

**Project PAJ3 - Combined Cyclic Loading
and Hostile Environments 1996-1999**

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Project PAJ3: Final Report

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ABSTRACT

Project PAJ3 *Combined Cyclic Loading and Hostile Environments* was one of the three projects in the Materials Measurement programme *Performance of Adhesive Joints (1996-1999)*. This report summarises the achievements of the project made in the six technical tasks.

1. Review of test methods, statistical methods and analytical models;
2. Test method development;
3. Data analysis and modelling;
4. Failure analysis;
5. Design methodology;
6. Standardisation.

An integrated approach was adopted to design/analysis of adhesively bonded joints for combined mechanical loading and hot/wet environmental conditions. The approach included statistical analysis techniques, finite element analysis (FEA), analytical modelling, fractography, chemical analysis and mechanical testing. The project developed test and design methodologies suitable for both quality assurance and design purposes. Statistical analysis techniques such as design of experiments (DOE) has been successfully employed to assess data that was either produced in-house or sourced from industry and published results.

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Approved on behalf of Managing Director, NPL, by Dr C Lea,
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1. SCOPE OF THE PROJECT

Adhesive joints are expected to retain a significant proportion of their load bearing capacity for the entire duration of the service life of the bonded structure. However, service conditions can often involve exposure to combinations of static or fatigue load and hostile environments - marine, hot/wet, weathering or chemical. It is therefore essential that the end user and the adhesive manufacturer possess the necessary tools for selecting and characterising an adhesive system, to ensure reliable bond performance under hostile operating conditions that may be experienced for 25 to 50 years. Optimising joint performance requires an understanding of the failure mechanisms involved in environmental degradation, and validated test methods and design methodologies suitable for predicting material degradation and life expectancy.

The general consensus is that wider use of adhesive technology has been impeded by a lack of reliable test methods, accelerated ageing procedures, quantitative data and predictive analysis for determining durability of structural adhesive joints. To address these issues, the DTI have funded through the MTS programme on the Performance of Adhesive Joints, ADH, (1993-1996) and the subsequent Materials Measurement programme, PAJ, (1996-1999) two projects on durability of adhesive joints.

Project PAJ3 ***Combined Cyclic loading and Hostile Environments*** was one of three research projects making up the DTI Engineering Industries directorate (EID) Materials Measurement Programme 'Performance of Adhesive Joints'. The project concentrated on structural adhesives used in engineering applications for bonding secondary structures and to maximise long-term strength retention under hostile service conditions. Project PAJ3 has built on some of the work from the previous MTS project ADH3 [1] and has attempted to ensure, wherever possible, continuity in adherend/adhesive surface treatment systems in order to provide cross-referencing between the two projects. Structural adhesives were also evaluated in Project PAJ2 ***Dynamic Performance of Adhesive Joints***, whereas PAJ1 ***Failure criteria and their Application to Visco-elastic/Visco-Plastic Materials*** concentrated on flexible, visco-elastic adhesives used by the packaging and footwear industries.

The project consisted of six technical tasks:

1. Review of test methods, statistical methods and analytical models;
2. Test method development;
3. Data analysis and modelling;
4. Failure analysis;
5. Design methodology;
6. Standardisation.

Initially, there was a task covering project dissemination. This task was subsumed in the over-arching dissemination project. The dissemination results from PAJ3 will be included in a final report for PAJ0. Therefore details of dissemination are not covered in this report which focuses on the technical outputs from this project.

PAJ3 has produced 18 technical reports, a Measurement Good Practice Guide, a number of conference papers/journal articles, and a PhD transfer document on Mode II testing of adhesive joints. This report summarises the findings reported in these documents. The major technical outputs are listed below.

Main Technical Outputs of Project PAJ3

TASK 1: Review of Test Methods, Statistical Methods and Analytical Models

- Review of durability test methods and environmental conditioning procedures (including national and international standards) [1].
- Review of life prediction models and adhesive joint design and analysis software [2].
- Guide to the use of Design of Experiment (DOE) methods [3].

TASK 2: Test Method Development

- Evaluation of commercial DOE software programs [4].
- Installation of environmental chambers and development of auxiliary equipment for continuous use with mains water supply.
- Evaluation of environmental degradation of bonded joints and environmental conditioning methods [5-6].
- Improved insight into the effects of geometry, material properties and moisture on test results.
- Mechanical properties data measured for two adhesives at different levels of moisture content.
- Improved understanding of cyclic fatigue behaviour of bonded joints [7-8]. Determination of a possible relationship between fatigue stress and life expectancy.
- Assessment of creep testing of bonded joints under hostile environments [9-10].
- Developmental work on a mode II fracture toughness test method for structural adhesives; suitable for use with metal and composites. This work will continue until September 2000 (PhD project - Imperial College) [11].

TASK 3: Data Analysis and Modelling

- Demonstrated the use of statistical methods (including DOE techniques) for analysing durability data [12-13].
- Improved scheme was developed to enable the assessment of moisture uptake in adhesive joints using FEA [14].
- Evaluation of moisture, specimen geometry, and adherend and adhesive properties on joint behaviour. FEA was conducted on four joint configurations (single-lap (with and without perforations) [15], T-peel [16], tapered strap [7] and scarf joints [7]).

TASK 4: Failure Analysis

- Fractographic analysis of adhesive joints that have failed due to cyclic fatigue, creep rupture and/or environmental degradation [17].
- Chemical analysis of adhesives and surface treatments before and after environmental exposure.
- Glass-transition measurements for three adhesives at different levels of moisture content.
- Improved understanding of failure mechanisms due to the combined effects of mechanical loading and environmental conditioning.

TASK 5: Design Methodology

- Evaluation of durability data using DOE techniques - industrial case studies [18].
- A methodology for maximising information from an experimental programme whilst simultaneously minimising spurious non-experimental effects.
- Measurement Good Practice Guide - Durability Performance of Adhesive Joints [19].

TASK 6: Standardisation

- Three draft procedures (thick adherend shear test (TAST) by compression loading, tapered strap test and skin-doubler test) [20].
- Submitted Mode I fracture toughness test method protocol (Document 98/123967) to BSI for new standardisation work. The protocol was submitted on behalf of the European Structural Integrity Society (ESIS).
- Development of a mode II fracture toughness test method for structural adhesives; suitable for use with metal and composites (see Task 2).

2. REVIEW OF TEST METHODS, STATISTICAL METHODS AND ANALYTICAL MODELS

2.1 TEST METHODS AND STANDARDS

One of the main objectives of the project was to develop and validate test methods and environmental conditioning procedures that can be used to measure the parameters required for long-term performance predictions. To meet this objective it was necessary to establish the current status of durability test methods, associated standards and data generated. A range of test methods (e.g. tensile, shear and fracture toughness tests) and accelerated ageing methods/schemes were critically appraised and reported [1]. The review was based on information compiled from previous government research programmes, a survey of industrial practices and requirements, and published literature and standards.

The test methods were assessed in terms of material compatibility, data generated, environmental and service conditions, long-term performance, costs of implementation (in

relative terms), ease of use, consistency of data, data reduction requirements and stress uniformity. Consideration was given to the practicality of using the test method in an industrial environment, in terms of ensuring “fitness for purpose”. The results of the review contributed to the formulation of the subsequent experimental programme.

It was observed that the two most important criteria when selecting a test method were the availability of a standard test method and the ability of the test method to produce consistent and reliable engineering data for a range of service conditions. Most of the methods assessed had been standardised at either national or international level. The standards generally applied to standard laboratory conditions and were usually limited to providing comparative data on adhesive systems and surface treatments, and were unsuitable for generating design data. There was also the added concern that there was insufficient data of reliable pedigree required to determine the degree of uncertainty and the sensitivity of these techniques. In many cases, an improvement in specimen design would improve performance and versatility, reduce measurement uncertainty and allow the methods to be applied to a wider range of materials.

2.2 PREDICTIVE METHODOLOGIES

A number of predictive models were evaluated in order to identify potential methodologies suitable for predicting the residual strength of adhesively bonded joints subjected to mechanical (i.e. static and cyclic) loading and hostile environments [2]. Many of the life prediction methodologies were generic and not specifically developed for adhesively bonded structures. Due to the limited number of publications in the general literature, it was not possible to compare the different approaches in terms of advantages and disadvantages. Evidence was available to suggest that efforts, although limited, were being made to incorporate life prediction methodologies into design procedures.

The majority of temperature-moisture and temperature-moisture-stress superposition relationships adopted were found to have the form of an Arrhenius law. This was often rather by chance than by design. As with most of the predictive models considered, this approach was empirical (i.e. curve fitting to experimental data). Relating actual damage mechanisms, such as a reduction in cross-linkage density of polymers to strength reduction through hydrolysis is still in its infancy. Statistical life prediction models were found to be well developed and being used on a regular basis by engineers for lifing materials. Again, the analysis was empirical. In practice, there is often considerable scatter in failure data, which increases with exposure time. Hence, the data needs to be subjected to rigorous statistical analysis to ensure meaningful results.

2.3 NON-FEA DESIGN AND ANALYSIS SOFTWARE

A number of non-FEA software packages, specifically developed for adhesive selection or bonded joint design and analysis purposes, and suitable with personal computers (PC), were reviewed [2]. The packages were either commercially available or in-house products, currently being used for a wide range of applications. Most of the packages offered similar features for a limited number of joint geometries. Often differences were limited to the

number of joint geometries or to the loading/boundary conditions available. The more versatile packages provide a construction facility, which enables the user to produce non-standard variants of well known joint configurations. Although non-FEA software provides the user/designer with relatively cheap design tools, their versatility or capability of expansion to account for a wider range of joints or non-linear stress/strain behaviour is severely limited. The approach of using a pre-processor program, such as GLUEMAKER (TWI) [21], to generate and analyse FE models of adhesive joints is probably the best way forward. GLUEMAKER has considerable industrial potential, combining the advantages of simplified joint construction with a versatile Material database facility and the powerful FE solver capability of ABAQUS.

2.4 STATISTICAL METHODS

In the previous programme, a series of case studies using adhesive bonding were investigated using DOE techniques (MTS Adhesive Project: ***Measurement for Optimising Adhesives Processing***). These case studies combined laboratory measurements and process control to optimise manufacturing processes. As a result of this work, it was considered that DOE techniques could be used to establish the relationship between the main factors that affect joint performance under hostile environments. The initial work within PAJ3 was directed towards producing a guide to the use of DOE techniques [3]. The document provides information on the underlying principles of DOE, and guidance to aid users investigating possible alternative designs for data analysis. The document also includes a worked example of a manufacturing process and a summary of some of the available commercial software is also included.

3. TEST METHOD DEVELOPMENT

The work conducted in this task was directed towards the development and validation of test methods and environmental conditioning procedures. In addition, testing was carried out to provide supporting data for assessing the statistical analysis techniques and for generating input data for modelling joints using FEA. The work was divided into four areas:

- Statistical methods for durability data analysis
- Cyclic fatigue
- Creep testing
- Environmental conditioning (including accelerated test methods)

3.1 STATISTICAL METHODS

A survey of commercial DOE software programs revealed that there were large differences between software packages and that comparing the results was not straightforward [4]. It was found that the program specific organisation of the factors within the arrays in the software programs were different, requiring the user to be aware of the representation of the 'standard' arrays within the selected software package. This is particularly pertinent when cross referencing data from different software packages. Considering these differences, it was decided that the proprietary package developed by IBM called AGSS

(Advanced Graphical Statistical System) would be used on the basis that rigorous on-site validated test data was available and project team members were familiar with the use of the software.

One of the first sets of data to be examined was from a series of experiments carried out in-house using a perforated single-lap joint immersed in water at ambient and elevated temperatures. The statistical analysis was used to identify critical interactions and their magnitude. The analysis enabled the formulation of an empirical equation for predicting the residual strength as a function of the main factors (i.e. number of holes, conditioning temperature and exposure time) [4, 13].

3.2 CYCLIC FATIGUE

Three different test configurations (single-lap, tapered-strap and scarf joint) were experimentally evaluated to determine the suitability of the techniques for generating shear data under cyclic fatigue loading conditions. The resultant report provides guidance on specimen geometry, manufacture and testing [7].

Constant amplitude (sinusoidal waveform) fatigue tests were carried out on all three test configurations in load control with the stress ratio \mathbf{R} ($S_{\text{MIN}}/S_{\text{MAX}}$) equal to 0.1. Test frequency ranged from 5 to 25 Hz. It was found that the fatigue performance for each configuration could be represented by a linear-logarithmic (or by a power law) relationship:

$$P_{\text{MAX}} / P_0 = 1 - k \log_{10} Nf \quad (1)$$

where \mathbf{k} is the slope, \mathbf{Nf} is the number of cycles to failure, \mathbf{P}_{MAX} is the maximum load applied to the specimen, \mathbf{P}_0 is the ultimate strength of identically conditioned specimens measured at the fatigue test loading rate.

Single-lap and tapered-strap joints with similar overlap lengths (i.e. 12.5 mm) were less resistant to fatigue loading than the scarf joint configuration. The endurance limit (i.e. set at 10^7 cycles) occurs at far lower load values for the single-lap and tapered-strap joints in comparison with the scarf joint. Reducing peel stresses at the ends of the overlap was observed to extend the fatigue life of the bonded component.

The scarf joint exhibited excellent static performance, particularly under compression loading, and cyclic fatigue characteristics, thus demonstrating the effectiveness of this configuration from a design perspective. The enhanced performance was attributed to low peel and shear stresses at the ends of the overlap. The tapered-strap joint, although less resistant to cyclic tension-tension loading, offers the potential for monitoring shear deformation under cyclic loading conditions and is compatible with standard extensometry.

Joint stiffness for all three test configurations was observed to remain constant throughout almost the entire life-time of the joint with the onset of failure marked by a rapid reduction in joint stiffness and an increase in the loss or damping factor.

The work was extended to examining the combined effect of cyclic loading and environment (i.e. elevated temperature and heat/humidity) on the residual strength of adhesively bonded joints [8]. Double-lap shear, tapered-strap and scarf joint configurations were considered. Residual strength/endurance limit data generated within the programme and obtained from industry were assessed to determine any synergistic effects that may occur between cyclic loading and environmental agents (i.e. temperature and moisture).

Test data, supplied to the programme by Aerospatiale (courtesy of British Aerospace, Sowerby), from the ABHTA “*Adhesives Bonding for High Temperature Applications*” Brite-Euram Project BE-5104 were analysed to determine the endurance limit of hot/wet conditioned double-lap joints. This work involved the combined effect of cyclic loading and temperature of moisture pre-conditioned bonded composite and metallic joints bonded with either an epoxy or bismaleimide adhesives. As with the ambient results, a systematic approach can be used to determine intermediate residual strength or endurance limits, and to estimate knock-down factors for the individual actions or the combined effects of the degrading agents.

3.3 CREEP TESTING

A series of creep tests were conducted on bonded T-peel (or 180°) joints exposed to heat and humidity using self-stress (spring) loading fixtures in order to evaluate the test method and the loading fixture [9]. A number of tools were employed in data interpretation (FEA, statistical techniques, fractography, thermal analysis). The results clearly showed that under hot/wet conditions the ability of the joint to sustain load was severely reduced. A number of conclusions and recommendations were made in respect to the results obtained from the assessment of the T-peel joint, creep test results and self-stressing fixtures. It was recommended that ISO 11339 [22] should include the following modifications:

- An alignment/bonding fixture.
- 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, mode I fracture toughness or wedge cleavage tests are probably more reliable techniques than the T-peel joint for discriminating between various adherend/adhesive/ surface treatment combinations.

The test results indicate that for moisture sensitive adhesives it should be possible to relate the strength reduction of Tjoints joints with changes in moisture content, provided a suitable failure criteria could be found and that failure is cohesive. Self-stressing devices could be used in other allied applications (e.g. PMCs). Instrumentation for monitoring specimen failure, although adding substantially to costs, would improve 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.

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 instead of the current approach of measuring the lifetimes of the first three specimens as specified in ISO/DIS 14615 [23].

The work was extended to the examination of data sourced from industry and from the previous adhesives programme [10]. The evaluation of the results indicated that a systematic approach, albeit empirical, can be used to determine the time-to-failure for different combinations of test variables on different adherend/adhesive/surface treatment combinations. Simple relationships can be used for interpolation purposes to determine failure times for intermediate stress levels. The large uncertainty associated with creep test results, especially those obtained under hot/wet conditions, requires considerably more data points than currently being used to generate full creep rupture curves for design purposes. Further work is required to identify sources of experimental uncertainty (which are large) in order to improve the reliability of environmental creep data.

3.4 ENVIRONMENTAL TESTING

Environmental conditioning methods (including accelerated testing) for inducing moisture degradation within adhesively bonded joints were evaluated. The evaluation considered the relationship between the degree of degradation (strength retention) with the level of degrading agent and exposure time. Tests were conducted on bulk adhesive specimens and bonded joints that had been either immersed in water at elevated temperatures or exposed to humid environments at elevated temperatures. The bulk adhesive data was used as input data for the FE predictive modelling. The resultant reports [5-6] provide guidance on the single-lap specimen geometry, manufacture and testing, and on the validity of extrapolating data obtained from short-term accelerated tests to predicting long-term behaviour of larger bonded structures.

The results indicate that for adhesives that underwent substantial changes in material properties it is possible to relate the strength reduction of single-lap joints with changes in both T_g and the conditioning temperature, thus enabling strength values to be determined at intermediate temperatures. This applies equally to water immersion and exposure to hot humid environments.

The compression TAST specimen, which was developed within the programme, was found to be suitable for measuring the shear properties of bonded panels with a total thickness of 5 to 6 mm. The method is potentially suitable for rapid environmental conditioning. Smaller specimens, however, are more sensitive to environmental attack than larger joints due to the larger bond-edge-to-bond-area ratio and therefore give a more conservative estimate of environmental resistance. Extrapolation of short-term data from accelerated tests using small specimens needs to be considered with due caution. The

compression specimen, unlike the tensile specimen, is not particularly suited to cyclic fatigue testing.

The results from tests conducted on PMCs indicate that autoclave conditioning may be a viable option for inducing accelerated ageing, particularly for those systems possessing glass transition temperatures in excess of 120 °C to 140 °C. The technique may prove however, far too destructive for materials possessing a low **T_g** value, such as polyester resins or moisture sensitive adhesives. This accelerated ageing procedure warrants further consideration.

4. DATA ANALYSIS AND MODELLING

4.1 STATISTICAL ANALYSIS TECHNIQUES

The use of statistical analysis techniques was extended to analysing durability data from published research (see [12]) and from round-robin data from MTS Adhesive Project: ***Environmental Durability of Adhesive Joints*** [24]. The round-robin exercise involved five organisations nominally testing the same substrate/adhesive/surface treatment combinations. The analysis was able to successfully identify where there were significant differences due to surface treatments and differences between Test Houses [13]. The statistical work was carried out at the National Physical Laboratory and by Mr T Twine (Beta Technology) and Mr M Hall (Xyratex, Havant).

Statistical data analysis was also used to determine the correlation between joint strength data from long-term weathering and accelerated laboratory tests [13]. The data was supplied by the Defence Evaluation and Research Agency (DERA) at Farnborough [25]. It was shown how linear regression equations and correlation coefficients could be used to differentiate the significance, or quality, of the correlation between factors of interest “to the experimenter”, and to relate accelerated test data with long-term weathering results.

Fraction analysis was also carried out on durability data in order to demonstrate that it was possible to perform just a fraction of a planned experiment and gain almost as much information as a full factorial experiment [13].

4.2 FINITE ELEMENT ANALYSIS (FEA)

Previously, the moisture distribution within the adhesive layer was determined using analytical expressions and the association with the FE mesh was performed manually. This resulted in a tedious and time consuming model-building process, particularly when large models were involved. Moreover, the analytical solutions are a one dimensional approximation of the mass diffusion problem and induce errors when the geometry is irregular. A general scheme was developed that incorporates a transient FE technique, sequentially coupled with a mechanical analysis. This approach results in more realistic representations of the moisture concentration field within the adhesive layer, allowing for irregular geometries in three dimensions. The scheme eliminates the need for manual association of nodal concentration values. FE and analytical results for a one-dimensional diffusion problem were in excellent agreement.

This modelling work was extended to analysing the stress and strain distributions within the T-peel joint and the single-lap joint (with and without perforations). The effect of environmental conditioning on the joint performance was investigated using the sequentially coupled mechanical-diffusion finite element model, which incorporated continuously varying adhesive material properties. The numerical predictions revealed that the stress distributions become more uniform along the adhesive layer when the adhesive contains increased amounts of moisture. Peel stresses and shear stresses at the edge of the adhesive fillet in the T-joints and at the ends of overlap for the single-lap joint decrease with increasing moisture content. The introduction of holes in the overlap of single-lap joints was shown to accelerate moisture uptake, however failure is dominated by the stress state at the ends of the overlap.

Parametric studies on the T-peel specimen geometry revealed that stress distributions are sensitive to adherend material properties, adherend thickness and flange radius, the flange radius being the least significant. In general, stresses were reduced when changes in the T-peel geometry resulted in smaller joint displacements for the same load. The parametric studies on the single-lap geometry revealed that stress distributions were sensitive to changes in adherend material properties, adherend and adhesive thickness and the applied load. In general, stresses were reduced when changes resulted in smaller joint displacement or an increase in the ability of the adhesive layer to plastically deform.

Elastic-plastic FEA was also carried out on the scarf joint and tapered-strap joint to determine stress and strain distributions along the centre line of the adhesive layer. The results showed that for short overlap lengths the stress and strain distributions in the adhesive layer of the tapered-strap joint were relatively insensitive to the external taper angle on the bridging (i.e. straps) adherends. The shear stress distribution along the bondline of the scarf joint with a 30° scarf angle was relatively uniform, although peel stresses were also present. Reducing the scarf angle to less than 20° would significantly reduce the peel stress component, particularly at the ends of the overlap.

5. FAILURE ANALYSIS

A programme of work was carried out to characterise the combined effect of environmental exposure and mechanical loading on the microstructure of adhesives and adherend surfaces in joints [17]. Joints were examined both before and after testing to failure to assess the level of damage accumulation. Three analysis techniques: (i) scanning electron microscopy (SEM); (ii) X-ray photoelectron spectroscopy (XPS); and X-ray refraction were employed to examine whether physical/chemical changes in the appearance/structure of these surfaces, due to the effects of mechanical loading and/or environmental conditioning.

SEM and XPS techniques were able to detect physical and chemical changes, respectively, in those adherends and adhesives effected by the various surface treatments and environmental conditioning regimes used. The X-ray refraction technique used for

analysis of composite joints showed promise, but further meaningful quantitative analysis will require specialist jigs and instrumentation.

Dynamic mechanical thermal analysis (DMTA) measurements were carried out on moisture conditioned bulk adhesive specimens to determine the change in glass-transition temperature (**T_g**) as a function of moisture content. The maximum amount of moisture that can be absorbed by the adhesive, and hence the minimum **T_g** value attainable, was found to be temperature dependent. A sigmoidal (Boltzmann Equation) curve fit can be used to relate **T_g** with moisture content **M**.

$$T_g = \frac{A_1 - A_2}{1 + e^{(M - M_o)/dM}} + A_2 \quad (2)$$

where **A₁** is the initial **T_g** value, **A₂** is the final **T_g** value, **M_o** is the centre and **dM** is the width. The **T_g** value at the centre **M_o** is half way between the two limiting values **A₁** and **A₂**: **T_g(M_o) = (A₁ + A₂)/2.**

6. DESIGN METHODOLOGY

6.1 INDUSTRIAL CASE STUDIES

A number of case studies were supplied by industrial organisations within the United Kingdom for the purpose demonstrating the use of statistical methods in evaluating durability data generated in high humidity weathering conditions and accelerated laboratory tests. The analysis was more intensive than the previous efforts and employed a wide range of statistical analysis techniques (including DOE). Additional case studies utilising data specifically generated within the experimental programme were also analysed. A series of case study reports were produced by Mr T Twine (Beta Technology) which were compiled in a single report along with two case studies analysed at the National Physical Laboratory (NPL) [18]. Factors considered in the report included: (i) test method; (ii) surface treatment; (iii) adhesive type; (iv) processing variables (time, temperature and pressure); and (v) environmental conditions.

Statistical analysis was used to determine the effect of high humidity weathering conditions (i.e. 95% humidity with the temperature cycled between 42 °C and 48 °C) on the performance of zinc and organic coated steel joints bonded with a range of adhesives (polyurethane, acrylic and epoxy). The data, supplied by British Steel Plc, was for a 12 month exposure period. Data, supplied by British Aerospace (Sowerby), relating to the lap shear and thick adherend shear test (TAST) methods conducted on two adhesives over the temperature range of -67 °F (-55 °C) to 350 °F (177 °C) were compared in order to determine relative sensitivity and data variability for the two methods. A third case study examined data supplied to the programme by Alcan International Limited, in which a full factorial analysis is used to determine the optimum combination of processing variables for long-term durability performance.

The report concludes with two case studies based on durability data generated within the programme. The first demonstrates the use of a DOE approach to maximise information

obtained from an experiment, and simultaneously minimise the impact of spurious non-experimental, or “noise” factors, during the execution of the experiment. The case study concludes with an analysis of the results of the controlled experiment. The final case study considered the interaction of temperature and humidity on residual strength of titanium alloy joints bonded with an epoxy structural adhesive. Single-lap joints were exposed to nine combinations of temperature (3 levels) and humidity (3 levels). A full factorial analysis was carried out on the data to determine empirical relationships between the various factors of time, temperature and humidity.

6.2 MEASUREMENT GOOD PRACTICE GUIDE

A Measurement Good Practice Guide “Durability Testing of Adhesive Joints” [19] was produced, which covered the measurement of durability data for quality assurance and design purposes. The document provides guidance on selecting the appropriate test methods, data on interpretation and the use of the data for design of actual joints.

7. STANDARDISATION

Three draft procedures were produced [20]:

- (i) TAST geometry loaded in compression.
- (ii) Tapered-strap joint.
- (iii) Skin-doubler test.

Two of these methods: (i) TAST loaded in compression; and (ii) tapered strap joint can be used to characterise the shear behaviour of adhesive materials. These methods complement existing techniques by providing alternative solutions to the measurement of shear properties. The compression TAST specimen is a much smaller specimen than the standard tensile loaded specimen (ISO 11003-2) and is suitable for measuring the shear properties of bonded panels with a total thickness of 5 to 6 mm. The smaller specimen offers the potential for rapid environmental conditioning. The tapered strap joint is compatible for use with standard type extensometers, thus making it useful for measuring changes in stiffness for cyclic fatigue tests and for monitoring creep strain.

The overlap length of lap shear specimens is too short to ensure maximum shear transfer in the adhesive, thus the strength results from this geometry can be misleading. The “apparent” shear strength obtained will be dependent on adherend thickness and material, and failure will be invariably result from peel stresses rather than shear. The skin doubler geometry in contrast is more representative of real-life structures. The large bond length associated with the technique enables maximum load transfer from the skin to the doubler. The can be used to measure the ply drop strength for co-bonded and co-cured composites. Symmetric and non-symmetric specimen configurations are covered in the procedure. The technique can be used under combined hostile environments and cyclic loading conditions. This standard draft stems from work (commercially confidential) carried out within the AMCAPS (Affordable Manufacture of Composite Aircraft Primary Structures) programme, supported by the DTI CARAD programme with BAE SYSTEMS as prime partner.

Work continues at Imperial College into the development of a mode II fracture toughness test suitable for use with metals and composites. The work is expected to be completed in September 2000 (PhD project - Imperial College) [11]. Mode I fracture toughness test method protocol (Document 98/123967) was submitted by NPL, on behalf of the European Structural Integrity Society (ESIS), to BSI for consideration as a new standardisation work. The proposal has the support of the adhesives committee. Results from the PhD project have been included in the supportive data for this submission.

8. CONCLUDING REMARKS

The project developed test and design methodologies suitable for both quality assurance and design purposes. Statistical analysis (i.e. DOE) techniques developed within the project were successfully employed to assess data that was either produced in-house or sourced from industry and published results. A DOE scheme was developed in order to maximise information from an experiment, and simultaneously minimise the impact of spurious non-experimental, or “noise” factors, during the execution of the experiment.

A general scheme was developed that incorporates a transient FE technique, sequentially coupled with a mechanical analysis. This approach resulted in more realistic representations of the moisture concentration field within the adhesive layer, allowing for irregular geometries in three dimensions. The technique was successfully applied to T-peel and single-lap joint configurations in order to determine stress and strain distributions as a function of moisture content. Future modelling work hinges on the development of a suitable failure model for structural adhesives, which can account for moisture and various loading modes (i.e. creep and cyclic loading).

The results relating to cyclic fatigue performance of adhesive joints indicate that it should be possible to relate the life expectancy for different stress ratios to the joint geometry (i.e. ratio of shear to peel stress). Indications are that the generic joint configurations behave in a similar manner and that joints whose behaviour is dominated by peel stresses behave less favourably than those where shear stresses are dominant. The behaviour however, can be expected to be complicated by environmental factors (e.g. moisture).

It is clear that further work is required to address the critical issue of creep behaviour in adhesive joints. The causes of the large uncertainty associated with creep test results, especially those obtained under hot/wet conditions, need to be identified in order to improve the reliability of environmental creep data.

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