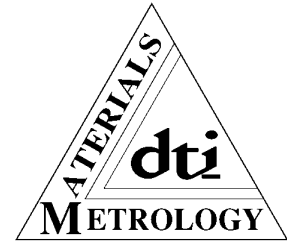


Durability of Adhesive Joints

A Best Practice Guide

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1. Background

Considerable uncertainty exists regarding the long-term durability of adhesive bonded joints, and this uncertainty is responsible for inhibiting the wider use of adhesive technology in many, but not all, industries. There has been an enormous prior investment in durability studies and several attempts have been made to predict life time under adverse environmental conditions. The development of simple, universal, test methods and models to predict the service life of real bonded assemblies from short-term laboratory experiments, however, remains an elusive goal.

One obvious, but nevertheless crucial, factor is that every substrate/adhesive interface is unique. It would be invalid to conclude that the response of a test method to changes in one joint condition, e.g. surface treatment, would be the same, say, with a different adhesive. The different test methods and their variants exhibit different degradation characteristics although, in general, the derived interpretation of durability is broadly similar.

Another important factor is that some bonded assemblies are subjected to a wide variety of loading and environmental conditions in service, perhaps for years or decades, whilst others are merely required to resist minor fluctuations in service environments for short lifetimes. With these factors in mind it may be appreciated that large numbers of test procedures exist which are materials-specific, applications-specific, or which have been developed to meet the objectives of a particular industry.

A general objective of most durability tests is to provide confidence in the long-term performance of a bonded structure. Even with simple screening trials there is an assumption that joint combinations with the lowest degradation should give the best performance in real-life service. While there is some validity in such a generalisation it is not possible to give a precise and accurate prediction of the long-term performance for the following reasons:

- the acceleration factors of different environmental ageing conditions cannot be determined;
- the real-life service conditions of typical bonded products, particularly engineering assemblies and structures, cannot be predicted or accurately simulated;
- the degradation mechanisms under accelerated ageing environments may be very different to real-life conditions;
- periods of load relaxation and mild, dry environments allows some recovery of properties in natural service conditions.

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Where there is an accumulation of historical experience on a particular bonded joint configuration, it can provide a good measure of long-term behaviour against which the results of durability tests can be compared. Where such data are available, the enhancement of results in accelerated ageing tests should reasonably correlate with improvements in long-term performance.

2. Industrial Requirements

2.1 Introduction

Eight industrial sectors, shown below, were identified as major users of adhesives. For each industry, the test methodologies and predictive methods were reviewed. These reviews contain information on past and current practice in relation to environmental durability testing in several industrial sectors. The sectorial groupings chosen cover a very wide range of industrial activity, and within some sectors there exist a large number of further sub-divisions either by applications or by adhesive materials. An enormous variety of substrate and 'adhesive' materials are covered, where the term 'adhesive' is used in the broadest sense.

- Automotive
- Aerospace/Defence
- Construction
- General Engineering
- Packaging
- Footwear
- Electrical/Electronic
- Timber Products

Appendix A.1 provides more details on the reviews which are reported in *MTS Adhesives Project 3 Report 1^[1]*. The reviews examine aspects such as: popularity; utility and ease of use; accuracy and reproducibility; relevance to industry sector; limitations on materials; environmental and service conditions; in-service correlation; qualitative or quantitative data; parameters recorded; interpretation and use of information; acceptance or pass/fail criteria; cost; limitations of procedure.

The quality and amount of information publicly available in the various industry sectors varies widely. Naturally there exist numerous in-house test procedures and associated data to which the authors had only limited access. However by a combination of literature searches, backed up by visits and discussions, it was possible to put together fairly comprehensive reviews covering the eight industrial sectors chosen. It is inevitable that some references are cited which cannot be accessed, and the reader is asked to bear this in mind in following up information of interest.

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2.2 Classification

One theme running through the environmental durability testing used in many of the industrial sectors is that of a broad division into three main approaches or methods, namely *Analytical, Developmental and Standard/Routine*. These approaches are characterised by their complexity and the types of organisation involved in their implementation. The industry sector reviews have considered methods falling into all three categories.

Analytical methods cover approaches aimed at providing a greater understanding of joint degradation mechanisms, seeking to combine a theoretical knowledge of physico-chemical processes with quantitative data obtained from a variety of test procedures including water absorption experiments on bulk adhesive materials. This information is then used to predict joint performance and long-term durability.

Developmental methods cover mechanical test procedures in which bonded joints are subjected to accelerated ageing regimes, often including extremes of service temperatures and moisture/humidity. The tests are generally used to provide comparisons in performance between different bonding systems where adhesive, substrate and pre-treatment represent the variables of interest. Where fracture mechanics-type specimens are employed it may be possible to monitor quantifiable parameters such as G_{IC} as a function of time and exposure condition, under static or cyclic loading. However the test methods generally provide a qualitative measure of joint performance prediction, requiring a lot of judgement and experience to make significant use of the data.

Standard/routine methods describe straightforward mechanical test procedures which may be used primarily for making broad comparisons between different adhesives. They may also be used for specification of materials at the design stage as well as for procurement, quality control and supply during manufacture. They are often developed as the basis of agreement between user and supplier.

An important but elusive aspect of testing relates to pass/fail or acceptance criteria. Although frustrating, it is inevitable that no consistent criteria emerged from the surveys. This is due, in part, to the multiplicity of materials, joint geometries, bonded assembly stiffnesses, loading configurations, environmental exposure regimes, and so on. Indeed in each industrial sector, for the same test joint configuration and exposure parameters, criteria either may not exist or else are adjusted to suit particular combinations of adherends, adhesives, and surface treatments matched against in-service conditions. Nevertheless it is clear, for example in the aerospace and footwear industries, that acceptance limits are placed upon the performance of peel and crack extension test joints in particular. Similarly, in the automotive and construction industries lap joint strength retention limits are indicated. Elaborate strain and environmental cyclic endurance tests exist for building construction sealants which, although not widely adopted, provide an unambiguous framework for acceptance - that is, getting to the end of the test!

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2.3 Conclusions from the reviews

Within the eight industrial sectors surveyed an enormous variety of adhesive bonded joints exist of all types and sizes, utilising a variety of adhesive and substrate materials. The fabrication conditions under which the joints are made also vary markedly. The applications, service environments and service lives encountered by these joints are also rather diverse. Since the range of applications, joint types and materials from which they are constructed may be enormous, even in each industrial sector, it is not surprising that a large number of durability test procedures exist.

In considering the durability performance of a bonded joint it is useful to separate the effects of moisture, temperature and stress on the:

- Adhesive material
- Adherend material(s)
- Adhesion between the adhesive and adherend materials.

A major question which therefore arises is the purpose of destructive mechanical joint tests. That is to say, whether the methods are intended to examine interfacial bond stability, adhesive material changes, or the performance of the bonded system. In some industrial sectors these concepts are acknowledged, understood and investigated, whilst in others such distinction is poor or completely overlooked. In most of the industries surveyed the need to include environmental exposure and imposed stress in trials used to investigate and optimise pretreatments is acknowledged. However there is much confusion over the choice of environment for example, should it be humid, wet, hot/wet (or hot/cold in the case of concrete repair materials), or should the environment simulate extremes likely to be encountered in service? And what about the type, nature and magnitude of an applied load? The same questions extend to assessing the durability performance of the bonded system, together with considerations of joint size.

Test joints, parts of real joints or even fairly small real joints (in electronics, packaging and footwear applications) are tested. However there may be substrate limitations on appropriate configurations where very flexible (eg. in footwear and packaging), very brittle (eg. concrete), or layered (eg. timber, polymer composites) materials are involved. It follows that the materials involved in many industrial sectors do not lend themselves to deformation in ideal joint configurations, or are inappropriate for use in some types of joint and/or stress regimes.

In principle there is a relatively limited number of basic test joint configurations, although variations in material thicknesses and subtle geometrical changes may influence joint behaviour dramatically. Conventional approaches utilise lap shear, tensile butt and peel joint configurations, the latter being appropriate to flexible adherend materials. Changes in joint strength are recorded as a function of time, and the mode and locus of failure noted. The data produced from these *Standard and Developmental* methods are not quantitative and extrapolation to larger real-scale joints is questionable. Stressed durability (fracture

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mechanics) test methods which can provide data on tensile opening (Mode I) fracture energy were developed in the aerospace industries. Cantilever beams and blister specimens are common, and quantitative information on the bonded system may be expressed in terms of fracture energy versus period of exposure. Such test procedures often fall within the categories of both *Developmental and Analytical* methods.

Despite the relatively limited number of available test joint configurations, the behaviour of any particular joint is a function of its' quality and the materials present. Changes in stiffness of adhesive or adherend can affect peel or lap shear joint behaviour markedly and this is not always appreciated. For example, the properties of adherends such as timber, leather and polymer composite materials may alter substantially as a result of exposure to hot, wet, or hot/wet conditions. Similarly, changes in moisture content or temperature can introduce large dimensional changes in concrete and timber, giving rise to major changes in bonded joint performance which are not strictly related to the fact that it is a bonded assembly. Changes in adhesive material stiffnesses can be favourable in terms of bondline plasticisation and redistribution of peak stresses in joints, at least in the short term. Thus somewhat conflicting and confusing short-term 'strength' data may be obtained from durability trials using unstressed lap shear joints. It is clear that the proper and complete interpretation of data leaves a lot to be desired, particularly from apparently straightforward test programmes. Misinterpretation of data, due to a lack of knowledge, remains a problem which is at least as great as the use of inappropriate test procedures.

Many environmental test regimes are employed, generally to simulate the conditions and extremes appropriate to the service conditions of joints. Thus low and high temperatures, humidity, liquid water, exposure to various chemicals, LTV, and various combinations of these, are used either as constant or cyclic conditions. Heat is generally used also as a proxy for time to provide accelerated ageing, but there are dangers involved in subjecting test joints to unrealistically high temperatures which may give rise to changes which would never occur in service. Simultaneously joints may be unstressed, subjected to constant or cyclic stressing (or straining in the case of construction sealant materials). Naturally the logistics and expense of imposing elaborate loading and environmental conditions on bonded joints become too prohibitive for routine use, and relatively simple procedures are preferred. BS EN29142 (1993)/ISO 9142 (1990) 'Adhesives - guide to the selection of standard laboratory ageing conditions for testing bonded joints', describes several single and multi-variable atmospheric ageing conditions for bonded assemblies. It will be interesting to see how many investigations adopt the suggestions as documented.

It is vital to collect information on test joints exposed to natural weathering conditions, particularly if subjected to stress (or strain). A comparison of such information with real joint data and laboratory data enables much useful understanding and knowledge to be gained. Such an approach exists particularly in the aerospace and footwear industries, although there are moves towards this in the automotive, general engineering and construction industries. *The Task 4 Forensic studies* provided an additional complement to existing knowledge. To such information must be added data on both adhesive and adherend materials, in order to

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provide sufficient data so that a prediction of service life may be made. Some *Analytical* procedures which encompass predictive durability models are employed generally only so far in the aerospace industry, but interest is growing at least in the automotive industry and for structural bonding in the construction industry.

The remaining obvious challenge facing most of the industrial sectors is the development of *Analytical* methods and models to predict the service life of real joints from short-term laboratory experiments and from a theoretical understanding of adhesion, joint degradation mechanisms and joint behaviour. With footwear, packaging and electrical/electronic bonded assemblies, the service lives tend to be relatively short and the scale of the product fairly small, enabling service performance feedback within a reasonable technical timespan. However in many industries the bonded joints are often large and of complex geometry, making accurate models and predictions of life extremely difficult.

The commentary in *MTS Project 3: Report No 2 Evaluation of Published Durability Data*^[2] explores some of these themes rather more fully, particularly in relation to current test joint behaviour and interpretation of test data.

3. Assessment of Test Methods

3.1 Introduction

The selection of a durability test method for a specific application clearly depends on the user's particular requirements. Generally the industrial application of adhesives requires a comprehensive definition of the total bonding system, i.e. the adherend material, the adhesive and surface treatment, as an integral unit. The durability test should replicate these conditions as far as possible. In practice, however, the selection of an appropriate test method for a particular durability study is likely to be determined by other criteria, such as ease of use and availability of material in suitable form for test pieces.

The following sections summarise the findings of work reported in *MTS Project 3 : Report No 8 Experimental Assessment of Durability Test Methods*^[3]. The general configurations for the various test methods are shown in Figure 1.

3.2 Review of Test Methods

3.2.1 Simple Lap Joint Durability Test

The single lap joint test is the most widely used test method for durability studies. Its popularity lies in its relative simplicity and perceived ease of interpretation. In view of its widespread use and acceptance by researchers and industrial practitioners, the lap shear test

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was selected as the core test method in the matrix of the environmental durability test programme. The test was applied to a range of adherend and adhesive combinations, and several variant combinations of joint size and ageing conditions. The method was also used for the most extensive series of inter-laboratory replication tests.

The summary conclusions of the assessment are as follows:

- The lap shear test provides a relatively simple and convenient method for comparative evaluation of durability of adhesive bonded joints in different environments.
- The method is sensitive to changes in adhesives and surface treatments in the selected joint combinations. However small differences in joint conditions arising from experimental procedures can give rise to uncontrolled variability which may mask the effects of changes in controlled variables. Typical coefficients of variance in the test programme were 20 to 30 percent and occasionally exceeded 50 percent. Batch variations with good bonding systems were 5 to 10 percent.
- This response of the test method to uncontrolled experimental variability can give rise to problems in reproducibility, particularly when failures are interfacial.
- The test method gives a measure of the response of the bonded system to the compounded effects of many different mechanisms and changes which occur during the ageing process. It is not possible to determine the relative magnitudes of the various effects in degradation.
- Some systems exhibit increase in strength during early weeks probably due to one or more of the following effects: plasticisation of the adhesive by the absorbed moisture; post cure of the adhesive by the elevated temperature of the ageing environment; relaxation of the residual stresses in the adhesive.
- The ageing time required to obtain reasonably confident degradation characteristics can be several months depending on the adhesive/adherend/surface treatment combinations. Reducing joint dimensions to give a smaller bond area does not significantly affect the degradation rate.
- Within nominally similar joint system combinations it is possible to draw broad generic observations on the effectiveness of different surface treatments and adhesives on durability performance. It is NOT possible to use the lap shear test to predict quantitatively the behaviour of joints when exposed to ageing environments, or their long-term behaviour in service conditions.

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3.2.2 T-peel Durability Test

Peel tests are quite widely used to evaluate adhesive performance of adhesive bonded joints, particularly with thin, flexible adherends. Various configurations, including climbing drum and floating roller, have been developed to provide particular test method characteristics. The T-peel test is a simpler arrangement which can be used on symmetrical joints.

The application of peel testing to durability studies typically consists of placing prepared peel test specimens in an ageing environment and measuring peel strengths after selected ageing periods. The graphical recording of peel strength values over a relatively long strip provides some degree of 'averaging'.

In this MTS project the T-peel durability was applied to thin aluminium alloy and polypropylene substrates bonded with different adhesives and surface treatments.

The general assessment of the test method concluded that:

- T-peel tests can clearly distinguish between various combinations of pre-treatment and adhesives.
- The coefficients of variation in measured values of peel strengths from typical batches are of about the same order of magnitude as lap joint tests. The more durable combinations give coefficients of variation in the order of 10 percent.
- Several test combinations give abnormal degradation characteristics, i.e. increasing peel strength at some stage during the ageing process presumably due to plasticisation effects.
- A qualitative measure of durability, in terms of poor or good, can be established within about 20 days of ageing. However the unstable behaviour in some systems can lead to difficulties in interpretation and uncertainty in the ranking of durability.
- The method requires greater effort and resource compared with the lap shear test. Its application is generally restricted to thin flexible adherends.

3.2.3 Wedge Cleavage Test

The wedge cleavage test is an adaptation of the double cantilever beam test and was developed to provide a cheaper and easier alternative for routine testing application. The method originated in the US aerospace industry and is often referred to as the Boeing wedge test. It was adopted as an ASTM standard (Designation D3762-79) under the title "Standard test method for adhesive bonded surface durability of aluminium." Abstracts from the standard record that

"This method simulates in a qualitative manner the forces and effects on an adhesive bond joint at metal-adhesive/primer interface. It has proven to be highly reliable in determining and

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predicting the environmental durability of adherend surface preparations. The method has proven to be correlatable with service performance in a manner that is much more reliable than conventional lap shear or peel tests.

NOTE 1 - While this method is intended for use in aluminium-to-aluminium applications it may be used for determining surface durability of other metals and plastics provided consideration is given to thickness and rigidity of the adherends.

NOTE 2 - This method is not a quantitative fracture strength test method."

The assessment of the test method in the MTS study included application of the test to mild steel, aluminium alloy and GRP substrates with 2-part epoxy, 1-part epoxy and toughened acrylic adhesives. The experimental experiences confirm that:

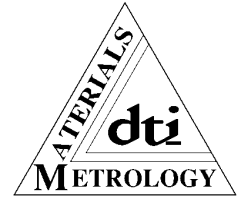
- The wedge test is capable of discriminating between the effects of different surface treatments on durability.
- The measurement of crack length, which provides the indication of durability, can be performed fairly accurately and coefficients of variance are typically better than 10 percent. However as a measure of crack growth, this variability is much higher.
- Different interpretations can be derived from the crack length-vs-time characteristic. The crack growth limit is reached after about 10 days with most systems and this can provide a comparative measure of durability. However an earlier indication of the effects of different surface treatments can be established within 24 hours. This is much more rapid than with any other durability test method and reflects the effects of imposed stress at the crack tip which contributes to the degradation rate.
- The effectiveness and sensitivity of the test is influenced by the ageing environment in which the test pieces are exposed.
- The method suffers from a number of possible sources of variability, some of which arise from interpretation and implementation of the test in practice. There is scope for closer definition of procedures described in the primary standard for this test method, particularly in relation to the starting conditions such as method and rate of wedge insertion and time of first measurement.

3.2.4 Tensile Butt Durability Tests

The tensile butt joint is less commonly used as an adhesive test method, perhaps due to the perception that it is more difficult to prepare the test specimens. Also the adherend materials typically used for bonded engineering structures may be less readily available in bar stock

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form. However it has been preferred as a durability test method by some leading researchers in the subject, particularly for detailed analytical studies on specific surface treatments.

Although a number of different configurations have been used by industry, the MTS study considered the most common configuration, the cylindrical butt joint. Tests were carried out on steel and aluminium alloy substrates in three different diameters with 2-part and 1-part epoxy adhesives. In comparison with the other test methods it was established that:

- The butt joint is capable of being more reproducible with average coefficients of variation of approximately 10 percent within batches. Good inter-laboratory reproducibility was also observed.
- The test is sensitive to changes in surface conditions and exhibits these changes more rapidly than the lap shear test.
- As in other test methods the degradation characteristic reflects a compounded response to a number of different changes and mechanisms which occur in the joint during the ageing process. Some of these changes such as post-cure effects and adhesive plasticisation may be temporarily beneficial and contribute to the occasional observation of increasing strength during ageing.

Compared with the lap shear test, degradation rate in tensile butt joints is more sensitive to the bond area. Thus smaller diameter joints can be used to give a more rapid indication of durability.

The general uniformity of, and absence of distortion on the fracture surface, provides a good facility for analysis of the locus of failure.

Although initial specimen preparation costs are higher than for lap, T-peel and wedge tests, the reusability of the test pieces partially offsets this disadvantage.

3.2.5 Perforated Hydrothermal Test

The application of a controlled stress to a bonded joint during environmental exposure can accelerate the degradation process. A number of test methods have been developed to combine stress with ageing conditions for durability studies. A popular method in the UK, sometimes referred to as the Maddison test, is being considered as a draft ISO Standard [ISO/CD 14615]. The test consists of simple lap joints, usually perforated through the bond area to accelerate moisture ingress, which are spring-loaded as a string of six specimens in a 'stress tube'. The prestressed assembly is then placed in the selected ageing environment and the "time to failure" of the joints is recorded as the measure of durability.

This perforated hydrothermal stress (or stress humidity) test has been evaluated in the MTS project while testing 1-part and 2-part epoxy adhesives with aluminium and alloy mild steel

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substrates. A variant of the test which uses a short tube to accommodate single specimens was also assessed.

Compared with other durability tests, the application of different stress levels in different environments required a greatly extended matrix of test conditions. The selection of appropriate load/environment combinations requires a well-defined rationale for the application of the test and some prior knowledge of the bond system characteristics.

The test work for the study was carried out in three different test centres, each with previous experience in use of the stress humidity test and the general conclusions were:

- The perforated hydrothermal stress test gives a clear discrimination of environmental durability of different bonding systems.
- The time to failure of individual specimens within a test group can vary widely, often in excess of 100 percent in poor systems and typically 20 percent in good systems. However, accepting that log-time provides a more realistic interpretation of durability, this variability may be acceptable.
- Although there is no published reference to the general characteristics of the test method, it is evident that it can operate in two distinct domains: either as a rapid screening test or as a long term test in which the ageing conditions are more representative of service life.
- Rapid failure and, per se, a quick indication of the durability of bonded systems can be produced by the application of high stress on the joint in combination with hot-wet environments. However there is a danger of selecting excessively demanding stress levels and ageing conditions which may give premature failure caused by mechanisms other than environmental degradation.
- The test is significantly more difficult to carry out than the simple lap test. It requires more laboratory resources and considerable user experience for its correct application and interpretation.
- The miniature stress tube configuration which tests individual specimens gives durability performance rankings which are in the same order as the large 6-specimen tubes, but there is large variation in some combinations. Individual values of time to failure either from the miniature tube tests or the 'standard' test should not be used as a quantitative measure of durability.

3.2.6 Fabric Peel Durability Test

The fabric peel test is based on a joint configuration which is similar to a peel joint between a rigid and a flexible adherend but differs in that the flexible element is a woven fabric which is

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embedded in the glue line. When the joint is exposed to wet environments, the open area of the adhesive surface allows rapid moisture absorption into, and diffusion through the adhesive. This gives a considerable acceleration to the degradation process both through changes in the adhesive properties and through interfacial mechanisms.

The technique has been fairly well established for the evaluation of building sealants which are characterised by low moduli and thick bond lines. The application of the concept to adhesives is more recent and should be regarded as developmental. The evaluation of the method within this MTS study has therefore been limited to two adhesives, 2-part epoxy and a 2-part polyurethane on mild steel adherends.

The results of the assessment were as follows.

- The method shows clear discrimination between joints with different surface treatments. For most systems reliable comparative data can be established after 7 days in an appropriate ageing environment.
- The repeatability of the test method depends on the amount of adhesive used. Careful measurement and control of the mass of adhesive on each test replicate gives good, low variability.
- The test provides a good failed surface area from which a detailed assessment of failure modes and mechanisms can be identified.
- Occasional problems arise with the tearing of the fabric in unaged high modulus adhesives. Alternative fabrics may need to be evaluated. Within the current experience the test is more suitable for relatively flexible (low modulus) adhesives.
- In comparison with other durability tests the test pieces are relatively easy to make, the method is easy to implement and the speed of acquisition of results is quite fast.

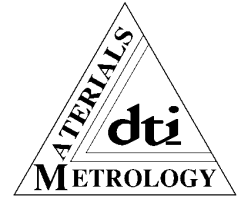
3.2.7 Bead Scrape Durability Test

The underlying concept of this test is not dissimilar to the fabric peel test (Section 3.2.6) in that it attempts to provide a rapid degradation by exposure of a large surface area of adhesive. However in the scrape test, testing is carried out by pulling the rigid substrate through a close fitting die which scrapes the adhesive away from the surface. The measurement of the scraping force is intended to provide an indication of durability. The method has been described in just one literature source and only a limited range of tests was carried out to explore its potential as part of the MTS project.

The results of this assessment showed that:

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- The scrape test provides a limited capability for measuring durability of some adhesive/substrate combinations.
- The mode of failure of the 'bond' exhibits an interesting characteristic, as the bead buckles away from the substrate in a series of progressive steps. This produces a force time curve as a sequence of peaks. Interpretation of mean scrape force from this data is doubtful.
- Small variations in experimental detail, particularly relating to bead dimensions and die-fit, lead to high variability in test results. The test therefore requires a higher degree of precision than could be provided in the present study.
- Within the limited range of tests carried out, the method gave a relatively rapid rate of degradation and sensitivity to surface treatment changes was evident after 3 days of ageing.
- Further developmental work would be required to establish the bead scrape test as a reliable durability test method.

3.2.8 Polymer Film Durability Test

This developmental test was also devised to provide more rapid water ingress into adhesive joints particularly with polymeric substrates. The concept consists of using thin film polymer adherends which allow rapid diffusion of moisture into the adhesive. In order to test the joints in shear, rectangles of the aged adhesive/film materials are rapidly bonded between metal strips to give lap shear joint, using a cyanoacrylate adhesive. The shear test then subjects the film/adhesive joint to similar shear loading.

The method proved to be very laborious and the validity of the results were doubtful due to the possibility of water loss from the interfacial regions prior to testing. Also the objective of the test, to provide rapid access of water to the interface, is achievable with a thin film T-peel test which is more convenient and reliable. The developmental work was therefore discontinued.

The test method may have occasional possible application for thin film polymer laminated materials in which the films are too weak to allow peel tests to be carried out.

3.2.9 Fatigue Lap Shear Durability Tests

This test is essentially the same as the method described in Section 3.2.1 "*Simple lap joint durability test*" except that the testing of the joints after ageing is carried out by cyclic fatigue rather than quasi-static loading to failure. In both tests the joints are unstressed during the ageing period.

The intention of the fatigue test was primarily to provide experimental information to support analytical studies in other tasks in the project. It was envisaged that cyclic loading at a

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subcritical level might induce different failure mechanisms to provide comparative data for fracture mechanics analysis. During the cyclic load testing after ageing, the test pieces were surrounded with wet cotton cloth to provide a high humidity environment and tested either at the ageing temperature or at ambient temperature. The test series was limited to mild steel adherends bonded with 2-part epoxy and 1-part epoxy adhesives.

The method presents a number of complications in its application and implementation as described in the following assessment.

- It is difficult to select and specify the cyclic loading conditions for each test. The control (unaged) test pieces failed predominantly through the adherends although this would be overcome by using thicker test pieces.
- The test pieces need additional processing to allow their fixture into fatigue equipment.
- The fatigue cycling equipment is very expensive.
- The results of fatigue tests under ideal conditions can exhibit considerable variability. The additional inherent variability in adhesive joints gives wide scatter, in necessarily small batches.
- The preliminary developmental work indicated that the preferred testing conditions should be a cyclic load of 50 percent of ultimate strength and carried out after the joint should have reached saturation.
- Within the very limited tests under these conditions, the fatigue test discriminated between different treatments and gave different modes of failure.
- The fatigue lap shear test should not be regarded as a suitable method for determining durability of different bonded joint combinations.

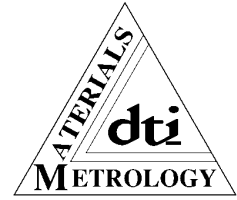
3.3 CONCLUSIONS

Nine different test methods used in the evaluation of durability of adhesive bonded joints were investigated. All of the test methods studied used accelerated ageing environments. In terms of actual performance during testing, this deliberate exaggeration of extreme test conditions gives a much more pessimistic measure of durability than would be encountered in most practical applications of adhesive bonded joints.

Of the nine methods evaluated, two tests (wedge and perforated hydrothermal stress) involved the application of stress on the joint during the ageing period. This provides an additional 'accelerating' feature, although it may be argued that stressed ageing may be more representative of real service life. Two other tests (bead scrape and polymer film) were

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studied as an exploratory assessment of possible new methods but they did not offer significant advantage over existing techniques and they were not developed for further evaluation. The fatigue durability test was also seen to be related to a more analytical experimental regime for fracture mechanics and is unlikely to be applicable as a simple durability comparison test. A more detailed assessment was carried out for lap shear, T-peel, fabric peel, tensile butt, wedge and perforated hydrothermal stress configurations.

For most test methods, the results give a reasonable qualitative comparison of the durability of bonded systems with different combinations of substrate, adhesive and surface treatment. It is also important to note that the locus of failure of durability test joints can give valuable information on degradation mechanisms.

A common feature of nearly all durability test methods is a fairly high degree of variability within test batches. Typically, coefficients of variation of about 20 percent are observed although this is considered to be acceptable in some standards. Care should therefore be exercised in interpretation of individual durability test results. There is scope for further refinement, revision and development of standards for some durability test methods.

The comparative evaluation of the test methods in this programme was based on a number of performance criteria which were categorised as measurement attributes, application attributes and interpretation attributes. Within each test configuration there is considerable scope for experimental variants such as test piece dimensions and ageing environment. The measured response of the test method to different adhesive joint conditions will depend on some of these variants. Therefore generic statements on the comparative abilities of the various tests may not be valid over the whole application range. However the summary comparisons given in the following table provides a general guide of the characteristics and potential areas of use of the different test method. The ability of a particular test method to discriminate is summarised in Figure 2, however, in principle the following holds.

- Lap joints are less discriminating particularly in high durability combinations even after 60 weeks of ageing.
- The tensile butt joints give a greater proportionate difference between durability levels after 12 weeks.
- Stressed joints are potentially better and quicker than unstressed test methods in discrimination of medium to higher durability combinations.

4. Summary

Adhesive bonded joints exist of all types and sizes, utilising a variety of adhesive and substrate materials. They are fabricated under various conditions, see a diverse range of

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service environments and have varying service lives. Since the range of applications, joint types and materials from which they are constructed may be enormous, even in each industrial sector, it is not surprising that a large number of durability test procedures exist.

Many environmental test regimes are employed, generally to simulate the conditions and extremes appropriate to the service conditions of joints. Thus low and high temperatures, humidity, liquid water, exposure to various chemicals, ultra violet light and various combinations of these, are used either as constant or cyclic conditions. Heat is generally used also as a proxy for time to provide accelerated ageing, but there are dangers involved in subjecting test joints to unrealistically high temperatures which may give rise to changes which would never occur in service. Simultaneously joints may be unstressed, subjected to constant or cyclic stressing (or straining in the case of construction sealant materials). Naturally the logistics and expense of imposing elaborate loading and environmental conditions on bonded joints become too prohibitive for routine use, and relatively simple procedures are preferred.

Although there are only a relatively limited number of basic test joint configurations, because the behaviour of any joint is a function of its quality and the materials present, subtle geometrical or material changes may influence joint behaviour dramatically. In many of the generic test types, changes in joint strength are recorded as a function of time, and the mode and locus of failure noted. In general the data produced from these methods are not quantitative and extrapolation to larger real-scale joints is questionable. Stressed durability (fracture mechanics) test methods which can provide data on tensile opening (Mode 1) fracture energy were developed in the aerospace industries. Cantilever beams and blister specimens are common, and quantitative information on the bonded system may be expressed in terms of fracture energy versus period of exposure.

The selection of a suitable durability test method for a specific application clearly depends on the user's particular requirements. Generally, this requires replication of the total bonding system, i.e. the adherend material, the adhesive and surface treatment, as an integral unit. However, in practice, the selection is likely to be determined by other criteria, such as ease of use and availability of material in suitable form for test pieces.

Durability tests are typically used to 'screen' the effectiveness of different combinations of joint detail as part of the pre-design specifications. They provide a general understanding of durability as a quality, however, it is not possible to establish numerical quantities in absolute units for an analytical assessment of durability.

The remaining challenge is, therefore, the development of durability test methods and models to predict the service life of real joints from short-term laboratory experiments and from a theoretical understanding of adhesion, joint degradation mechanisms and joint behaviour. In many industries, however, the bonded joints are large and of complex geometry, making accurate models and predictions of life extremely difficult.

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In order to achieve this goal, it is vital to collect information on test joints exposed to natural weathering conditions, particularly if subjected to stress (or strain). A comparison of such information with real joint data and laboratory data enables much useful understanding and knowledge to be gained. Such an approach exists particularly in the aerospace and footwear industries, although there are moves towards this in the automotive, general engineering and construction industries.

The work carried out as a part of the ADH MTS programme suggested that different test methods were more appropriate to different sections of the durability spectrum. It found that where possible the tensile butt test should be preferred to the lap shear test. If experimental resources and skills are available the wedge test and the perforated stress humidity tests are recommended for greater discrimination on high durability systems.

5. References

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TEST METHODS		ATTRIBUTES			RECOMMENDED USAGE
		Measurement	Application	Interpretation	
Unstressed Joints	Lap shear	Fairly high variability Limited discrimination on high durability joints	Simple, commonly used Extensive published data Relatively slow degradation	Easy to derive degradation curve Occasional unusual characteristics due to complex mechanisms Prediction uncertain	General purpose testing Comparison of less durable systems for bonding specification Routine process control tests
	T-peel	Quite high variability	Restricted to thin flexible adherends Fairly rapid More difficult to use than lap shear	Peel curve gives some 'averaging' Occasional unusual characteristics difficult to interpret	General testing for comparison of durability in flexible adherends
	Fabric peel	Lower variability Fairly sensitive	Most applicable to low modulus adhesives Care needed in accurate preparation Rapid acquisition of data	As T-peel Large failed surface area allows good analysis Locus of failure important to assessment	Developmental studies with lower modulus adhesives and sealants on rigid adherends
	Tensile butt	Better reproducibility Good sensitivity	Generally requires material in bar form Fairly easy to assemble accurately Quite quick degradation	Easy to analyse Good surface area for analysis of failure mechanisms	Preferred for more accurate measurement of durability in development applications
Stressed Joints	Wedge test	Fairly high variability Good sensitivity Discriminates treatments on high durability joints	Easy to prepare but care needed in implementation High modulus compliant substrate needed to avoid yield Quick indication of durability No special equipment required	Problems in precise measurement of crack growth Locus of failure effects evident Fracture energy derivation can be useful	General purpose and developmental tests on high durability systems Quality control tests on critical surface treatments
	Perforated hydrothermal stress	Wide variability Good discrimination on high durability joints	Long preparation process Requires special facilities and skills Acquisition of durability data can be rapid Only suitable for highly cross-linked adhesive systems	Yields large amount of data Experience needed in interpretation May give false indication of durability through premature failure	Developmental analysis in higher durability systems Provision of longer term confidence in stressed assemblies

Summary Table

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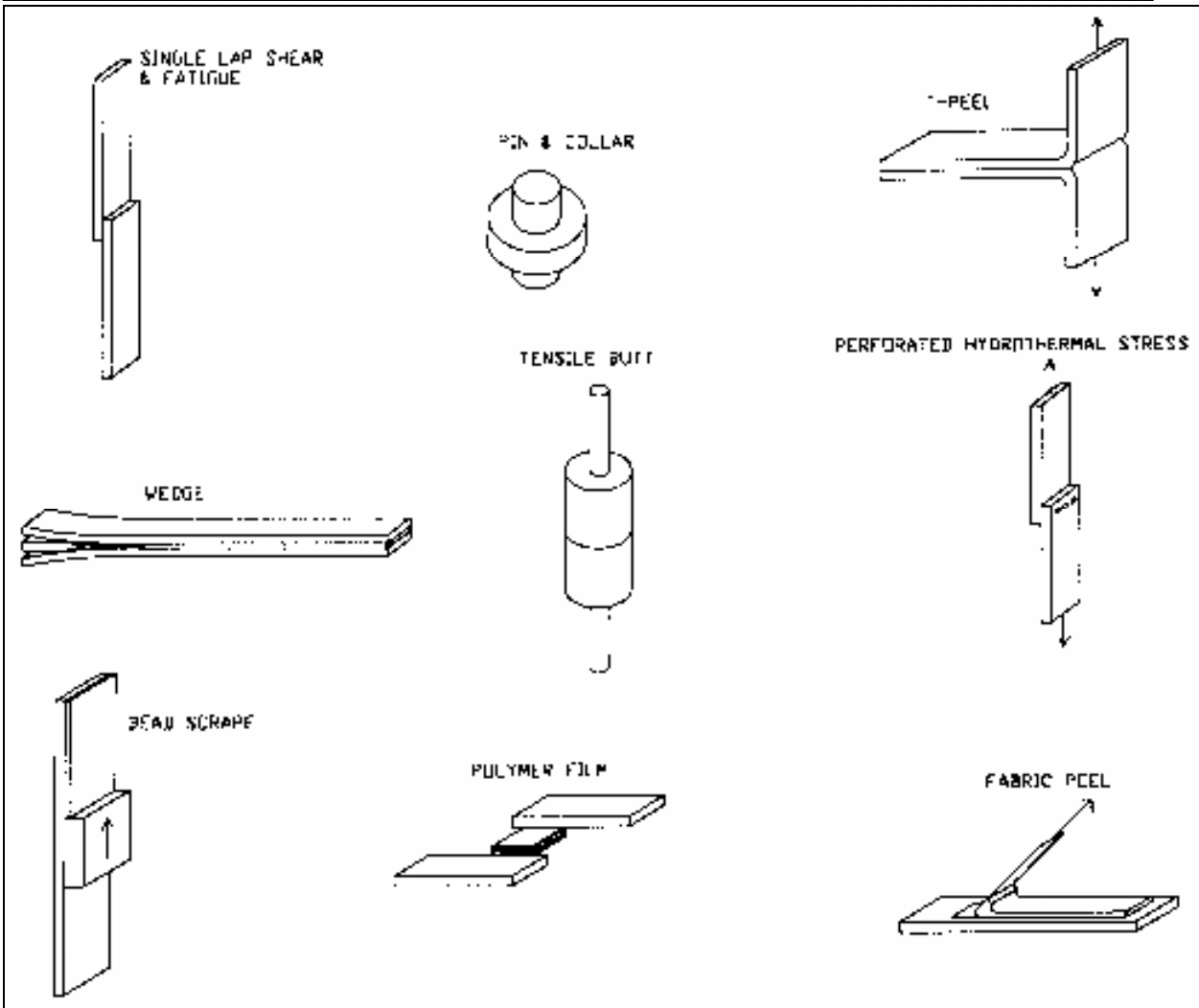
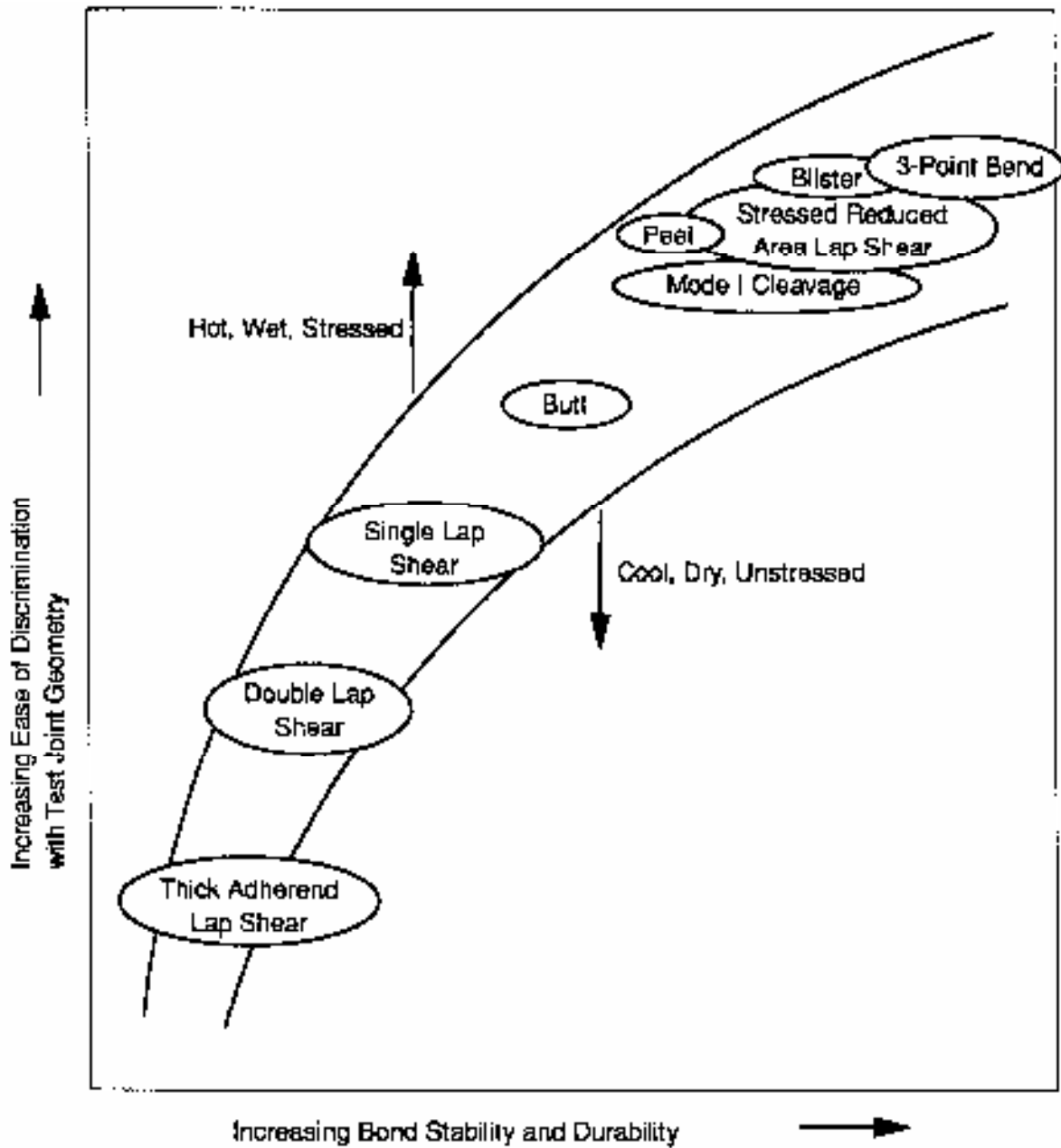
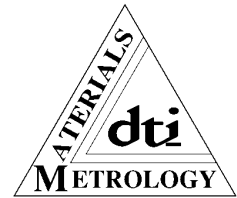


Figure 1: General configurations for the various test methods

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Note that there are often adherend and adhesive material limitations in test method selection.

Figure 2: Ability of a particular test method to discriminate

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Appendix A.1 Reviews by Industry Sector

Automotive Industry

The review of durability test methods for adhesive bonded joints in the automotive industry was based on a survey of literature, company specifications and interviews with industrial practitioners.

It was found that the majority of durability testing carried out for car body applications is based on in-house standards. These are essentially acceptance tests for adhesive materials for particular applications and are identified as *Standard/Routine tests*.

For more quantitative data to support design and selection of bonding systems, the industry also uses a number of *Developmental* test methods which give comparative information on different combinations of substrate, adhesive and surface treatments. These developmental methods are usually based on lap shear joints which are exposed to a range of environments. Degradation is monitored over prescribed time intervals and this provides some indication of expected life. The environmental ageing parameters for such tests are generally chosen to represent the extremes of service conditions.

Some manufacturers are attempting to develop more accurate predictive models based on an understanding of the mechanisms involved in the degradation process. These studies have led to the development of durability test methods which are more *Analytical* in nature and which reflect the scientific effort associated with the aerospace industry.

Aerospace/Defence Industries

It has often been observed that the application of adhesives to metal fabrication, in common with many other technical innovations, was pioneered by the aircraft industry. The same could probably be said of the application of adhesives to high performance polymer composite structures. The review begins with an overview of the types of applications, and the materials involved, for a range of applications of adhesives in the aerospace and defence industries.

A variety of durability procedures and test methods are used in an assessment of adhesives and substrates widely employed in the aerospace and defence sectors. These fall mainly into the category of *Standard/Routine* methods using long-term stressed or unstressed test coupons, exposed to realistic environmental conditions. Lap shear joints are of course very common but peel tests are also used. The realisation that test procedures which place a high stress concentration at, or near, the adhesive/adherend interface are very discriminating for joint durability assessment led to the use of more sophisticated stressed fracture mechanics-type joint specimens. Simple crack growth experiments (for example, the Boeing Wedge test) may be considered as *Standard/Routine* whilst more carefully controlled testing with double cantilever

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beams, perhaps undergoing cyclic stressing, may be regarded as *Developmental* or even *Analytical*. Tests using joints are often supplemented by information on bulk adhesive behaviour and a few *Analytical* approaches have sought to combine these facets within predictive models.

Construction Industry

A wide range of adhesive, mortar, sealant and substrate materials are encountered in building, and in civil and structural engineering. The number of applications, and associated demands placed upon bonded assemblies, is enormous and consequently there exist numerous test procedures. The main approach has been to simulate reality in terms of specimen size, extremes of loading, environmental regimes and long experimental timescales. Outdoor exposure is frequently used as a 'test environment'. Whilst the individual components of a bonded assembly are quite often tested separately, adhesion, and changes in adhesion as a function of environmental exposure, have not generally been investigated.

Substrate materials such as concrete or glass, which are brittle and weak in tension, impose restrictions on testing which limit suitable joint configurations mostly to compressive shear or flexure. At the other extreme, sealant materials take a very long time to fully cure and thus impose different considerations: for example, when should accelerated ageing begin or cyclic movement take place? A whole range of cold, hot, wet, thermal and imposed load/strain cycles are employed, together with thermal shock, thaw-freeze, sustained and transient loading cycles.

The test procedures adopted fall into the categories of both *Developmental and Standard/Routine* methods. Many **structural joints** are designed to be loaded in shear, so that the majority of test procedures employ shear loading. The main degrading factor with **repaired concrete** is the mechanical and thermal mismatch of parent concrete and repair materials, and there is little in the way of a co-ordinated approach to environmental durability testing. Tensile pull-off testing is commonly adopted for an assessment of short-term adhesion. **Sealant materials** are required to accommodate cyclic strain; only in the case of structural silicone glazing are they required to carry modest loads. Thus load/extension behaviour and failure mode represent important parameters to be collected from tensile adhesion and peel joint tests. A variety of very rigorous procedures have been defined by national and international bodies for assessing environmental durability and cyclic strain accommodation.

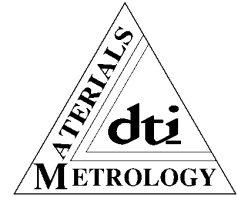
General Engineering

The range of materials and product assembly types in this category is very wide and, to provide a more definitive boundary for the survey, the investigation addressed machine tools, general machinery/plant engineering, transport, marine and off-shore requirements.

Within this interpretation of general engineering it is observed that a large proportion of applications of adhesives are characterised by bonded joints in relatively thick sections and heavy structures. Also production quantities are relatively low compared with most of the other industry sectors. In many products the bonded assemblies are used in relatively benign environments and adhesive joints can usually be specified on the basis of general performance

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data provided by the adhesive supplier. Most adhesive manufacturers derive their technical product information from *Routine/Standard* test methods which are complemented by accumulated user experiences.

For applications such as marine structures and critically-stressed products which are exposed to more demanding environments, the durability of the bonded joints becomes a primary parameter in the design specification. While the industry as a whole uses durability data obtained from 'traditional' test methods from related sectors, a number of special durability tests have been developed to meet a number of different needs. These *Developmental* techniques have been reviewed under the headings of adhesive-specific tests, application-specific tests and substrate-specific tests.

Some of these specific techniques offer potential for further development and wider application. In particular, several versions of adhesive bead test have been identified and there is some scope for refinement and rationalisation, which might provide a valuable standard durability test.

Packaging Industry

There are many sectors of the packaging industry, all of which depend, in some way, on adhesive bonding. This dependency may either be for the manufacture of the base structure of the packaging material or for the formation of the geometric shape of the package. Each sector has tended to develop its own set of procedures to assure the durability of the bond. Therefore, the methods are often specific and are tailored to a particular end-use. Many of the methods are comparative and pass/fail criteria are based on internal standards, defined from experience.

There is only a limited amount of published durability data available in the Packaging Industry. Most test data are proprietary, although adhesive manufacturers often provide illustrative examples of their products' suitability for particular applications.

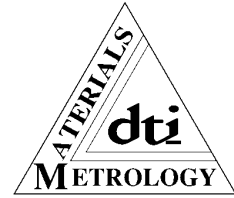
There are, therefore, opportunities for some unification of *Standard and Developmental test* methods throughout packaging. Such an initiative will help the mutual understanding of requirements for material properties and performance, and will hopefully define criteria for acceptability which can be employed throughout the industry. There is evidence that this is happening to a limited extent, with protocols being developed between expert bodies, research organisations and some parts of the industry. An extension of these activities, to develop a commonality of approach, throughout all sectors should help to provide improvements in packaging design and quality assurance.

Footwear Industry

The footwear industry has been using large quantities of adhesives for many years, in order to provide unobtrusive joints between a variety of flexible, dissimilar, materials. The most critical joint is between the sole and upper, although adhesives are used in a variety of ancillary operations. The review is based upon information obtained largely directly, or indirectly, through SATRA.

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There are several aspects of the footwear industry which set it apart from most of the other industries surveyed. Firstly, the volume production of pairs of footwear is enormous. Secondly, individual bonded articles are relatively small, enabling full-scale testing. Thirdly, the life-span of articles of footwear is measured in a few years or less, enabling useful feedback from wear trials and user experience. It therefore follows that environmental durability testing is not viewed as a priority over other types of testing. Indeed a very large number of test procedures exist for mimicking in-service demands such as flexing, toe-peeling and high temperature resistance. Parts of the real article as well as the whole article may be tested. Peel testing represents the main appropriate technique for assessing bonds between the flexible substrate materials involved.

Standard/Routine testing is carried out as a check on adhesive materials and for bonding new footwear materials; such peel tests are essentially acceptance tests. Peel and, to a lesser extent, shear joints are used within a suite of *Developmental* test methods which provide comparative information on different substrate, surface treatments, adhesive materials and the resistance of bonded assemblies to severe flexing, peeling and creep loads. Degradation is monitored in various environments over prescribed time intervals, the ageing and heat resistance parameters representing maxima from service conditions. Correlations are made between whole articles subjected to repeated laboratory flexing over long time periods, and between regular users of footwear such as postmen.

SATRA have developed a comprehensive range of test procedures and test equipment for experimental use. More recently substrate surface analysis has been used, in part, to investigate the mechanisms involved in degradation, together with scientific modelling and recording of the stresses imposed on the materials and joints between them. These methods, although not specifically geared towards environmental durability assessment, are *more Analytical* in nature and serve to underline the maturity of the industry.

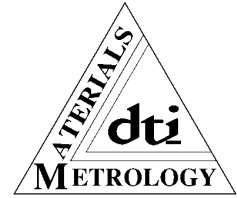
Electronic/Electrical Industries

In the electrical and electronic industries the most widely used method of assessing the performance of adhesive joints, whether for aged joints or simply at different test conditions, is some form of peel test (generally at 90°). The effects of temperature and humidity on the performance of adhesive joints is the primary concern. Durability of these joints when exposed to other conditions is sometimes necessary, depending on the particular application in question. For example, in space applications some joints may need to be able to withstand very large temperature changes. Environmental durability is generally seen to be less important. In addition to the requirement for the adhesive joint to remain intact under normal service conditions of the component/product, there is also often concern as to whether the components may be easily separated at a later date for reworking purposes.

There are consequently two different types of test programmes which are carried out for the assessment of the performance of the adhesive joints in service. Some are concerned with the durability of the product, when the joint is exposed for various periods of time to a selection of

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different ageing environments prior to testing; others are concerned with the performance of the joints under different test conditions. Durability tests which are carried out range from short- or long-term, and also from extreme to reasonably tolerable conditions. In some cases test programmes have been designed with specific applications in mind, whereby the processing conditions which may be experienced by the adhesives during product manufacture are inflicted on the joints. These may be interpreted as *Developmental* test methods, and may or may not also involve the use of *Standard/Routine* methods.

There does not appear to be any one particular preferred accelerated ageing regime, these depending to a great extent on the application. For instance, the effects of castor oil are considered in the case of materials used for encapsulants. Generally, also, the accelerated ageing regimes employed in the industry, are imposed over relatively short time scales, although some long-term effects have been investigated. On the other hand, there are more examples of extreme conditions being used than may be found in other industry sectors.

Timber Products Industries

The durability of bonded timber products is influenced by a number of different factors, the most important being water and temperature. The anisotropy of timber also complicates the picture, causing potentially large dimensional changes across the grain in bonded assemblies as the water content changes. The water content of the timber prior to bonding is also important, and this can have marked effects on the performance of the product.

The majority of the work carried out on bonded timber products is related to the performance of plywood and particleboard applications, since these products account for over 75% of the 180,000 tonnes of adhesives used annually in the UK. *Standard test* methods have been specially developed for these materials, and are widely used. Other *Standard test* methods for bonding directly to wood are also numerous.

Adhesive joints are exposed to a fairly wide range of accelerated ageing regimes in the wood bonding industry and the test methods employed are *Developmental* in nature. Where specific applications of an adhesive are concerned, the ageing conditions selected may reflect the conditions to which the joint will be exposed in service. The most commonly used environments for ageing of wood adhesives, however, involve variations in temperature and/or humidity. There are also a number of studies which have involved a comparison of the accelerated ageing effects on the joints with real-time ageing over long periods of time; these have generally shown that the results which are obtained after accelerated ageing are representative of the actual effects after many years. Accelerated ageing is therefore considered to be very useful for estimating the life of a joint.