FATIGUE AND DURABILITY
OF
ADHESIVE JOINTS

20th April 2005

Society of Chemical Industry
15 Belgrave Square London

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Programme

10.00	Registration and Coffee.
10.30	The Fatigue Behaviour Of Structural Adhesive Joints Tony Kinloch, Imperial College London
11.05	Development of the RDCB Test for Assessing the Durability of Bonded Joints Mike Samulak, MERL
11.40	Ultrasonic Calibration Of Accelerated Aging of Silicone Sealant And Elucidation Of Degradation Mechanisms Greg Schueneman, Henkel Corporation, USA
12.15	The Corrosion Characteristics And Corrosion Protection Of Automotive Hybrid Hem Flange Joints: A Review Lisa Young, Ford Motor Company
12.50	Lunch
14.20	On the Need For Durability Testing Of Bonded Joints For Marine Structures Jan Weitzenboeck, Det Norske Veritas, Norway
14.55	Investigating The Evolution Of Fatigue Damage In Adhesively Bonded Joints Andy Crocombe, University of Surrey
15.30	The Effects Of Moisture On The Durability And Strength Retention Of Single Lap Joints Bob Adams, University of Bristol.
16.05	Discussion

Close and Coffee.

This one-day symposium is one of an ongoing series organised by the Society for Adhesion and Adhesives

The next symposium will be held at the SCI on Thursday 8th December 2005, and is entitled 'Bonding Composite Materials'.

For further details and booking queries please contact:

David Tod
QinetiQ
FST/X50
Fort Halstead
Sevenoaks
Kent
TN14 7BP

Telephone	01959 514492
Fax	01959 516014
Email	datod@qinetiq.com

THE FATIGUE BEHAVIOUR OF STRUCTURAL ADHESIVE JOINTS

A.J. Kinloch, H.Hadavinia, M.S.G. Little and A.C. Taylor

Department of Mechanical Engineering, Imperial College London, Exhibition Road, London SW7 2BX, UK

e-mail: a.kinloch@imperial.ac.uk

Introduction

A major concern when using structural adhesives is that the mechanical performance of adhesive joints involving metallic or ceramic substrates may deteriorate upon being exposed to aqueous environments [1-6]. Further, previous research has clearly revealed that it is the interphase of the joint, i.e. the region adjacent to the interface between the substrate and the polymeric adhesive, which is susceptible to such attack and on which attention must be focussed. Also, a common type of mechanical loading encountered by aerospace structures, especially adhesively-bonded components, is cyclic-fatigue loading. For most materials, the presence of this type of loading is found to lead to a much lower resistance to crack growth than under monotonic loading, and polymeric adhesives are no exception to this observation. Thus, as would be expected, the combination of an aqueous environment and cyclic-fatigue loading is a severe test for any adhesive system.

Hence, in the present study the cyclic-fatigue behaviour of adhesive joints has been investigated, and an assessment has been made of the environmental performance and durability of the joints under such loading conditions. A typical epoxy-film adhesive, as used by the aerospace industry, has been employed. The fracture energy, G_c, of the adhesive is 1700 J/m^2. In particular, the effect of using various surface pretreatments has been studied, namely: (i) grit-blasted and degreased (GBD); (ii) chromic-acid etched (CAE), (iii) phosphoric-acid anodised (PAA); and (iv) phosphoric-acid anodised with a typical aerospace formulated-primer also applied (PAAP). Fracture-mechanics tests have been used to obtain the relationship between the rate of fatigue crack growth per cycle, da/dN, and the maximum strain-energy release-rate, G_{max}, applied during the fatigue cycle. These cyclic-fatigue tests have been conducted in both a 'dry' environment of 23±1°C and 55 % relative humidity and a 'wet' environment of immersion in distilled water at 28±1°C. X-ray photoelectron spectroscopy and electron microscopy techniques have been used to identify the locus of joint failure and the mechanisms of environmental attack.

Mechanisms of Failure

It is evident that the fracture-mechanics approach provides an excellent method for evaluating the effects of the different surface pretreatments on the durability of the adhesively-bonded joints. In particular, the combination of cyclic-fatigue loading and the presence of an aqueous environment leads to an assessment of the environmental resistance of the bonded joint within a matter of weeks, as opposed to the more typical accelerated ageing tests which involve exposing the joint, unstressed, in water for many months. Also, in such unstressed tests, the water temperature is often relatively high, well above any likely service-temperature, in order to try to produce a large accelerated-ageing factor. This frequently leads to unrepresentative failure mechanisms being observed in these tests, and very misleading results being obtained. Clearly, the present fracture-mechanics approach also circumvents such problems, since the temperature of the water bath is only 28±1°C. Further, the 'wet' cyclic-fatigue tests revealed the presence of a threshold value for the strain-energy release-rate, G_{th}, below which crack growth was found not to occur. The value of G_{th} provides a quantitative measure for the effectiveness of a given surface pretreatment in an aqueous environment.

X-ray photoelectron spectroscopy and electron microscopy techniques have been used to identify the mechanisms of environmental attack. It has been shown that the role of the interphase is crucial in determining the durability of the joints. Indeed, the joints which experienced the poorest durability (as evidenced by significant decreases in the value of G_{th} when a 'wet' as opposed to a 'dry' test environment was used for the cyclic fatigue test), i.e. the GBD- and PAA-pretreated joints, failed in the adhesive/ oxide interphase.

For the GBD-pretreated joints, the locus of joint failure arising from the 'wet' cyclic-fatigue loading was exactly along the adhesive/oxide interface. For these GBD-pretreated joints, a relatively high-degree of surface contamination and macro-surface roughness leads to (i) poor wetting and spreading of the adhesive and (ii) the presence of many large macro-voids at the interface, as has also been reported previously [7]. These macro-voids will allow the relatively rapid ingress of water and enable pockets of water to be developed along the interface. Further, such voids will create relatively high stress concentrations at the interface. These factors, coupled with the thermodynamic work of adhesion becoming negative for the interface (and hence the interface being intrinsically unstable) when exposed to water [1], lead to the interfaces of these joints failing at a very low value of G_{th} of 25 J/m^2 in the 'wet' cyclic-fatigue tests.

For the PAA-pretreated joints, failure arising from the 'wet' cyclic-fatigue loading was through the oxide layer. Cross-sections of the interphase region, observed using transmission electron microscopy, revealed that the mechanism of environmental failure was a weakening and failure of the oxide layer, and this failure mechanism was accompanied by a relatively low value of G_{th} of 50 J/m^2. The evidence suggests that this weakening results from a subtle hydration of the uppermost regions of the oxide layer. This conclusion is in agreement with the results from a recent study [8] which employed electrochemical impedance spectroscopy to detect hydration, albeit on a larger scale than in the present work, on an aluminium substrate under an unconstrained epoxy coating. In the present work, the reason why the oxide is susceptible to such attack by ingressing moisture appears to be due to the inability of the high-viscosity adhesive to penetrate into the relatively deep porous microstructure of the PAA-generated oxide. This leads to unfilled pores which act as sites for water molecules to aggregate, which then attack and weaken the oxide.

Indeed, if a low-viscosity primer is applied prior to bonding (i.e. the PAAP-pretreated joints), then complete penetration of the oxide layer now occurs [9]. Thus, water cannot now accumulate in unfilled pores and no hydration of the oxide is observed. Further, a 'micro-composite' interphase is formed between the underlying aluminium-alloy substrate and the adhesive layer. This results in a greatly increased surface area for interfacial bonding, compared to a planar interface. Also, the formation of the 'micro-composite' interphase will tend to reduce the local stress-concentrations, since this region will possess an intermediate modulus between that of the relatively low-modulus polymeric-adhesive and high-modulus aluminium-alloy substrate. Hence, an interphase with a graded stiffness, with respect to the various layers, has now been created. Finally, from the aspect of possible surface chemistry effects, the primer does contain phenolic- and silane-based additives which may increase the adhesion to the oxide surface and may help inhibit hydration of the oxide layer. Thus, for the PAAP-pretreated joints, these various factors lead to a value of G_{th} being ascertained from the 'wet' cyclic-fatigue tests which is very similar to that obtained from the 'dry' fatigue tests, namely about 240 J/m^2.

For the CAE-pretreated joints, no failure of the interphase is observed from the 'wet' fatigue tests, and no decrease in the value of G_{th} is seen compared to the 'dry' fatigue tests. In these joints it appears that the less deep and more open, whisker-like, microstructure of the CAE-generated oxide allows the adhesive to penetrate to give a well-wetted interface with the formation of a 'micro-composite' interphase, albeit thinner in depth than for the PAAP-pretreated joints. Again, as would be expected, no hydration of the oxide is observed from studying cross-sections of the interphase after 'wet' fatigue testing. Hence, it would appear that basically the benefits which result from the formation of a 'micro-composite' interphase, and have been discussed above for the PAAP-pretreated joints, are also responsible for the good durability for the CAE-pretreated joints.

Finally, the fatigue data which is generated may also be used to predict the lifetime of bonded joints and components exposed to various environments under cyclic-fatigue loading [5,6,10,11].

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Development of the RDCB Test for Assessing the Durability of Bonded Joints

***M Samulak**, J Harris, C Harrison*
Materials Engineering Research Laboratory Ltd. Tamworth Road, Hertford, UK SG13 7DG
www.merl-ltd.co.uk.

1 Introduction

At present, Standard test methods for adhesive systems typically measure short-term properties and form a useful guide to the qualitative ranking of different systems. The aims of two recent research programmes, supported by the automotive sector and the European Commission, via its 5th Framework research programme, were to develop and Standardise an accelerated environmental durability test that would better the understanding of the long-term performance of typical bonded systems. Joints would be subjected to both cyclic fatigue loading and an environmental climate cycle, to represent typical service conditions for bonded automotive components. Quantitative, geometry independent, data would also be generated that could be used for subsequent design analysis, using fracture mechanics techniques.

This paper covers selective aspects of the full development of the durability test. The test is based on a fracture mechanics approach applied to the reinforced double cantilever beam (RDCB) test piece geometry, shown in Figure 1. Development of manufacturing and testing procedures will be discussed, together with the test results for a variety of adhesive systems. Additional results from a recently performed European Round Robin test programme will be shown during the lecture. The use of statistical analysis to help interpret the accuracy of these results will also be shown.

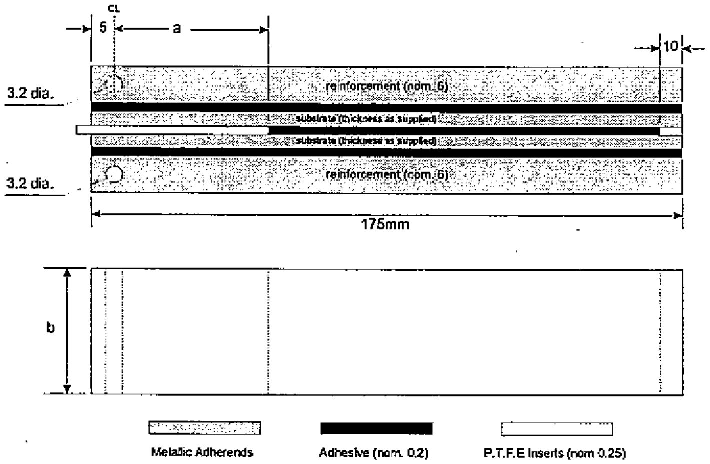


Figure 1 The RDCB test piece used for fatigue testing

2 Summary of the RDCB Test Methodology

The developed test method involves the cyclic tension loading of the RDCB test piece to induce mode I stresses at the crack tip and promote crack propagation. Based on the peak cyclic stored energy, the strain energy release rate (G_{Imax}) can be calculated at any given fatigue cycle. Having determined the relationship between test piece compliance (C) and crack length (a) – from a compliance calibration test – the crack length at any given cycle can be calculated. This technique for determining crack length is efficient and practical, since the use of optical measurements during climate testing is often difficult.

From the measured test data, a plot of G_{Imax} versus crack growth rate (da/dN) is constructed that defines the fatigue resistance of the adhesive system, shown schematically in Figure 2-2. A Paris Law fit is applied to these data, and represents the crack growth model for the adhesive system. A threshold G value (G_{th}) can be measured, under which only negligible crack growth would be expected. If the measurement of G_{th} is not possible (for practical reasons) projected 'thresholds' $G|_{10\text{-}6}$ and $G|_{10\text{-}7}$ can be calculated from extrapolation of the Paris Law fit, shown in Figure 2-2. The crack growth model and the threshold G value can be used during subsequent design analysis of service joints, since these results are geometry independent. Fatigue testing is performed at 23°C 50% RH ('dry') and 6Hz.

Once the room temperature fatigue resistance has been measured, the effect of environmental conditions on crack growth behaviour can be assessed. Firstly, fatigue testing is performed at 40°C 'dry' to determine the effects of elevated temperature only. After around 0.5Mc, a climate cycle is introduced to determine its effect.

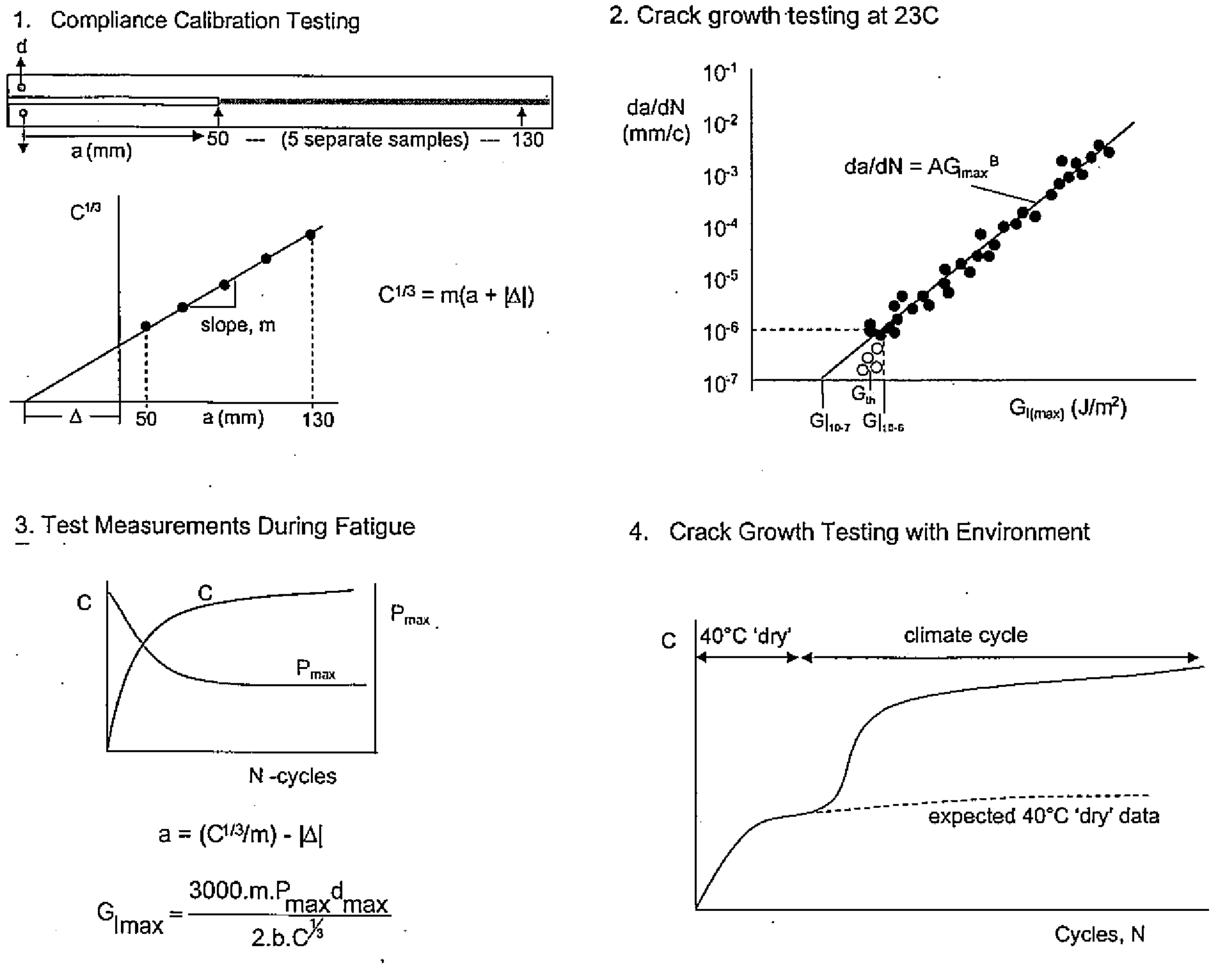


Figure 2 Schematic showing the applied methodology of the RDCB fatigue test

Climate Cycle Definition – loops continuously

- 15 minutes salt fog (5% NaCl, pH 7)
- 105 minutes drying at 40°C and 45% RH
- 22 hours at 40°C and RH 90%

3 RDCB Test Piece Manufacture

Adhesive System Definitions

System A	2mm extruded aluminium substrate (alkaline etched), adhesive A.
System B	0.8mm electro-galvanised steel substrates (de-greased/re-greased), adhesive B.
System C	1.2mm sheet aluminium substrate (Alodine finish), adhesive C.
System D	0.8mm hot dip galvanised steel (de-greased/re-greased), adhesive D

Reinforcement beams are all 6mm thick and used either 7000 series aluminium (systems A and C) or mild steel (systems B and D).

Test Piece Preparations

For efficiency, a 4-test piece plaque (175mm x 110mm) is assembled and heat cured in a hot-press to form all 3 bonds simultaneously. All bondlines are controlled to a nominal thickness of 0.2mm using P.T.F.E. inserts placed around the edges of the plaque. The starter crack length of the substrate-substrate test bond is controlled using a P.T.F.E. mask. After curing at the specified manufacturers times, the plaques are allowed to cool prior to precision cutting of the RDCBs, using a water-cooled abrasive disc cutter. Variation within the test piece widths is controlled to ±0.2mm.

4 Development of Test Equipment

At the start of the project, a dedicated environmental fatigue test machine was developed which can test 2 different adhesive systems (in triplicate) simultaneously. A servo-hydraulic test machine was designed and built by MERL, with an accompanying environmental conditioning unit (ECU), supplied externally, shown in Figure 3. An accuracy study and over 200Mc of testing has shown the design of the test equipment to be robust.

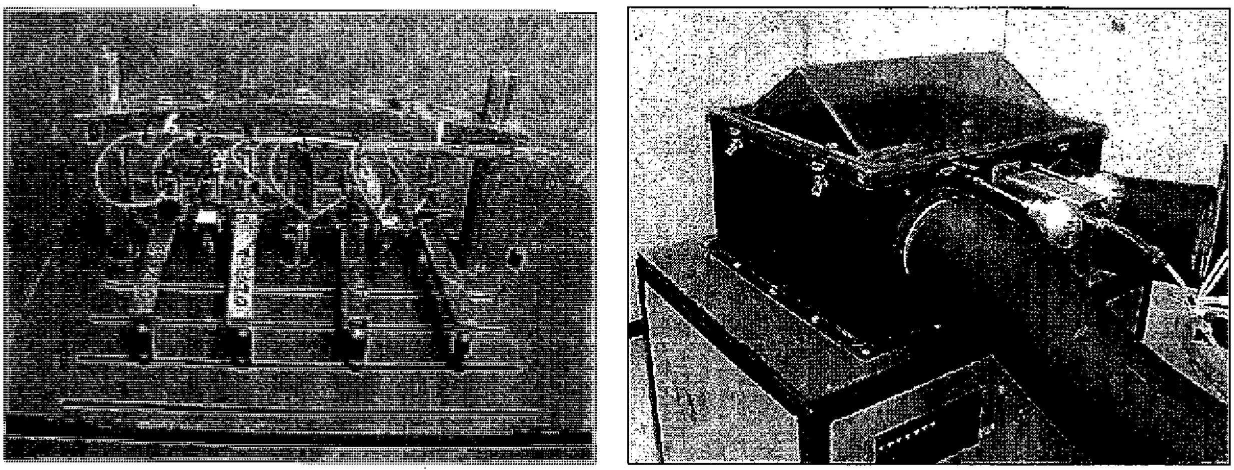


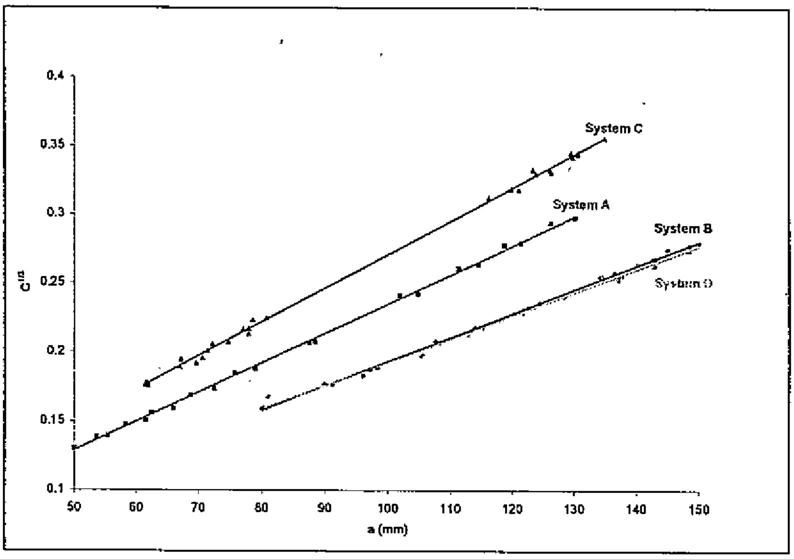
Figure 3 Custom built multi-station fatigue machine with ECU chamber fitted (right)

5 Test Results

Selective results from the test work are shown below. These results help highlight the sensitivity of the developed test method in assessing the environmental durability of different adhesive systems.

5.1 Compliance Calibration Testing

Compliance measurements were taken at different, measured, crack lengths for each of the 4 adhesive systems. These data are used to construct the graph shown in Figure 4, and yield m and Δ values, also shown. The different metal types give significantly different calibration constants, due to inherent modulus properties. The two aluminium systems (A and C) also yield different values, due to their differences in thickness. The two steel systems (B and D) show very similar results, as expected, since the thickness of the substrate is the same for both steel types.



System	m	|Δ|
A	0.00212	10.75
B	0.00174	11.06
C	0.00244	10.95
D	0.00169	14.25

Figure 4 Compliance calibration test results and constants m and Δ (right)

5.2 *Crack Growth Testing at 23°C 'dry'*

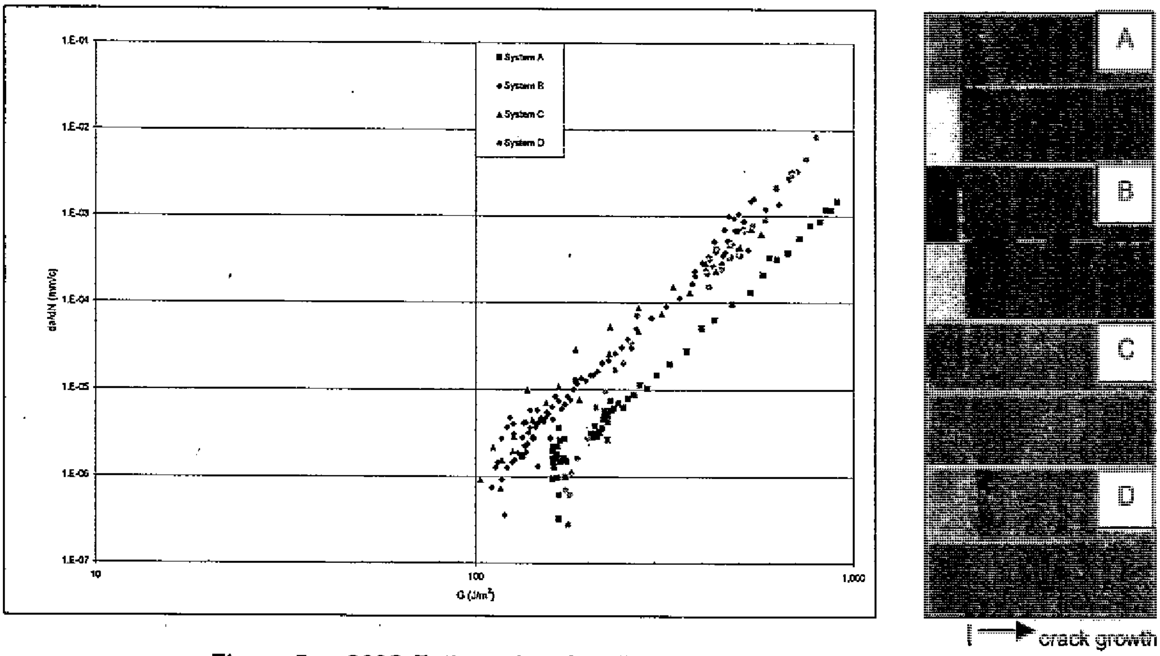


Figure 5 23°C Fatigue data for all systems with fracture surfaces (right)

Fatigue testing produced repeatable results for each system (typical data are shown in Figure 5) with scatter levels comparable to those found from other types of fatigue crack growth testing. Cohesive failure is observed within the adhesive, shown right. From all of the test data, values of G threshold (G_{th}) are estimated at 165, 110, 100, and 165 J/m^2 for systems A, B, C, and D respectively. Although all systems show similar fatigue behaviour, system A appears to display the highest fatigue resistance at room temperature.

5.3 *Crack Growth Testing with Environment*

Testing at, firstly, elevated temperature (40°C) and then with the application of the climate cycle (defined in section 2) has had significant effects on the fatigue performance of the different adhesive systems. These effects are summarised in Table 1.

Table 1 Summary of environmental effects on fatigue crack growth behaviour

System	Effect of 40°C 'dry'	Effect of climate cycle
A	Slight weakening, failure closer to the aluminium	No further reduction in fatigue resistance, locus of failure unchanged, compared to 40°C 'dry' condition
B	Significant weakening with locus of failure very close to steel substrate	Near immediate reduction in fatigue resistance with clean interfacial failure mode. Increased scatter in data as a result of this locus of failure
C	No effect	No effect, cohesive failure remains
D	Significant weakening with locus of failure very close to steel substrate	After 1 day of commencing the climate cycle, a further reduction in fatigue resistance is seen with clean interfacial failure mode. Increased scatter in data as a result of this locus of failure

Test piece compliance changes for system C show a gradual increase over the entire test. The resultant fatigue resistance plot (Figure 6) reveals that neither increasing the temperature nor applying the climate cycle affects the fatigue behaviour of this adhesive system. System D shows lower fatigue resistance under 40°C 'dry' conditions, however, cohesive failure remains (Figure 7, right plate - Dry). Environmental breakdown occurs in system D after about 1.2Mc (0.6Mc after the start of the climate cycle) and interfacial failure is observed, leading to much lower fatigue resistance, shown in Figure 6.

A similar result is seen for system B, however, interfacial breakdown occurs almost immediately on application of the climate cycle.

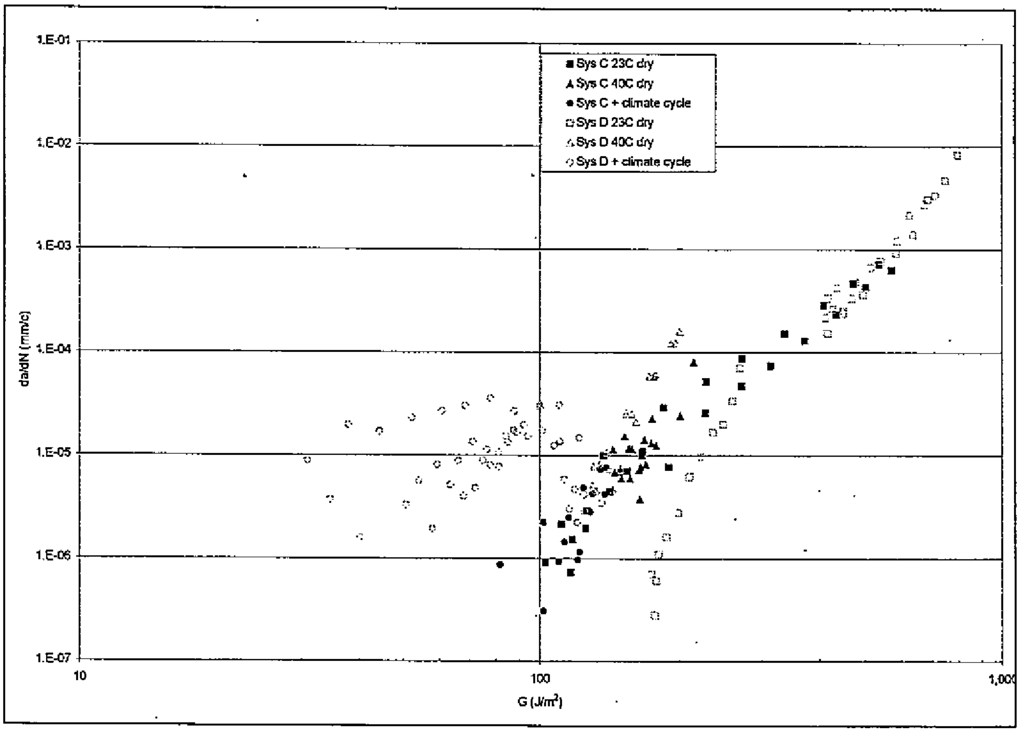


Figure 6 Fatigue resistance plot for systems C and D from environmental testing

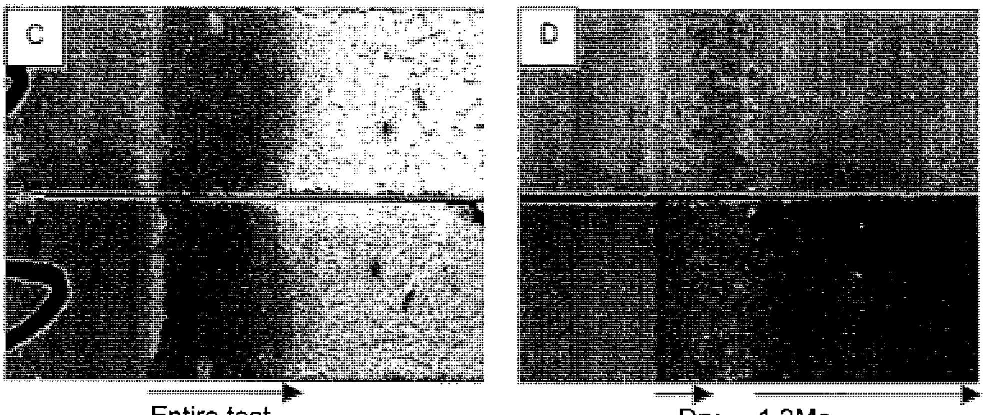


Figure 7 Fracture surfaces of system C and D specimens after environmental fatigue testing

5.4 *Round Robin Fatigue Testing at 23°C 'dry'*

During the last 3 years an EC 5th framework project (LTD-BAMS) has been used to further develop the Standardisation of the RDCB test methods. During this period a parametric study has been performed to determine the robustness of the test methods to test piece manufacturing and testing variables. An initial, project partner led, Round Robin study was performed to determine the robustness of both manufacturing and test procedures. A second Round Robin testing study was then performed, using external test laboratories – unfamiliar with the test methods, to determine the precision levels within all measured results.

From this research work a draft CEN Standard, *Structural adhesives – Test Methods for Assessing Long Term Durability of Bonded Metallic Structures*, has been submitted to the CEN /TC 193, "Adhesives" WG 2 committee. If successful the Standard will be released at the end of 2005.

The Round Robin test programmes included adhesive system 'A', as used above. Testing throughout the second Round Robin test programme has provided reproducible data, with good inter-laboratory repeatability. Due to project partner confidentiality agreements, no data can be shown here in print, however, slides will be presented at the lecture.

6 Conclusions

The purpose built test equipment has performed well throughout the project, completing over 200Mc without significant problems. The ECU has also proved robust, performing the corrosion cycle consistently during all tests.

Continual improvements to working procedures have led to the repeatable manufacture of high quality test pieces.

The compliance calibration method has been shown as a very robust technique for determining crack lengths, when optical measurement would be impractical.

Repeatable fatigue data have been generated at ambient conditions for all four different adhesive systems with the determination of respective threshold G values. These, geometry independent, quantitative values can be used for subsequent design analysis. Consistent cohesive failure within the adhesive layers are observed for all systems.

Elevating the test temperature to 40°C, alone, lowers the fatigue resistance of systems A, B and D, by varying amounts, and moves the locus of failure closer to the substrate surface. System C is unaffected by the increased test temperature.

The use of a corrosive climate cycle, representative of automotive service conditions, has been successful in assessing the fatigue (crack growth) performance of each system. Differences between the aluminium based systems and the steel systems have been highlighted. Substrate types and surface treatments, and adhesive types will also contribute to system performance.

From all of the performed tests it can be concluded that the test equipment and the test methods developed in this project to assess bond durability have proved very successful. Environmental crack tests have shown each of the four systems behave in markedly different ways to one another.

Further Round Robin studies on system A have shown that the developed test procedures and draft Standard have proved successful in helping to produce repeatable results across 9 different European test laboratories. Statistical analyses of these data has shown that a high level of precision can be obtained using this test method.

7 Acknowledgements

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Ultrasonic Calibration of Accelerated Aging of Silicone Sealant and Elucidation of Degradation Processes

Gregory T. Schueneman, Ph.D.
Henkel Corporation
1001 Trout Brook Crossing
Rocky Hill, CT 06067
greg.schueneman@us.henkel.com

The goal of developing an accelerated test to evaluate engine gasket durability and predict service life resulted in a sealant key life test that degrades silicone sealants most severely with exposure to synthetic motor oil at 140°C, with aeration and regular changing. Removing or lessening any of these conditions greatly reduces the resultant degradation. This observation reveals that the degradation process(es) are thermally activated and chemically accelerated. This presentation introduces methods that quantify changes in the mechanical properties and chemical state of silicone sealant specimens exposed to accelerated testing and those harvested from vehicle engines. The consequence of obtaining these two data sets is the calibration of accelerated testing and a relation between exposure time and engine miles.

The Corrosion Characteristics and Corrosion Protection of Automotive Hybrid Hem Flange Joints: A Review

Lisa Young [1 & 2], Jeff Kapp [2], Frank Walsh [1] and Julian Wharton [1].

The Anatomy of a Hem Flange Joint

Automotive closures such as doors, hoods and tailgates are made up of an outer panel, an inner panel, reinforcements, hinges and other fixings. In a conventional hem the steel outer panel is formed around the steel inner panel to form a hem flange joint as below in Figure 1.

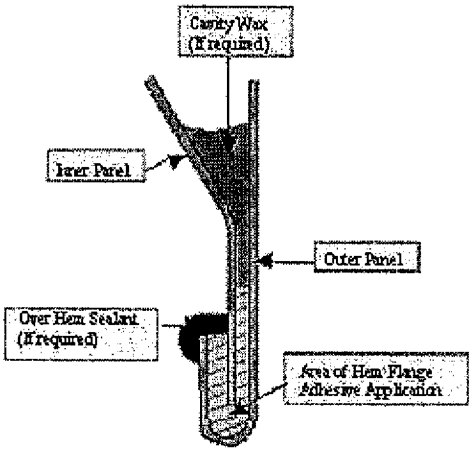

Figure 1. A section of a typical hem flange joint in a closure.

It is common for automotive manufacturers to apply a hem flange adhesive to the clinch area before assembly. Depending on the size of the closure and the manufacturing method a one or two part adhesive could be applied, this could be epoxy or butyl based. The adhesive can receive an initial pre-cure to give handling strength during assembly and to improve adhesion and reaches its full strength after heat curing in the paint ovens.

Applying adhesive to the hem flange offers benefits in the noise, vibration and harshness (NVH) performance of the vehicle by increasing the stiffness of the closure. Adhesive application also gives benefits in fatigue/durability properties of the joint and improved corrosion resistance. Additional corrosion prevention measures such as cavity wax and over hem sealer act with the vehicle paint system and hem flange adhesive to reduce water ingress into the joint.

[1] University of Southampton, UK. [2] Ford Motor Company Limited, UK.

Corrosion Characteristics of a Typical All Steel Hem Flange

A conventional hem formed in a sheet steel closure is called a flat hem as the metal is formed into a 180° hem. This gives very small gaps that would form a crevice if unprotected.

A general definition of crevice corrosion is given by Trethewey and Chamberlain [1]: as the attack, which occurs because part of the metal surface is in a shielded or restricted environment, compared to the rest of the metal which is exposed to a large volume of electrolyte. Fontana and Green [2] describe crevice corrosion as an autocatalytic reaction. Dissolved oxygen required for the cathodic reaction to take place (1) is in short supply in the crevice. Aggressive chloride ions are drawn from the bulk electrolyte (available from de-icing salts and coastal atmospheres) to balance positive metal ions from the anodic reaction (2). Metal and chloride ions react with water as in (3) and the hydrogen ions generated then accelerate metal dissolution further.

$$2H_2O + O_2 + 4e^- \rightarrow 4OH^- \qquad (1)$$
$$M \rightarrow M^{z+} + ze^- \qquad (2)$$
$$M^{z+} + H_2O \rightarrow MOH^{(z-1)} + H^+ \qquad (3)$$

In conventional zinc coated steel hems the zinc acts as a sacrificial layer and will corrode preferentially to the steel, since zinc is anodic to steel in most environments seen by a hem flange. Cut edges where the zinc is unable to protect the steel are subject to galvanic corrosion.

The combination of zinc/steel galvanic coupling at the cut edge, crevice geometry and other factors such as: moisture, salt, extremes of temperature and humidity, make the hem flange joint a very challenging system to protect from corrosion. Davidson et al [3] have investigated the influence of hem design and wet/dry ratio on the corrosion rate of simulated hems. This paper will review some of the research on this joint geometry, the influence of environmental factors and material variables.

Hybrid Hem Flanges

Although a conventional hem could be viewed as a hybrid system due to the extensive range of materials used. In this paper hybrid refers to a mixed sheet metal hem flange joint, specifically an aluminium/zinc coated steel hem.

A system that electrically couples two different metals will experience some degree of galvanic corrosion. So, in order to minimise galvanic corrosion it is desirable to electrically isolate dissimilar metals from one another. This can be achieved by using organic paint layers and adhesive bond lines as insulators; however, not all adhesives and organic systems provide full electrical insulation.

Organic paint layers can be applied to sheet metal before or after assembly, either as a roll applied coating at the sheet metal manufacturers or as conventional automotive paint systems. There is very little published research available on hybrid hems flange joints. Townsend [4] has investigated painted aluminium/steel couples for corrosion resistance.

Electrochemical convention recommends that in a galvanic couple the cathode should be coated to avoid galvanic corrosion. If the anode is coated and the cathode is left uncoated, any damage to the coating on the anode will result in unfavourable area ratio effects. Mansfeld and Kenkel have investigated these area ratio effects for aluminium/steel galvanic couples [5]. A damaged anode coating will mean that the current flowing from the cathode to the anode would be concentrated accelerating the corrosion rate. This may be sufficient to cause unacceptable corrosion within the service life of a vehicle.

A steel/organically coated aluminium hem system is being investigated for corrosion resistance. Laboratory electrochemical tests using a Zero Resistance Ammeter (as described by Jordan et al [6]) and correlation tests are underway to validate this technology. A robust steel/organically coated aluminium closure could provide sufficient corrosion resistance and feasibility in production. Organically coated aluminium, bonded to steel, could also give improved adhesive joint properties. A test program is running to evaluate the static and dynamic mechanical properties of single lap shear hybrid joints.

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Contacts

Lisa Young
Engineering Doctorate Centre, Department of Civil and Environmental Engineering,
University of Southampton, Highfield, Southampton, SO17 1BJ, UK.
lisay@soton.ac.ukor lyoung35@ford.com

Jeff Kapp
Materials Technology Centre, Ford Motor Company Limited, Dunton, Basildon, Essex, SS15 6EE, UK.
jkapp3@ford.com

Frank Walsh
Electrochemical Engineering Group, Materials Engineering Research, School of Engineering Sciences, Room 1001/1002, Building 30, University of Southampton, Highfield, Southampton SO17 1BJ, UK.
f.c.walsh@soton.ac.uk

Julian Wharton
Materials Research Group, School of Engineering Sciences, Room 4085, Lanchester Building, University of Southampton, Highfield, Southampton, SO17 1BJ, UK.
jaw6@soton.ac.uk

On the need for durability testing of bonded joints for marine structures

Jan Weitzenböck* and Dag McGeorge,
Det Norske Veritas AS, Veritasveien 1, 1322 Høvik, Norway

Introduction

There is a growing interest in adhesive bonding in shipbuilding, but there are still very few practical applications. Potentially interesting areas of application are bonded joints to replace bi-metallic joints between aluminium and steel, to compensate for large relative movements due to differences in thermal coefficient and for joining of aluminium parts.

The aim of this paper is to discuss the role of (durability) testing in classification and approval of ship structures and highlight the challenges that this approach creates for adhesive bonding. A possible solution is presented.

Classification and approval of ship structures

The management of safety at sea is based on a set of internationally accepted rules and codes, governing or guiding the design and operation of ships. The rules most directly concerned with human safety and protection of the environment are in general agreed internationally through the International Maritime Organization (IMO). Rules for the structural design and equipment required to make the ship "fit for purpose" are mainly established by independent classification societies. On safety-critical structural issues, the International Association of Classification Societies (IACS) provides common international standards. These rules have been developed incrementally over many decades, responding to accident experience, and represent a massive accumulation of expertise in how to allow designers as much freedom as possible while still achieving a good common level of safety.

Joints that contribute to the structural strength and integrity of essential parts of the ship's hull and its appendages need to comply with classification rules and are approved according to them. In traditional ship structure design for large SOLAS convention (Safety Of Life At Sea) type ships different grades of steel are being used. Welding is the dominating joining method. For fast ferries that follow the HSC code (Code of Safety for High Speed Craft) the material mix is more diverse but welding is still the most common joining method. The design, fabrication and repair of welded joints are very well documented. Local joint geometries have been standardised, designers have a thorough understanding of good joint design and how to design for long-term loading; they can refer to comprehensive design guidelines; quality assurance of the production processes is well understood; processes are qualified based on detailed qualification procedures and certified personnel is widely available. On this basis designers can design structures that meet with confidence the lifetimes stipulated by ship owners and operators although replacement of steel due to corrosion is not uncommon.

Failure criteria

The basis for qualifying an adhesive joint is a set of failure criteria (limit state equations) that represent all the critical failure mechanisms. These failure criteria contain at least one

* Jan.Weitzenboeck@dnv.com

characteristic strength parameter, other material properties, load effects and safety factors. One of the most relevant failure criteria for bonded joints is fracture due to a single extreme load. The failure criterion in terms of fracture load of joint samples: is $P_{fc}/\gamma_m > P_{max}$ where P_{fc} is a characteristic value of the measured load at fracture, γ_m is a partial material factor and P_{max} is the maximum predicted loading of the joint in response to environmental actions.

The maximum load effect P_{max} must be calculated taking due account of any partial factors that represent uncertainties in environmental loads and model uncertainties. The load effect P_{max} is output of an analysis of the response of the structure to the extreme loading events. The characteristic strength value P_{fc} should account for effects of the intended service environment (e.g. temperature, humidity etc.) may have on the capacity of the bonded joint. Thus the mechanical tests to obtain these values should include such effects.

Alternative approach for approval of bonded joints

Adhesive bonding is faced with a dilemma: there is little service experience to provide documentation of the long-term performance of bonded joints in a marine environment. Hence, it is not straightforward to demonstrate, say, 25 years lifetime for a new joint design. One therefore needs to find an alternative way for obtaining acceptance and approval of bonded joints. It is recommended to:

1. Use best practice in material selection, joint design and production technology. Best practice is used in materials selection to eliminate unsuitable combinations of adhesives and adherends and to limit the probability of severe long-term degradation. Best practice in production is used to establish robust processes with minimal variability. Furthermore it is used to ensure tight quality control of each production step as it is not possible today to check the quality of a finished joint non-destructively.
2. Ensure that the design allows detection of damage before ultimate failure: the joint is e.g. starting to leak or develops visible cracks. To ensure that joint failure or the onset of failure does not affect overall safety requires that the structure is designed with sufficient redundancy and reserve strength so that detectable damage in a joint is tolerable (at least until it can be repaired). One possibility is to introduce redundancy e.g. by interlocking joints.
3. Develop and demonstrate a repair procedure for the joints to be able to repair damage. These repair procedures are to be developed by the shipyard or subcontractor responsible for the bonding operation in consultation with the ship owner and e.g. the adhesive supplier.

This procedure reduces the consequences should the joint fail due to environmental degradation. However, in order to also make the probability of joint failure due to degradation as small as practically possible, the material selection process (item 1 above) should be based on results from environmental degradation tests. Considering the aim of, say, a 25 years lifetime, these tests should not only include the effects on bulk adhesive properties of plasticization and swelling due to diffused moisture. The tests should also provide for the possibility to detect any progressive physical or chemical degradation mechanisms that may attack the adhesive, primers and interfaces. This represents a challenge because the effects of plasticization and swelling of the adhesive can be significant and can mask other degradation mechanisms.

Investigating the Evolution of Fatigue Damage in Adhesively Bonded Joints.

Andrew D Crocombe,
School of Engineering, University of Surrey, Guildford, GU2 7XE, a.crocombe@surrey.ac.uk

1. Introduction

This paper gives an overview of the research carried out by the author and his co-workers over the past few years in the area of the fatigue life of adhesively bonded structures. Fatigue failure can be considered as the initiation and propagation of a fatigue crack. Initially the paper considers "whole life" approaches. This is followed by sections that consider fatigue crack propagation and finally the focus shifts to the evolution of fatigue damage and the crack initiation.

2. Whole Life Approaches

Work in this area generally tends to focus on the use of Miner's law and its extensions, following the traditional approach used for metals.

2.1 A simple design approach

Perhaps the simplest approach to quantifying the fatigue life of adhesive joints is to undertake tests at various levels of fatigue load and measure the life. This leads to the well known load-life (or S-N) curve. This has been done by a number of authors. If a fatigue limit is defined at a suitably large number of cycles, typically 10^6, it can be shown that a conservative lower limit for the fatigue load is around 20% of the static joint strength. Using this approach it is also possible to design a joint to carry a given fatigue load (P_f) for a finite fatigue life. This would be achieved by interpolating the SN curve to find the fraction of static load that can be sustained (f_{fl}) and then design the joint to have a static strength of P_f/f_{fl}

2.2 Load-life and stress-life laws

Unlike metals, fatigue testing of adhesive joints is seldom carried out for fully reversed loading (load ratio R=0.0). Conventionally a load ratio, R=0.1 is used for adhesive joints. Testing has been undertaken [1] to investigate the effect of load ratio (mean stress) on the fatigue response. It was found that for a given load range (ΔP) the fatigue life decreased with increasing R and that a Goodman curve can be used to define this effect. For cohesive failure of the adhesive it should be possible to express the fatigue response in terms of maximum adhesive stress rather than joint load. By considering the fatigue response of a number of different joint configurations, manufactured with the same adhesive and substrate materials, it was shown that a hydrostatic dependent stress measure was the most suitable

2.3 Variable Amplitude Fatigue loading

The fatigue data above has all been for constant amplitude loading. Typical in-service loading conditions are often far from constant amplitude. The simplest method for assessing these more complex periodic loading spectra is to group the load cycles into different amplitudes and calculate cumulative damage as the sum of the life fractions at each load amplitude. This has been assessed for a number of different loading spectra [2] and it was found that the load interactions significantly shortened the fatigue life (ie fatigue failure occurred at cumulative damages of considerably less than 1). It was further shown that by considering both mean stress ranges and overloads a cycle-mix damage predictive model gave good life predictions.

3. Fatigue crack propagation

Crack propagation in fatigue is most readily modelled using fracture mechanics, relating the energy release rate G to the crack growth rate through the Paris Law. For simple joint configurations it is possible to obtain analytical expressions for G which can then be combined with the Paris Law and integrated to give the crack length as a number of cycles. For more complex joint geometries it is necessary to obtain G using techniques such as finite element analysis (FEA). If FEA is used to find G it is a relatively simple step to also include it in the numerical integration of the Paris Law and hence obtain the variation of crack growth with cycles directly from the FEA. This is the approach generally adopted in the work outlined below.

3.1 Constant Amplitude

The approach outlined above has been applied to a number of joint configurations including single lap, double lap and lap strap joints with composite substrates of various lay-ups and steel [3]. It was found that using G_I generally gave closer predictions of the experimental data but use of G_T gave more conservative results.

3.2 Variable amplitude

The effect of variable amplitude, discussed in section 2.3 was extended to fatigue crack propagation [4]. The form of loading considered involved isolated overloads on an otherwise constant amplitude loading. Experimental studies were carried out on adhesively bonded and co-cured composite DCBs. When low levels of load were used the numerically integrated approach tended to underpredict the crack growth. This was attributed to the overloads causing damage, thus enabling the normal loads to propagate a crack more quickly. When the initial value of G was higher (around 2Gc/3) a sudden jump occurred in the experimental data, however predictions for higher crack growth were more consistent with the data. It is conjectured that the sudden jump occurred when the damage was significant and thereafter the crack was propagating in undamaged material.

3.3 Variable Frequency

Work by the author some years ago [5] revealed that for some adhesive systems the cyclic fatigue crack growth rate (da/dN) can increase significantly with decreasing test frequency. When the temporal crack growth rates (da/dt) were compared for these systems the frequency effect was much less evident. This, along with the increased damage zone, suggested that creep can play an important part. Later work [6] considered both steel and composite DCB specimens and investigated the effect of frequency over a two-decade range. Integration of the Paris law to determine crack propagation were extended to include variable frequency by using frequency dependent Paris law parameters and dividing the block loading into various constant frequency stages. Good agreement was found between the experimental and predicted variable amplitude fatigue crack propagation rates. It was also found that the fatigue threshold decreased with decreasing frequency.

4. Damage and Fatigue crack initiation

Although damage and fatigue crack initiation can often be considered together the field of continuum damage mechanics (CDM) can be used to assess fatigue life directly without considering the initiation and propagation phases. This is an aspect that will be addresses first, before progressing onto fatigue crack initiation and damage evolution.

4.1 Continuum Damage Mechanics and Fatigue Life

A thermodynamically consistent dissipation function was used to derive a cyclic damage rate (dD/dN) evolution law [7] in terms of the stress range experienced by the adhesive material. By integrating this until the fully damaged state it is possible to determine the fatigue life of the joint. The damage parameters were evaluated for unidirectional composite double lap joint data and then used to determine the S-N curves and thresholds (10^6 cycles) for other composite architectures and joint configurations. Good agreement was found between the measured and predicted threshold. This work has not considered the progress of damage through the joint and this aspect forms the remainder of the paper.

4.2 Backface strain

In order to investigate the evolution of fatigue damage in a non-destructive manner as technique known as backface strain measurement has been applied. Essentially, as damage occurs in the adhesive the load path through the joint changes and this results in changes in the substrate strain local to the region of damage. This is detected by a strain gauge placed on the "backface" of the substrate. This technique is an order of magnitude more sensitive than compliance changes and detailed analyses [8] have shown that the sensitivity can be further enhanced by appropriate positioning and size of gauge. Good correlation has been achieved between predicted damage and that revealed by microscopy carried out on the sectioned joint. Additionally, this method has been used to determine damage rate as a function of applied fatigue load.

4.3 Predictive algorithms for damage at both ends of the overlap

Correlating the fatigue damage with the backface strain output is made more complex by the fact that damage does not appear to occur at the same rate at both ends of the joint and the damage at one end effects the backface strain at both ends. To account for the coupled nature of the problem an optimisation algorithm was developed [9], based on surface fitting of the backface strain at both ends of the joint as a function of the extents of damage and the applied load. The algorithm was shown to give reliable measure of fatigue damage.

4.4 3D crack growth

When cycled at loads above the threshold the backface strain often increases slowly even at low cycles. It is possible that this could be due either to the growth a of thumbnail crack emanating from the joint mid-plane at the overlap end or from creep deformation of the adhesive at the overlap ends. To investigate the former, 3D FE modelling has been undertaken to assess the effect of the growth of a small thumbnail crack on the measured backface strain. By matching the predicted backface strain change with the experimentally measured response it was possible to identify possible crack growth scenarios [10]. With only a single backface strain being measured at either end of the joint it was not possible to identify the most actual crack growth scenario from all those that would match the backface strain output. This then has lead to measuring multiple backface strains at both ends of the joint, as discussed in the next section.

4.5 Enhanced damage measurement

To measure multiple backface strains it has been necessary to develop multi-channel, high speed datalogging hardware and software. This has been achieved using a NI DAQ card and LABVIEW [11]. A cyclic, damaged elasticity model has also been developed and integrated in the FE code ABAQUS. This will provide progressive failure analysis and enable the initiation and propagation phases to be combined in a single analysis. This progressive damage modelling methodology will be used in conjunction with the multiple backface strains in order to determine the specific damage mechanism active within the joint and the value of the damage model parameters. This will then be used to predict the damage and life in other joints manufactured using the same adhesive system.

5. Conclusions

A significant body of research into the fatigue response of adhesively bonded structures has been achieved. Traditional load-life approaches can be used to predict the response of such structures and extensions to cover varying mean stresses, and varying load amplitudes have been outlined. It was found that conventional cumulative damage laws could significantly over-predict the fatigue life of joints with overloads.

More insight is gained by adopting a more mechanistic approach. Crack propagation analysis can be undertaken for joints of any complexity by using FEA to determine energy release rates and implement the integration of the Paris Law. This methodology has also been extended to include the effect of varying load amplitudes and loading frequency. Complex load interactions were observed in the former but life predictions were generally reasonable.

The best understanding of the fatigue response of bonded joints is obtained by studying the crack initiation process. It has been shown that to do this effectively requires the use of multiple backface strain gauging. Such experimentation and associated damage modelling will lead to a characterisation of the evolution of initial damage within the joints and form the basis of completely general lifetime prediction procedures.

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THE EFFECTS OF MOISTURE ON THE DURABILITY AND STRENGTH RETENTION OF SINGLE LAP JOINTS

Professor R D Adams
Department of Mechanical Engineering
University of Bristol, Bristol BS8 1TR

ABSTRACT
Adhesive joints are designed and tested on a short-term basis, but are expected to perform satisfactorily over a long period. It is therefore important to understand why joints break after a long period of use, and how (or if) we can prevent this from happening, to avoid needing high safety factors.

Long-term failure in adhesive joints is due to a combination of loading and environmental conditions. The effect of water, temperature and time is reasonably well understood. But if stress is added, the joint strength is seriously reduced, even though the same stress by itself is unimportant.

Fick's equations can be used to show how, for a uniform adhesive layer, water or other solvents can diffuse into a lap joint. By looking at lap shear strengths and correlating these with diffusion profiles, a useful insight has been gained into the weakening mechanism involved.

INTRODUCTION
The effects of water on an adhesively bonded joint are almost certainly related to the adhesive's ability to absorb water. In most cases, an adhesive will absorb water very readily since its backbone is made up of many hydrophilic units. Diffusion through the adhesive can occur in all joints and is regarded as the primary access route. Cracks and porosity provide an additional and difficult to assess mechanism of absorption and transport of water.

In a wet or humid environment, water may easily penetrate into the bondline, causing swelling and deformation of the adhesive matrix, as well as the breakage of chemical or other bonds and consequent weakening of the polymer backbone. Furthermore, water may migrate to the adhesive/adherend interface, displace bonding sites formed along the substrate or otherwise weaken the surface layer. In any case, the overall strength of an adhesive joint is usually severely reduced as a result of exposure to moisture.

THEORETICAL BACKGROUND
Fick's first law of diffusion states that the rate of transfer of diffusing substance through unit area of a section is proportional to the concentration gradient measured normal to the section, i.e.

$$F = -D\frac{\partial C}{\partial x} \qquad (1)$$

where F is the rate of transfer per unit area of section, C is the concentration of diffusing substance, x is the space coordinate measured normal to the section and D is called the

Diffusion coefficient. The first law can only be applied to diffusion in the steady state which is where the concentration does not change with time.

For a thin adhesive film immersed in a liquid or a vapour at a constant pressure the solution to the equation is expressed in the following equation:

$$\frac{M_T}{M_E} = 1 - \sum_{n=0}^{\infty} \frac{8}{(2n+1)^2 \pi^2} \exp\left[\frac{-D(2n+1)^2 \pi^2 t}{4h^2}\right] \qquad (2)$$

where M_T and M_E are the masses absorbed at time t and at equilibrium and h is the thickness of the film. For short times, this simplifies to

$$\frac{M_T}{M_E} = \frac{4}{h}\left(\frac{Dt}{\pi}\right)^{1/2} \qquad (3)$$

The term "short time" refers to the region where the water uptake is linearly increasing with the square root of time. For Fickian diffusion, the plot of absorbed moisture is plotted against the square root of time. The behaviour of the graph can be essentially split into two stages. The initial stages of water uptake is a smoothly increasing function of the square root of time, $t^{1/2}$ and the second stage corresponding to apparent equilibrium is attained. This behaviour is termed Fickian (*see Fig 1*).

EXPERIMENTAL PROGRAMME

A structural adhesive film was used in this experiment. The AF 191 K/191U adhesive films are manufactured by the 3M company. Both films are a single-part, thermosetting, modified epoxides designed for bonding composites, metal to metal, and metal to honeycomb components where high strength is required. The adhesive is recommended to be stored at minus 15°C for achieving a maximum storage life. The curing temperature for AF191 adhesive films is around 170 -180°C. Solid samples of the adhesive were made from several layers of adhesive film that were bonded together under pressure and temperature.

For the lap joints, surface preparation is important to the strength and especially the durability. Therefore, to achieve optimum adhesion, the surfaces to which the adhesive was applied must be cleaned and pretreated. In this investigation, no surface treatment was used apart from grit blasting and degreasing. However, an extensive series of similar tests using the same adhesives had also been carried out by Critchlow et al (1), and their excellent experimental data have been used to broaden the in-house results.

The lap joint specimens were exposed to water at 70°C, but cooled and tested at 20°C.

RESULTS AND DISCUSSION

Measured weight increases for the test specimen, immersed in water at 70°C, was plotted in the form of weight gain against the square root of time, as shown in *Figure 1*. All the graphs behave quite similar to the ideal Fickian water uptake plot. As can be seen from the graphs, the weight gain is initially linear against square root of time, and then the initial uptake leads directly to water uptake equilibrium. The diffusion coefficient, D of water into the specimen is calculated from the initial linear slope of the graph using equation (3).

Lap shear tensile tests were carried out on joints bonded with AF 191 U adhesive film. The results obtained from the tests are displayed graphically in *Figure 2*. The graph shows that there is a progressive drop in the joint strength as the immersion time increases. The percentage loss of joint strength after one week's exposure is approximately 64.7 % with respect to the initial joint strength. This may be partly due to the effect of plasticization of the adhesive by the water, but the more likely reason is that the adhesion strength has been seriously reduced, by disbonding at or near the interface.

Since water diffusion is obviously the reason for the reduction in lap shear strength, the question arises as to just how this is taking place. Various authors have proposed that it is primarily due to interface disbonding, and that the water travels along the interface very much faster than through the bulk adhesive (albeit in the thin film form) in a lap joint. Unfortunately, there is little evidence to show that the transport at the interface <u>is</u> faster than in the bulk. Indeed, if there were a high concentration at the interface, it would rapidly diffuse into the interior of the joint since the concentration gradient would be enormous. It is therefore worth studying the actual concentration levels in an exposed lap joint, and how these compare with measured joint strengths.

Fig. 3 shows the concentration levels in a 12.5 x 30 mm lap joint (the dimensions used by Critchlow et al (1). The lines indicate the percentage of the joint which is below a given concentration at a particular time.

Fig. 3 also shows the results of Critchlow et al (and our own) on the Fickian concentration-time curves. The initial strength reduction (up to 1 week) seems to follow the concentration-time curves. After this time, the actual joint strengths change much more slowly with time. One of the surface treatments, Bonderite 901 plus Pyrene 8-90 has, after 8 weeks, almost 80% of its original bond strength, even though the adhesive layer, and hence the interface, is essentially at 100% concentration. Even their 'worst' surface treatment shows this levelling-off of strength with time. On the other hand, our own results, which were for a grit blasted and degreased surface, show much faster strength reduction, the results lying between the 20% and 30% concentration curves, and no levelling off.

CONCLUSIONS

It is clear from this work that there does not exist a critical moisture concentration at which disbonding will rapidly if not instantaneously occur. The reasons for the rapid initial loss in strength seems to relate to Fickian diffusion, but this cannot explain the levelling off of the strength-time relationship.

Finally, we must solve the simple question first, before adding the technologically important interaction with the applied stress.

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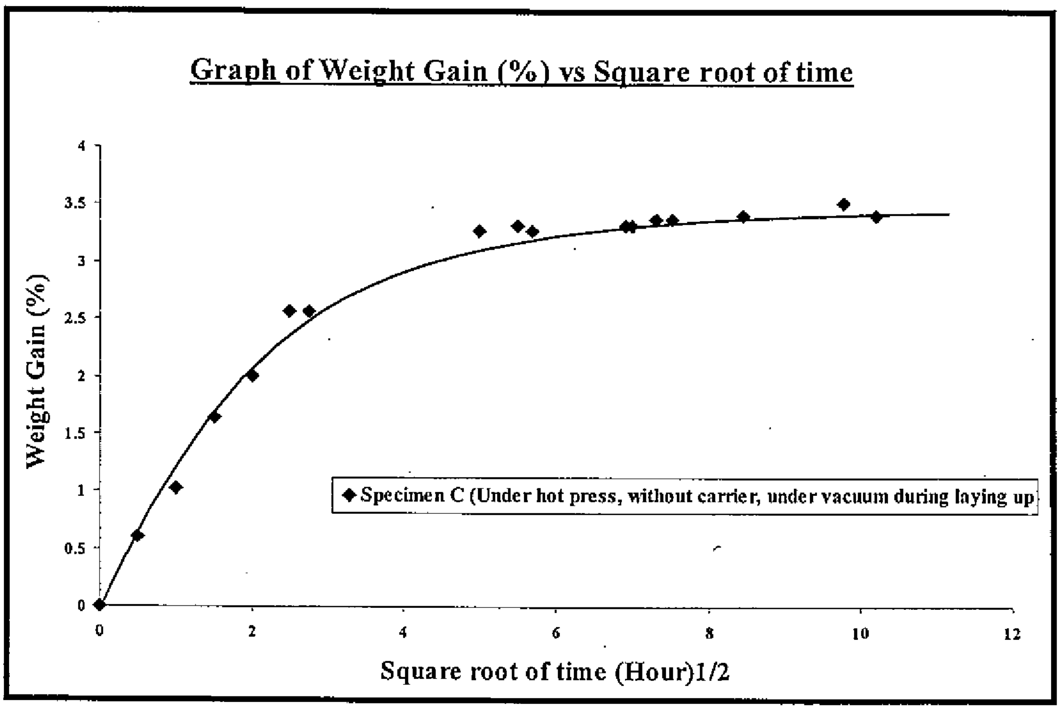

Figure 1 Water uptake curve for specimen C from the immersion experiment at 70°C

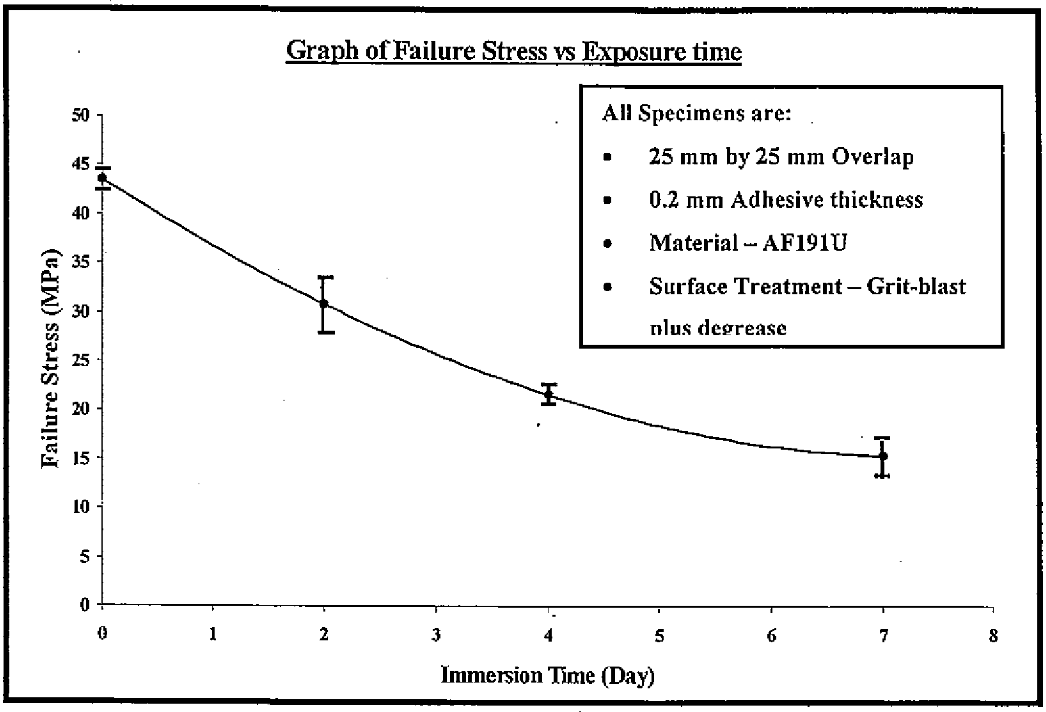

Figure 2 Effect of water on the strength of single lap joints at various immersion times

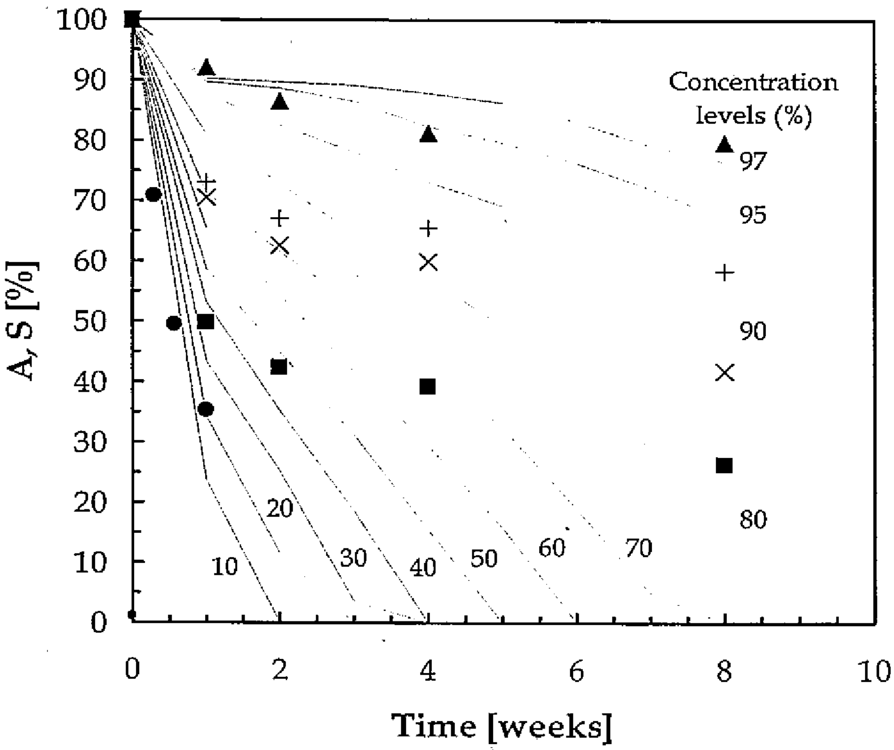

Figure 4 Proportion of the joint area (A) which is below a given water concentration level after a given time of exposure.
S [%] is the proportion of the initial strength retained: (●) Grit blast and degrease (present work), (▲) Bonderite 901 – Pyrene 8-90 [7], (1) Grit blast and degrease plus silane [7], (3) Pyrene 16-30 [7], (■) Bonderite 245 [7].