer thickness is not specified. The force is applied to the unbonded ends of the specimen. The angle between the bond line and the direction of the applied force is not fixed. Figure 1 shows a schematic of the standard T-peel geometry. Variations of this test method are included in both national and international standards [1-3].

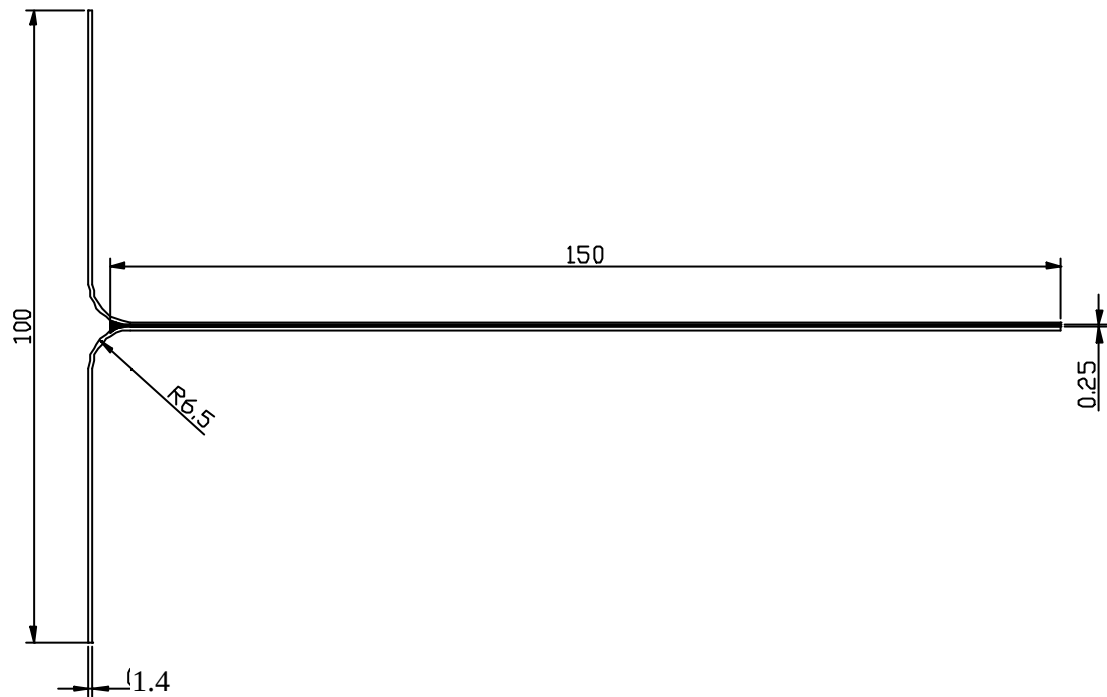


Figure 1 Schematic of standard T-peel specimen dimensions (mm).

The dimensions of the standard test geometry shown in Figure 1 were incompatible with self-stressing fixtures used for testing T-peel specimens exposed to combined heat, humidity and static loads. Figure 2 shows the dimensions for a smaller (miniature) T-peel specimen used in conjunction with self-stress (spring) loading fixtures for measuring life expectancy under static loading conditions. Specimen width was 25 mm.

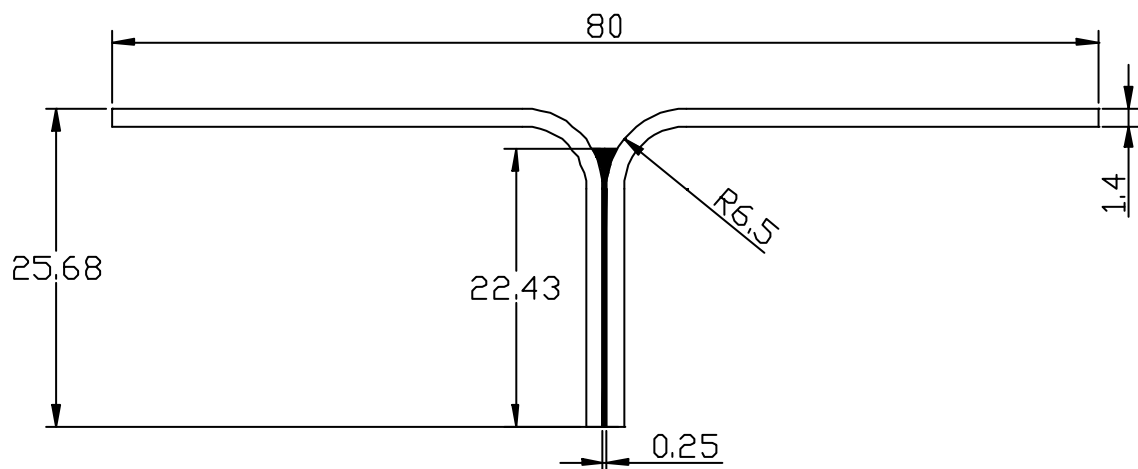


Figure 2 T-peel test geometry dimensions (mm) used for creep tests.

2.2 SPECIMEN PREPARATION

Test specimens of the dimensions shown in Figure 2 were prepared individually from pre-shaped adherends. Rectangular sections of CR1 mild cold rolled steel (supplied by British Steel) were milled to the required length and width and then bent into a right angle. The right angled sections were bonded with AV119 (Araldite® 2007) adhesive supplied by Ciba Speciality Chemicals. Comparative tests were carried out on both the miniature and standard test geometries.

The larger specimens had a bond length of 100 mm rather than 150 mm. Both specimen geometries had a 6.5 mm external radius **R₀** and a 50% adhesive fillet. The definition of fillet size is shown in Figure 3. **The fillet size was controlled using a special tool shaped to fit within the bonded joint.** A 50% adhesive fillet (Figures 4 and 5) was selected following consultation with members of the Industrial Advisory Group (IAG). Previous work [5] has shown that the **fillet size is the most important parameter controlling T-peel static strength.** As the fillet size increases, the strength of the joint also increases.

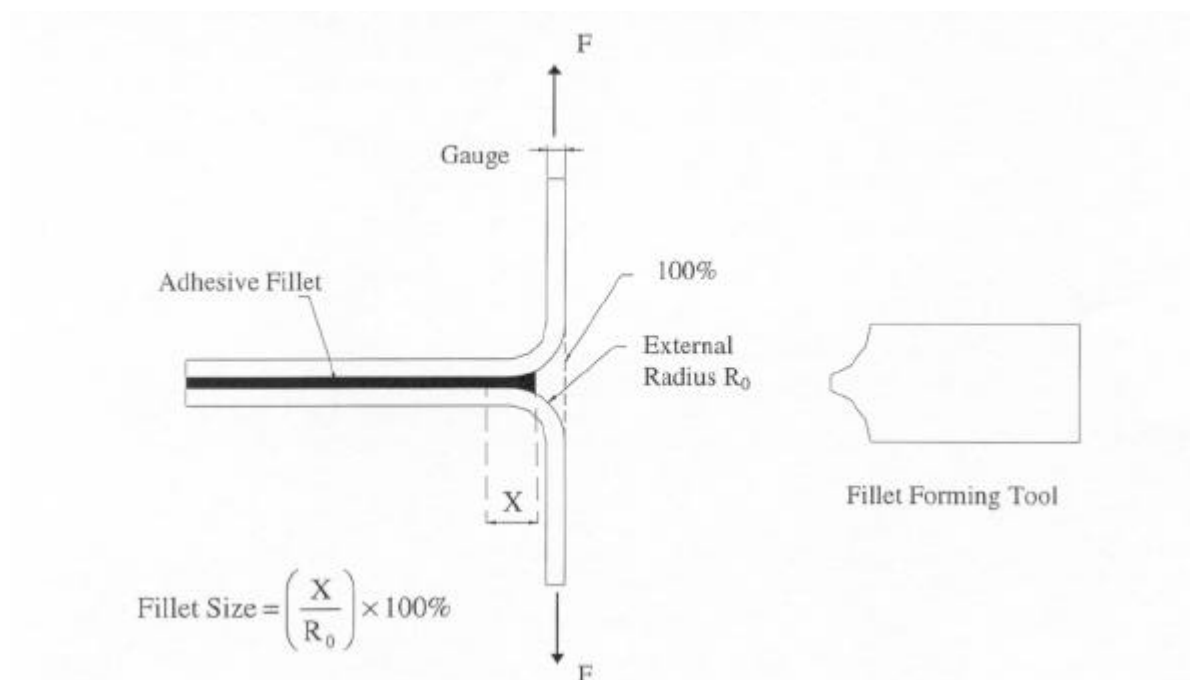


Figure 3 Definition of fillet size in T-peel joint [5] - including fillet forming tool.

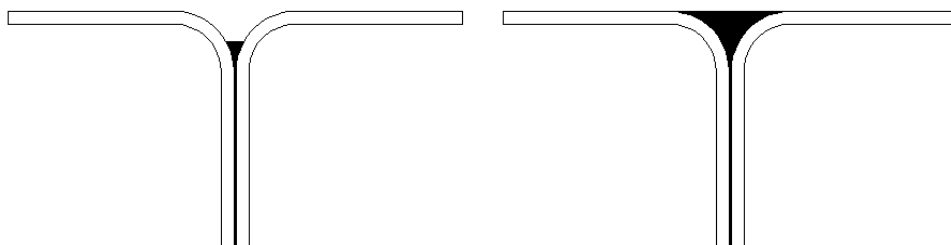


Figure 4 Schematic of the T-peel joint with (left) 50%; and (right) 100% adhesive fillet.

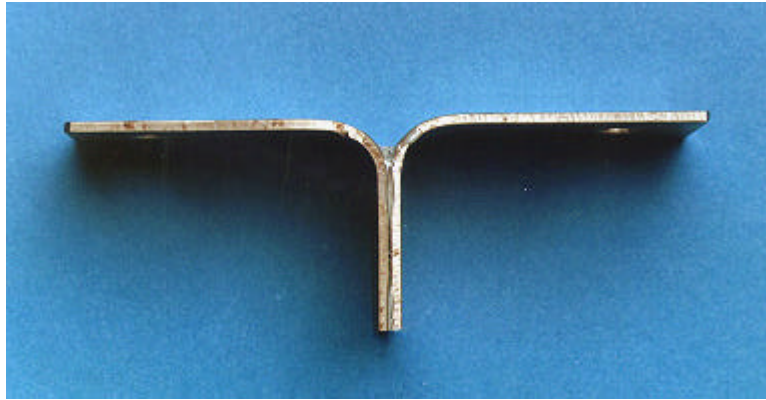


Figure 5 T-peel specimen.

Specimen misalignment can also detrimentally influence the T-peel static strength, although the effect is only small for the bondline thickness (i.e. 0.25 mm) used in the test programme. This effect is more apparent with increasing bondline thickness [5].

Prior to bonding, the adherends were degreased with 1,1,1-trichloroethane and then grit blasted using 80/120 alumina. The surfaces to be bonded were then degreased again with 1,1,1-trichloroethane. The bondline thickness (0.25 mm) was controlled using 250 μm ballontini glass spheres. Ballontini glass spheres may cause premature failure. A small quantity of the glass spheres, 1% by weight, was mixed into the adhesive. Specimens were clamped in a special bonding jig and then heated to 140°C for 75 minutes to cure the adhesive.

Note. The use of 1,1,1 tricholorethane is not recommended. It has only been used in this test programme to maintain continuity with previous work carried out within the DTI funded ADH programme.

2.3 QUASI-STATIC TESTING AND DATA ANALYSIS

Testing was straightforward requiring no special fixture. Tensile tests were carried out at a displacement rate of 100 mm/min on both standard and non-standard geometries (Table 1) under standard laboratory conditions (23°C/50% relative humidity) to ISO 11339 [2] specifications. An Instron 8501 servo-hydraulic test frame was used to load the specimens. The specimens were held by a pair of well aligned servo-hydraulic operated wedge-action grips with a lateral pressure of 100 psi. Instron Series IX software was used to control the test machine and to collect the test data. Five specimens per condition were tested.

It is important to ensure that the bonded portion of the specimen remains perpendicular to the applied load. Self-aligning grips are required to hold the specimen with each set of jaws of the grip firmly engaging the outer 25 mm of the unbonded ends of the flexible adherend. **The unbonded ends need to be accurately aligned between the grips to ensure a uniform tensile load is applied evenly across the specimen width.** The specimen is bent backwards and peeled. Adherend thickness (i.e. 1.4 mm) was sufficient to minimise deformation (i.e. stretching and bending) prior to detachment.

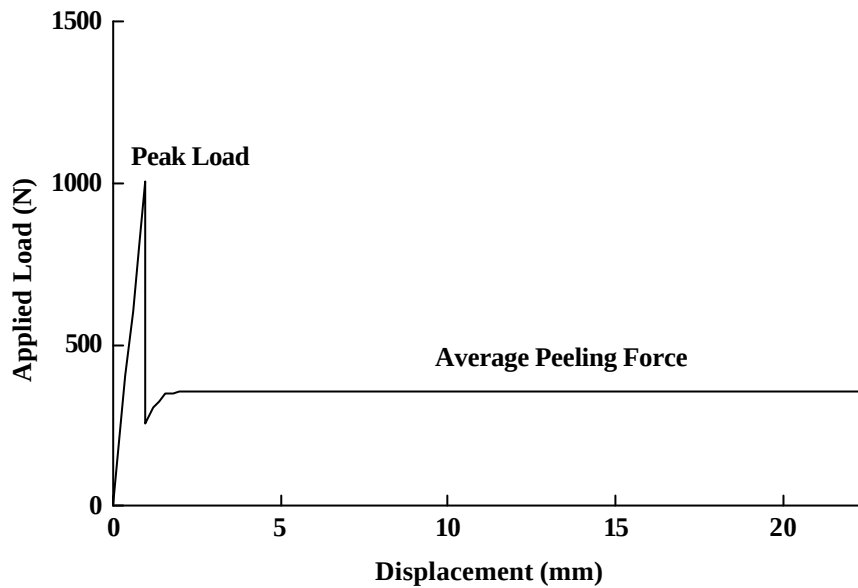


Figure 6 Schematic of load-displacement curve for T-peel tests.

Peel strength is the force per unit width necessary to initiate failure and/or maintain a specified rate of failure by means of a stress applied in a peeling mode [2]. Two loads were recorded for each test: (i) static strength (peak load/force to initiate failure); and (ii) average peeling force (Figure 6). The peel strength data (Table 1) show that the static strength (peak load) for standard and the miniature (i.e. creep) test geometries were significantly different. The load to initiate failure and the average peeling force were far higher for the smaller specimen. This may be attributed to higher and more uniform clamping forces being applied to the smaller specimen during cure. Average peel force decreases with increasing crack length, and hence there may not be sufficient bond length to achieve a reliable average peel force for the smaller specimens.

Table 1 Comparison of Failure Loads (N) for Standard and Miniature T-Peel Specimens

Test Geometry	Static Strength	Average Peeling Force
Standard (100 mm bond length)	1374 ± 104	103 ± 18
Miniature (22.43 mm bond length)	1873 ± 123	368 ± 47

2.4 FINITE ELEMENT ANALYSIS

This section briefly discusses the results of finite element modelling of the T-peel joint geometry and summarises some of the main points from the analysis [6]. The finite element models were constructed and solved using the ABAQUS [7] program. Mesh generation was performed using the FEMGV [8] pre-processor. A full description of the numerical analysis and results for T-joint geometry are presented in the NPL Report CMMT(A) 207 [6].

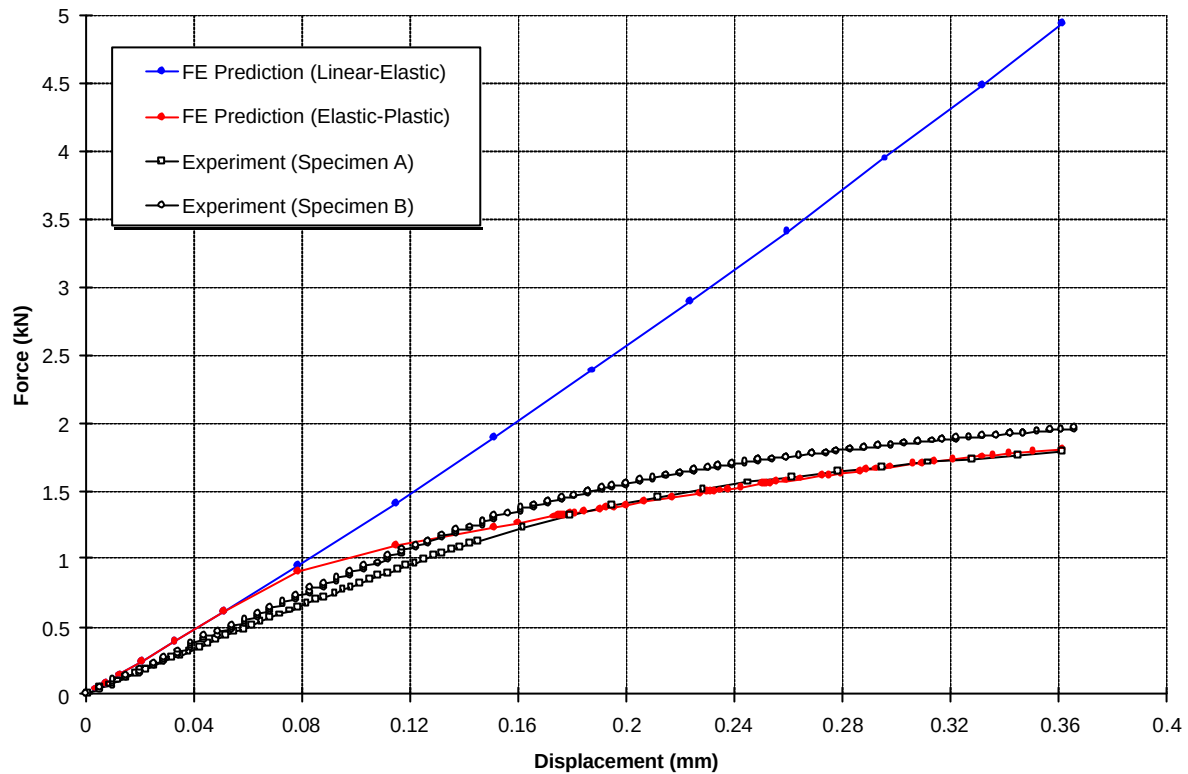


Figure 7 Comparison of measured and predicted force-displacement response.

There was good agreement between the predicted and the measured force-displacement curves when plastic yielding of the adhesive and adherend materials was taken into account (Figure 7). The linear-elastic finite element model, however, over-predicted the stiffness of the joint at high loads by more than 100%.

An important observation is that experimental results were lower than the non-linear finite element model predictions. This was consistent with results for other joint geometries analysed within the Performance of Adhesives (PAJ) programme. Lower stiffness values in test results have also been reported in the literature [9]. This may be attributed to a combination of reasons. Possible manufacturing inaccuracies in the fillet formation or slight defects in the bond line would give stiffness values on the low side. **Manufacturing errors are critical in the T-peel joint, which has a high proportion of Mode I loading.**

Key Observations

- Peel stresses and joint deformation increase with decreasing adherend thickness.
- Peel stresses decrease with increasing fillet size (substantial effect).
- Peel stresses relatively unaffected by increasing the external radius R_o .

NB. Peel stresses can also be expected to decrease with increasing adherend stiffness and to increase for more flexible adhesives.

The results of the finite element analysis of hot/wet conditioned CR1/AV119 joint were markedly different from those of the dry joint. Figure 8 shows the effect of moisture ingress on Mises equivalent stress distribution along the centreline of the adhesive layer for

the miniature T-joint geometry. The Mises stress distribution is governed by peel stresses (Figure 9). Peel stresses, and hence Mises stresses, in the vicinity of the adhesive fillet diminish with exposure time (NB. AV119 epoxy is a moisture sensitive adhesive, undergoing rapid loss of strength and modulus when exposed to hot/wet environments [6]).

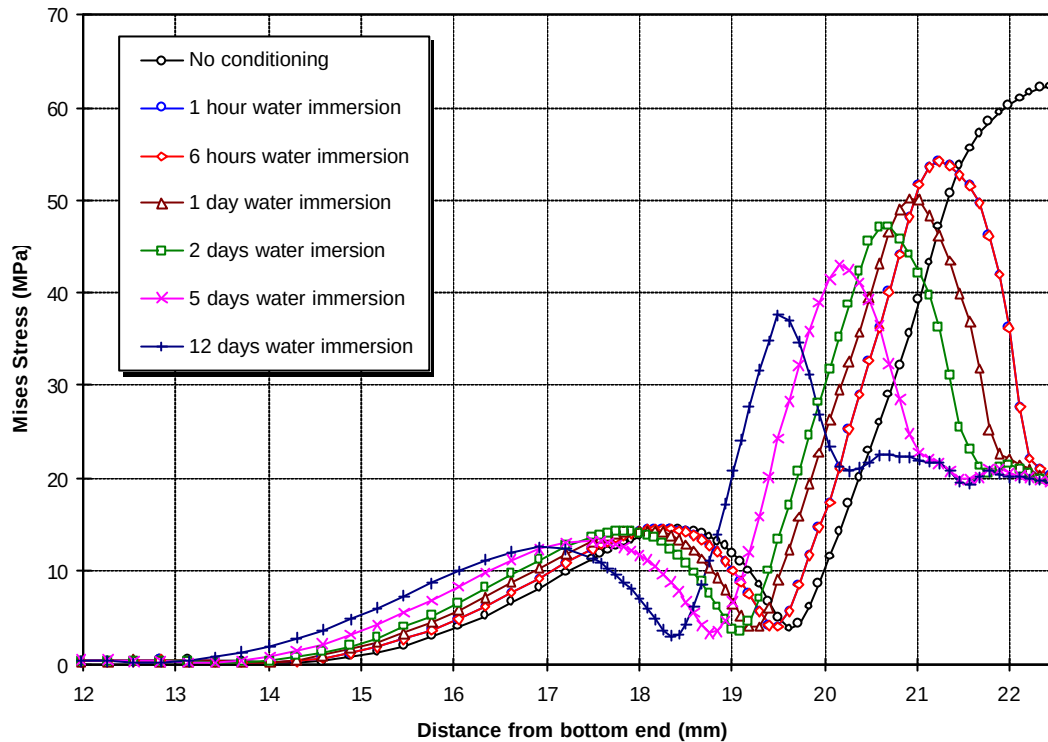


Figure 8 Mises equivalent stress distribution along the centreline of the adhesive layer.

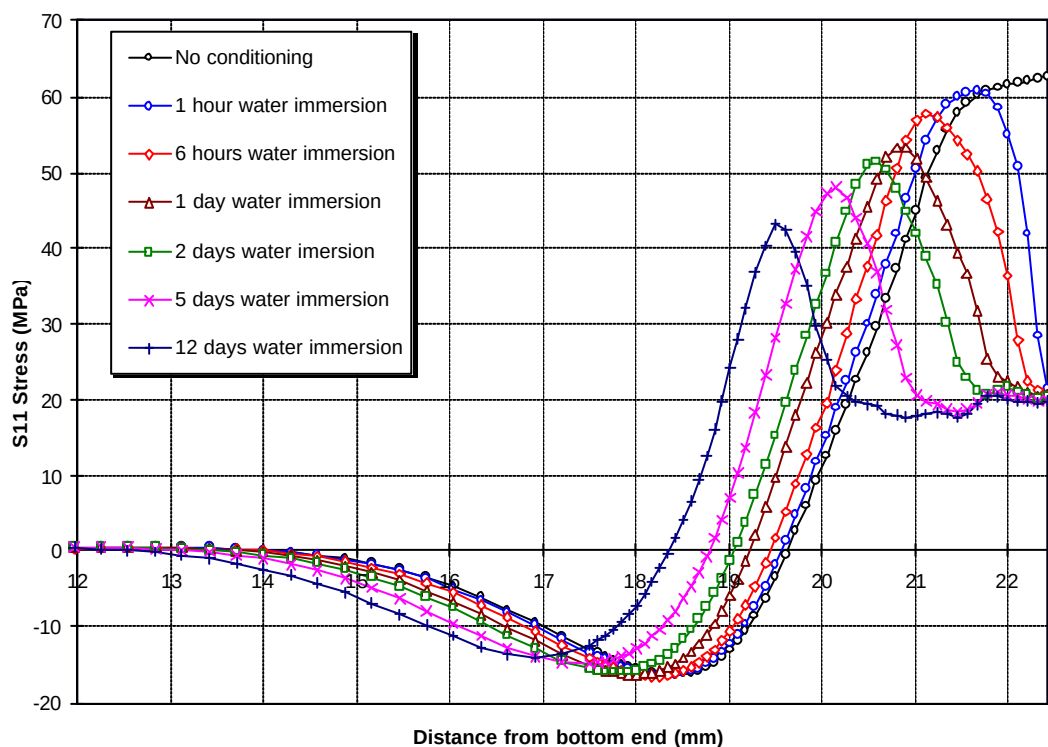


Figure 9 Peel stress distribution along the centreline of the adhesive layer.

3. QUASI-STATIC TESTS

This section presents the experimental results of tensile tests conducted on the smaller T-peel geometry shown in Figure 2. Specimen preparation, testing and data analysis were identical to that employed for the unconditioned specimens described in Section 2. Tests were conducted under standard laboratory conditions (i.e. 23 °C, 50% RH). Specimens were pre-conditioned at 70 °C and 85% RH for up to 1,000 hours (42 days). Conditioning was undertaken using Climatic System environmental chambers. The temperature and humidity were controlled to within ± 2 °C and ± 5 % RH, respectively. **Humidity control (% RH) can also be achieved at various temperatures by saturated solutions of salts and salt mixtures.** Five specimens per condition were tested to failure.

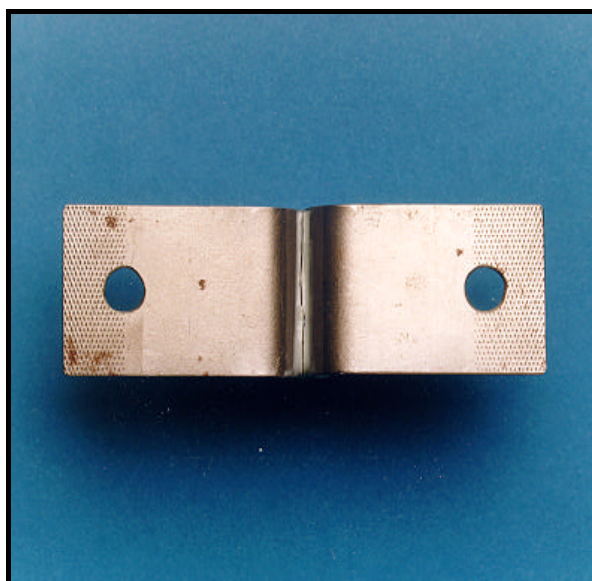


Figure 10 Failed T-peel joint.

All specimens (i.e. dry and conditioned) exhibited cohesive failure within the adhesive fillet (Figure 10) with crack propagation predominantly through the adhesive layer with a small amount of interfacial debonding at longer exposure times (i.e. 42 days). Striations were observed on the failure surface. The results of the quasi-static tests conducted on hot/wet conditioned specimens are shown in Table 2 and Figure 11.

Table 2 Effect of Hot/Wet Conditioning on Failure Load for Unstressed T-Peel Joints

Exposure Time (days)	Static Strength (N)	Average Peeling Force (N)
0	1873 $\pm$ 123	368 $\pm$ 47
3	1402 $\pm$ 187	360 $\pm$ 41
7	1378 $\pm$ 121	320 $\pm$ 12
14	1533 $\pm$ 198	341 $\pm$ 22
21	1330 $\pm$ 138	306 $\pm$ 25
42	1087 $\pm$ 293	334 $\pm$ 36

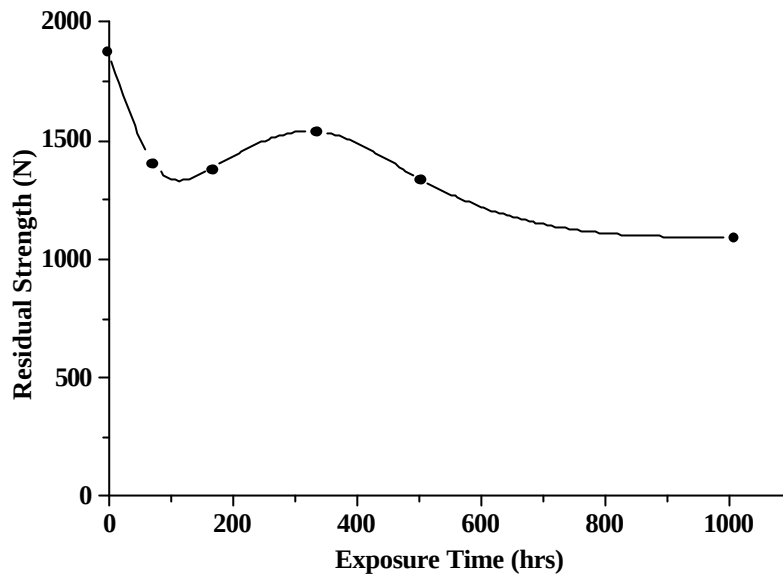


Figure 11 Residual strength (peak load) as a function of exposure time for unstressed joints (line added simply to guide the eye)

The results show that the load required to initiate failure in the adhesive fillet decreases with exposure time, whereas the average peeling force remains relatively constant. Tests conducted at the National Physical Laboratory (NPL) on hot/wet conditioned dumbbell specimens manufactured from AV119 epoxy have shown that the tensile strength of the bulk adhesive also decreases (i.e. linearly) with moisture content (Figure 12). It may be argued that the smaller specimens do not have sufficient bond length for reliably determining the average peeling force, but the load-displacement response was observed to asymptotically increase to a constant value following initial failure.

Specimens stored under standard laboratory conditions (23 °C, 50% RH) were observed to degrade. The peak load for standard test geometry decreased from 1374 N to 1100 N within 42 days of manufacture. A similar reduction could also be expected for the smaller specimens. **Adhesive joints should be stored in a dry (i.e. low humidity) area.**

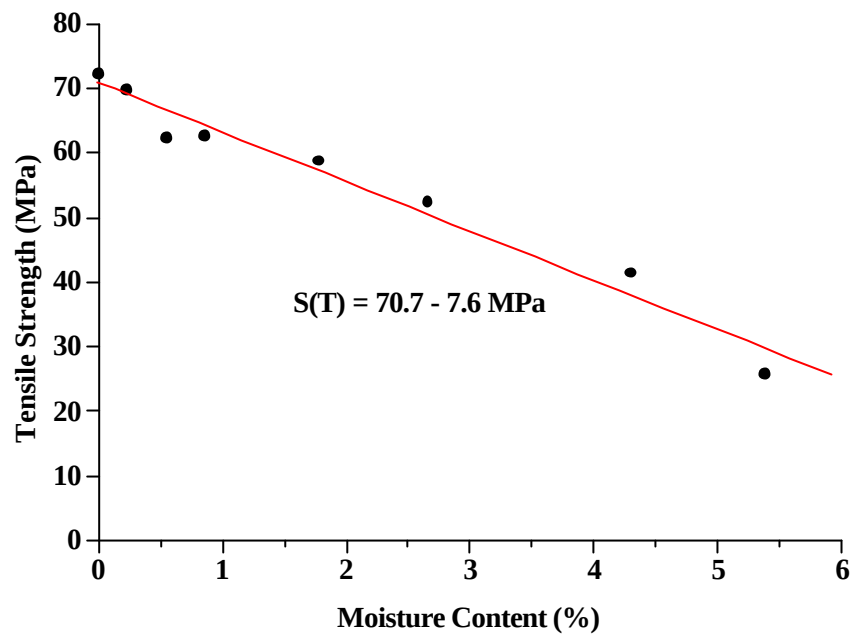


Figure 12 Tensile strength as a function of moisture content for AV119 bulk adhesive.

4. RESIDUAL STRENGTH OF PRE-STRESSED T-JOINTS

This section examines the results of quasi-static tests carried out under ambient conditions on T-peel joints that have been statically loaded for predetermined periods of time whilst being exposed to 70 °C and 85% RH. Tjoint specimens were loaded to 25% of the ultimate static strength (USS) as measured in an unconditioned (i.e. dry) state.



Figure 13 Specimen loading tube.

Creep loading was carried out using self-stressing fixtures (Figure 13) where specimens were placed in a tube equipped with a pre-calibrated spring system for loading the specimens. The spring system can be compressed and locked in place to apply the required load with the spring stiffness determining the load range. The components of the self-stressing fixture were manufactured from stainless steel. Details of the of the tube construction and components can be obtained from ISO / DIS 14615 [10].

Six specimens were bolted together in series with stainless bolts and then loaded. The bolt holes were 6.35 mm (1/4 inch) in diameter and located 10 mm from each end (Figure 10). The specimen-loading tubes were suspended vertically within an environmental chamber for periods up to 42 days at either 23 °C and 50% RH or 70 °C and 85% RH. The results of tensile tests on the pre-conditioned specimens are shown in Tables 3 and 4, and Figure 14. Mechanical test procedures and data analysis methods were identical to those described in Section 3.

The results in Tables 3 and 4 clearly indicate that hot / wet conditioning has no substantial effect on the average peeling force for pre-stressed specimens. There is a small decrease in the average peeling force within the first 3 days, but no noticeable effect for the remaining time span. The general trend is for the peak load to decrease with exposure time with the decrease being more marked for those specimens pre-conditioned at 70 °C and 85% RH. At intermediate exposure times, the static strength (peak load) actually increases. A similar phenomena was observed for single-lap joints that were immersed in water at 60 °C [11-12].

The increase in strength is attributed to plasticisation of the adhesive. This results in a reduction in peel stress concentrations in the vicinity of the fillets to levels lower than experienced by drier specimens (Figure 9), thereby masking loss of tensile strength of the adhesive adhesive and any interfacial effects. The combined effect of pre-stressing and environmental exposure enhances this effect (Figure 14). Tensile loads tend to open existing cavities within the adhesive layer, thus accelerating the diffusion process.

**Table 3 Effect of Combined Loading and Environmental Exposure
(23 °C and 50% RH/25% USS)**

Exposure Time (days)	Static Strength (N)	Average Peeling Force (N)
0	1873 ± 123	368 ± 47
3	1598 ± 155	315 ± 19
7	1467 ± 107	307 ± 14
14	1619 ± 107	303 ± 13
21	1623 ± 105	331 ± 24
42	1431 ± 208	310 ± 14

**Table 4 Effect of Combined Loading and Environmental Exposure
(70 °C and 85% RH/25% USS)**

Exposure Time (days)	Static Strength (N)	Average Peeling Force (N)
0	1873 ± 123	368 ± 47
3	1048 ± 64	302 ± 13
7	1200 ± 208	299 ± 17
14	1340 ± 282	318 ± 11
21	789 ± 153	286 ± 70
42	566 ± 266	330 ± 11

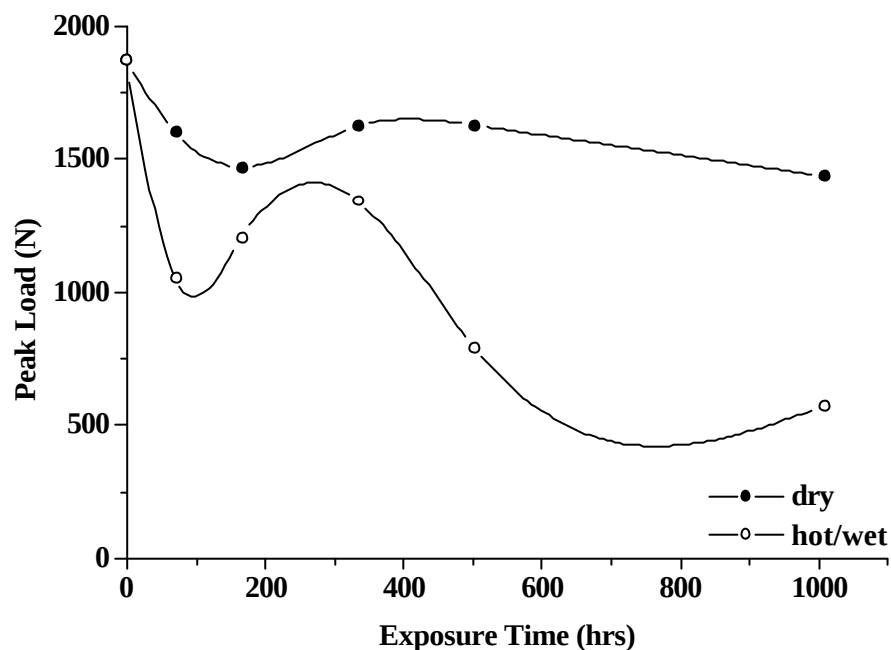


Figure 14 Residual strength as a function of exposure time for stressed joints.
(lines added simply to guide the eye)

5. LIFE EXPECTANCY

An alternative approach to that presented above is to measure the time to failure of pre-stressed bonded joints. In this procedure, the failure times are measured at which the first three specimens fail [13]. Failed joints are replaced with spacers and the remaining specimens re-stressed. When the third specimen fails, the remaining specimens are removed from the loading tube and tested to failure to determine residual strength. The average life-time of the failed specimens and the residual strength of the remaining specimens are used for ranking surface treatments and discriminating between various adhesives.

For this programme, the maximum exposure period was set at 500 hours (21 days) with failed specimens continuously being replaced throughout this period. Failed joints were replaced with spacers and the remaining specimens re-stressed. The remaining specimens after 500 hours were tested to failure to determine the residual strength. Specimens were pre-stressed at five stress levels (i.e. 80%, 70%, 55%, 40% and 25% of quasi-static strength measured at ambient) using the loading tube shown in Figure 13. Pre-stressing procedure and environments were identical to those employed previously. Six specimens were loaded in sequence. At each stress level, time to failure was recorded for all failed specimens. The results of the creep tests for both ambient and hot/wet conditioning are shown in Table 5.

Table 5 Effect of Combined Loading and Environmental Exposure on T-Peel Joints

Applied Load (%USS)	Ambient	Hot/Wet Conditioned
0	1873 ± 123 N*	1330 ± 138 N
25	1623 ± 105 N	789 ± 153 N
40	1163 ± 235 N	192, 264, 264, 264 > 500 hrs (2 off) > 500 hrs (493 - 833 N)
55	1123 ± 248 N	168, 168, 168, > 500 hrs (3 off) > 500 hrs (658 ± 40 N)
70	1238 ± 90 N	< 16 hrs (6 off)
80	16, 156, 360, 408, 480, > 500 hrs (1 off) > 500 hrs (1213 N)	< 16 hrs (6 off)

* Specimens stored under standard laboratory conditions will degrade.

Key Observations

A number of key observations, listed below, can be made in regard to both the test results and test procedure.

- Residual strength is not necessarily a reliable measurement of residual life. Bonded joints can maintain a substantial proportion of quasi-static strength up to the moment of failure.
- Creep limit of T-peel joints was approximately 1200 N for ambient conditions.
- The onset of failure is relatively rapid at load levels approaching the unexposed / unstressed joint strength.
- The average time to failure decreases with increasing load with the onset of failure occurring earlier for hot/wet conditioned specimens. Failure tended to initiate in cohesive manner in the adhesive fillet. Interfacial debonding was observed in specimens that had been exposed for 42 days at 70 C and 85% RH, and then well away from the adhesive fillet.
- **There is a tendency for surviving specimens to be damaged in the re-stressing process with the probability of occurrence increasing at high loads (see hot/wet conditioned results in Table 5).** Creep/relaxation history of specimens due to the replacement of a failed specimen and re-loading will be different. For long term tests over months or years, this effect will probably be minimal [10]. The loading

tubes are best employed at low stress levels ($\leq 25\%$ USS) over considerable timescales.

- **Load calibration of the self-stressing fixtures as a function of spring extension is relatively straightforward, however, pre-setting load and maintaining constant load throughout the test is difficult .** The procedure of applying a pre-set load relies on accurate in-situ measurement of the spring extension. Unless either a load cell, displacement transducer or a small contact (circuit breaker) switch are employed it is impossible to determine the time to failure accurately. **For tests expected to last 1-2 years, or more, and where inspection is on a daily basis this may not be relevant.**

An alternative approach to using the self-stressing fixtures employed in this programme would be load specimens individually in smaller tubes, which will add to the overall costs of testing, but would ensure a more reliable method of determining average life expectancy. Ideally, the loading chain should be instrumented to ensure an accurate indication of time to failure. The usual approach is to carry out a routine visual inspection for failed specimens. A servo-hydraulic or pneumatic system that maintains constant load rather than a spring would be a preferable option for short-term tests.

6. CONCLUDING REMARKS AND DISCUSSION

The results clearly show that under hot/wet conditions the ability of the joint to sustain load is severely reduced. The combined effect of environmental exposure and mechanical loading can be estimated using self-stressing fixtures provided the loads are relatively low in respect to the unstressed/unconditioned joint strength. The T-joint specimens were observed to increase in strength following short duration's of environmental exposure. This increase in strength was attributed to plasticisation of the adhesive, resulting in a reduction in peel stress concentrations within the adhesive fillet region to levels lower than experienced by drier specimens, thereby masking loss of tensile strength of the adhesive and any interfacial effects. The combined effect of pre-stressing and environmental exposure enhances this effect.

A number of conclusions/recommendations (see also Sections 2 and 5) can be made in respect to the results obtained from the assessment of the T-peel joint, creep test results and self-stressing fixtures.

- ISO 11339 [2] should include the following modifications:
 - An alignment/bonding fixture (see Sheasby et.al [5]).
 - Specification on fillet size and method of controlling fillet size.
 - Specifications on external radius **R_o**.
 - Specifications for non-standard plate thickness and non-metallic materials
- Average peeling force as defined by the ISO standard was relatively small and insensitive to changes in adhesive properties, whereas changes in the peak load were large and reflected moisture degradation of the bonded joint. Coefficients of variation of peak load measurements were high, typically 10 to 20% and higher for long-term environmental tests. Compact tension or mode I fracture toughness test

are probably more reliable techniques than the T-peel joint for discriminating between various combinations of pretreatment and adhesives.

- The test results indicate that for moisture sensitive adhesives, such as AV119, it should be possible to relate the strength reduction of T-joints joints with changes in moisture content, provided a suitable failure criteria could be found and that failure is cohesive.
- The implied simplicity of use and low cost of testing has encouraged a number of industrial concerns to employ self-stressing fixtures in large quantities for long-term and accelerated testing. On closer scrutiny these test fixtures need to be used with due care for the reasons outlined in Section 5 (see Key Observations). However, these devices could be used in other allied applications (e.g. polymer matrix composites) if instrumentation were to be introduced. Instrumentation, although adding substantially to costs, would also have a direct impact on improving the reliability of durability data obtained with these devices. Interpretation of failure (i.e. complete separation of adherends or percentage loss of stiffness) could be better defined.

Finally, as previously mentioned residual strength may not necessarily be a reliable measurement of residual life since bonded joints can maintain a substantial proportion of quasi-static strength up to the moment of failure. The preferred approach would be to monitor the lifetime of bonded joints at prescribed stress levels. For improved reliability, a larger sample population should be considered than the current approach of measuring the lifetimes of the first three specimens as specified in ISO / DIS 14615 [10].

ACKNOWLEDGEMENTS

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