Bonding of Advanced Composites

One Day Symposium

Thursday March 4th 1993
The Institute of Materials
Adhesives Group

Symposium: Bonding of Advanced Composites
SCI, Belgrave Square, London
Thursday, March 4th 1993

0945  Registration

1015  Joining of Composites: An Overview
A.J. Kinloch, Dept. of Mechanical Engineering, Imperial College, London, UK.

1045  Modified BMI Adhesives for bonding High-Temperature, Structural Carbon Composites
T.J. Irvine, Ciba Composites, Duxford, Cambridge, UK.

1115  Coffee.

1140  Bonding of Thermoplastic Composites

1210  Patch Repair of Fibre Reinforced Aluminium Laminates

1240  Lunch.

1400  Repair of Structural Composites
J. Robson, A.J. Kinloch and F.L. Matthews, Composites Centre, Imperial College, London, UK.

1430  The Bonding of Metal Matrix Composites
M.R. Bowditch and D.A. Moth, DRA, Maritime Division, Holton Heath, UK.

1500  Tea

1520  The Study of Strain Distribution in Stressed Composite Bondlines
R. Davison and R.J. Lee, AEA Technology, Harwell, UK.

1550  Aspects of Adhesively Bonded Joints for Pultrusions and Fibre Reinforced Pipes
G.C. McGrath and M.A. Girardi, The Welding Institute, Abington, Cambridge, UK.

1620  Closing Remarks.

Note: Presenting Author is given in bold type
JOINING OF COMPOSITES: AN OVERVIEW

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Abstract

The present paper will concentrate on several important aspects when using adhesives to bond fibre-composites.

Firstly, the surface of the composite may have to be treated in order to obtain (i) adequate initial wetting and adhesion of the adhesive, and/or (ii) good long-term durability of the adhesive/composite interface. Examples will be given for polymeric fibre-composites based upon both thermosetting and thermoplastic matrices and for metallic matrices. Also, attention will given to the special problems associated with repair, when the surface of the composite may be heavily contaminated with water, oils, etc.

Secondly, the design of the adhesive joint is always an important feature, but this is particularly the case when bonding anisotropic materials, such as multidirectional fibre-composites. For example, many joint designs, such a lap joints, give rise to high tensile, or 'peeling', stresses which act normal to the main fibre-direction; i.e. parallel to the very direction where the composite has a relatively low strength. It will be shown that by careful attention to the design of the ends of typical overlap joints that such 'peeling' stresses can be minimised and the strength of the joint can be greatly increased.
MODIFIED BMI ADHESIVES FOR BONDING
HIGH TEMPERATURE STRUCTURAL CARBON COMPOSITES

T.J. Irvine
Ciba Composites, Duxford, Cambridge, UK

INTRODUCTION

With the ever more stringent requirements of the aerospace industry, the development of structural adhesives to meet these demands today, more than ever, requires very careful, novel formulating work.

This, traditionally involved the use of epoxy resins, but these, although they can be designed to cover a wide range of properties, do have their limitations. Thus, it has become necessary to turn to resins with different chemistries, particularly to achieve the strengths at high temperatures required by the industry today, to a great extent, due to the appearance of high temperature composites, which can only really be successfully bonded by using adhesives.

BACKGROUND

Epoxy resin adhesives can be formulated to cure at temperatures ranging from below room temperature to 175° - 180°C. The final cured products can be made to possess a wide range of properties depending on the choice of additives incorporated. For instance, incorporation of a rubber modifier can produce an adhesive with high peel strengths; high functionality resins tend to produce adhesives which retain their shear strength to high temperatures.

However, epoxy resins have an upper service temperature limit of approximately 180°C and today's end users are demanding oxidative durability at temperatures in excess of 200°C; to be compatible with the high temperature composites being produced.

FORMULATION OF HIGH-TEMPERATURE ADHESIVES

The initial work carried out to formulate adhesives meeting these requirements centred on polyimides, which certainly had the high temperature capability, but were notoriously difficult to process. They require very high pressures, together with very long cure and post-cure times to achieve full conversion.
In addition, the curing reactions of polyimides involve the loss of small molecules, thus volatiles are given off during cure, causing many problems within the glueline.

Bismaleimides, or BMI's as they are more commonly referred to, are a special type of polyimide, being prepared from maleic anhydride and diamines, preferably aromatic diamines. They are much easier to process than polyimides, having processing characteristics similar to epoxies, although post-cure for a short time is usually required to achieve full conversion and/or optimum properties.

The other major advantage, over other polyimides, is that the cure and post-cure reaction mechanisms do not involve any condensation products, so that no volatiles are produced.

BMI formulations inevitably tend to be brittle, due to their high cross-link density, which is, after all what enables them to retain joint strengths to such high temperatures. However, they can be made less brittle by the incorporation of polymeric modifiers, such as rubbers although these, having low glass transition temperature may reduce shear strengths at higher temperatures.

However, modifications of BMI's with epoxy resins can improve peel strength without loss of high temperature shear strength as long as the epoxy is carefully chosen.

Other modifications to BMI formulations can be accomplished in just the same way as they are for epoxies. For instance, the use of fillers can adjust the flow characteristics to achieve a system which is easier to process, incorporation of blowing agents produces foaming adhesives and subtle changes in co-cure chemistry can be made to achieve compatibility with substrate and speed of cure.

BMI STRUCTURAL ADHESIVES

Careful formulation work has led to a product (Redux 326) which has a significantly improved service temperature profile over even the best temperature resistant epoxies [Figure 1]. This system cures at temperatures which are typical of epoxy chemistry, is resistant to both wet and dry heat and is versatile in that it will bond both metal and composite substrates. Further, and more importantly, it has been formulated so that its
chemistry complements that of many of the substrates to which it will be required to bond. Co-cure with advanced composite substrates of similar chemistry gives particularly encouraging results [see Tables 1 and 2].

The neat resin properties of the system [Figure 2] show it to be stiff with a low fracture toughness (G_c = 364 J/m²) compared to most epoxy systems, but when compared to other BMI systems, for instance DLS 811, with a G_c value of 180 J/m², its improved toughness may be seen.

CONCLUSIONS

Careful formulation of readily available bismaleimide resins can yield structural adhesives which offer strengths above the 180°C limit of high temperature epoxies without difficult and time-consuming cure cycles being necessary. Toughening of these systems with high temperature epoxies and fillers can enhance their properties, making them very useful materials for bonding of today's high temperature advanced composites, as well as allowing the final formulation to be tailored to the chemistries employed by these same advanced composite materials.
Table 1: BMI/Epoxy Adhesives
Properties on BMI CFRP Adherends

| ADHESIVE | Redux 326 a supported film adhesive of 300 gsm areal weight |

DOUBLE LAP-SHEAR STRENGTH [MPa]
Pre-cured Adherends: Fibredux 926 / T800 UD Fabric

<table>
<thead>
<tr>
<th>Test Temperature [°C]</th>
<th>Strength [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>22°C</td>
<td>16</td>
</tr>
<tr>
<td>220°C</td>
<td>20</td>
</tr>
</tbody>
</table>

HONEYCOMB C-D PEEL [N/76mm]
Skin - Fibredux 926 / 5H Satin Fabric
Core - 7.91b / 0.25in Aluminium

<table>
<thead>
<tr>
<th>Co-Cure</th>
<th>Pre-cured Skins</th>
</tr>
</thead>
<tbody>
<tr>
<td>600</td>
<td>450</td>
</tr>
</tbody>
</table>

Figure 1: BMI/Epoxy Film Adhesives
Comparison with a High Temperature Epoxy

Metal/Metal Lap-Shear Strength [MPa]

Test Temperature [°C]

Redux 326: Cured 2 Hrs at 175°C + 1 Hr at 250°C
Redux 322: Cured 1 Hr at 175°C
Figure 2: Neat Resin Data

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile Strength</td>
<td>62 MPa</td>
</tr>
<tr>
<td>Tensile Modulus (E)</td>
<td>4.67 GPa</td>
</tr>
<tr>
<td>Elongation at Break</td>
<td>1.6 %</td>
</tr>
<tr>
<td>Poisson's Ratio ((\nu))</td>
<td>0.363</td>
</tr>
<tr>
<td>Calculated Shear Modulus</td>
<td>1.71 GPa</td>
</tr>
<tr>
<td>Flexural Strength</td>
<td>117 MPa</td>
</tr>
<tr>
<td>Flexural Modulus</td>
<td>4.08 GPa</td>
</tr>
<tr>
<td>Compression Yield Strength</td>
<td>193.1 MPa</td>
</tr>
<tr>
<td>Deformation at Yield</td>
<td>10.8 %</td>
</tr>
<tr>
<td>Fracture Toughness - (K_{IC})</td>
<td>1.39 MPa/m</td>
</tr>
<tr>
<td>Fracture Toughness - (G_{IC})</td>
<td>364 J/m²</td>
</tr>
<tr>
<td>Uncured Sg</td>
<td>1.258</td>
</tr>
<tr>
<td>Cured Sg</td>
<td>1.276</td>
</tr>
</tbody>
</table>

*: Calculated as \(G_{IC} = (K_{IC}^2 (1 - \nu^2))/E\)

1: Tested to ISO R527 Type I at a cross-head speed of 1 mm/minute.
2: Tested to BS 2762 Part 3 [Method 335A] or ISO 178 at a cross-head speed of 2 mm/minute.
3: Tested to ISO 604 at a cross-head speed of 1 mm/minute.
   [Right-Square Prism: 10 * 10 * 17.3 mm]
4: Tested in accordance with the EGF Task Group on Polymers and Composites Protocol "A Linear Elastic Fracture Mechanics (LEFM) Standard for Determination of \(K_c\) and \(G_c\) for Plastics" [Compact Tension], at a cross-head speed of 0.5 mm/minute.
Table 2: BMI FILM ADHESIVES
REDUX 326 – Profile

2024-T3 Al clad CSA pickled substrates
Cure: 2 hours at 180°C + 2 hours at 230°C

<table>
<thead>
<tr>
<th>TEST</th>
<th>FILM WEIGHT</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>250 gsm</td>
</tr>
<tr>
<td>Lap–Shear Strength [MPa]</td>
<td></td>
</tr>
<tr>
<td>22°C</td>
<td>19 {17.5} ¹</td>
</tr>
<tr>
<td>150°C</td>
<td>23</td>
</tr>
<tr>
<td>200°C</td>
<td>24 {17.5} ²</td>
</tr>
<tr>
<td>220°C</td>
<td>20</td>
</tr>
<tr>
<td>Ageing for 7 Days at 220°C</td>
<td></td>
</tr>
<tr>
<td>22°C</td>
<td>17</td>
</tr>
<tr>
<td>220°C</td>
<td>15</td>
</tr>
<tr>
<td>48-Hour boiling water</td>
<td></td>
</tr>
<tr>
<td>22°C</td>
<td>21</td>
</tr>
<tr>
<td>220°C</td>
<td>17</td>
</tr>
<tr>
<td>Honeycomb Flatwise Tensile [MPa]</td>
<td></td>
</tr>
<tr>
<td>22°C</td>
<td>2.1</td>
</tr>
<tr>
<td>Honeycomb C-D Peel [N/75 mm]</td>
<td></td>
</tr>
<tr>
<td>22°C</td>
<td>55</td>
</tr>
<tr>
<td>Matrix Minimum Viscosity after:</td>
<td></td>
</tr>
<tr>
<td>3 Days at 22°C [Pa.s]</td>
<td>0.98</td>
</tr>
<tr>
<td>23 Days at 22°C [Pa.s]</td>
<td>1.04</td>
</tr>
</tbody>
</table>

²: { } brackets enclose the values for joints prepared with film which had been pre-aged for 21 days at 22°C
Earlier work on bonding of thermoplastic fibre composites has been extended. APC-2 has been successfully bonded following an oxygen plasma surface pretreatment yielding high, cohesive, mode I fracture energies. High rate tensile tests have been used to simulate mode I impact conditions in adhesive joints. Tests have been conducted at rates of up to 10m/s. Finally, the environmental durability of bonded APC-2 and, for comparison, U/C Epoxy has been studied.

INTRODUCTION

A continuing challenge which faces the adhesives technologist is the necessity to ensure that the development of new advanced materials does not vastly outpace his ability to bond them successfully. A recent example of such a challenge has been the development of fibre composites based upon thermoplastic matrices. Not only are these tougher than those based on thermosetting matrices, but also have the ability to be readily moulded into complex shapes. It has been previously reported how very effective a corona discharge pretreatment was in obtaining adhesion between thermoplastics using toughened epoxy adhesives [1]. Recent advances in plasma science have permitted some exciting new developments in the field of surface treatment. It will be shown that an oxygen plasma treatment can be employed equally effectively.

Clearly, once adhesion has been achieved it is necessary to investigate how the joint behaves over a wide range of conditions before it can be adopted into service. Two key requirements are sufficient environmental durability and impact resistance. The ability to predict how these materials behave under such conditions is of vital importance to the manufacturer of, for example, military equipment where impact loading and varied environments may be encountered during service.

MATERIALS

The thermoplastic composite employed in this work was APC-2 reinforced with unidirectional AS4 carbon fibre from ICI plc. For comparison, a thermosetting composite was also studied. This was a unidirectional carbon fibre (U/C) epoxy composite (XAS/913C) from Ciba-Geigy. The adhesives used were a rubber toughened epoxy-film adhesive (FM73M) from American Cyanamid and a rubber toughened epoxy-paste adhesive (EA9309) from Hysol Dexter. Adhesively bonded double cantilever beam specimens were prepared as shown in Figure 1.

ANALYSIS

Fracture mechanics has been widely utilised in the study of crack growth in adhesive joints [2]. Assuming that a DCB specimen behaves in a linear-elastic manner upon loading, then beam theory may be used to calculate the fracture energy from the data contained in the load versus displacement traces [3]. Firstly, the corrected displacement method may be expressed as:
where $P$ is the force, $\delta$ is the opening displacement, $B$ is the width of the DCB specimen, $h$ is the thickness of one substrate, $a$ is the crack length and $F$, $N$ and $X$ are various correction factors discussed in [3]. Secondly, the corrected load method is given by:

$$G_c = \frac{3P\delta}{2B(a + \chi h)} F$$

(1)

where $E_s$ is the substrate modulus. Finally, eliminating the load from eqn. 1, it can be shown that:

$$G_c = \frac{12FP^2(a + \chi h)^2}{B^2h^3E_s}$$

(2)

It will be shown that Eqn. 3 has proved to be very useful for the analysis of high rate tests, because it is independent of the load values which have been shown to be in error at high rates.

OXYGEN PLASMA TREATMENT

EXPERIMENTAL

The effectiveness of an oxygen plasma surface pretreatment has been previously well documented [4]. In the present work APC-2 has been treated in an oxygen plasma unit prior to bonding. Mode I fracture energies have been measured for DCB joints bonded with either of the two rubber-toughened structural adhesives. The effect of employing different treatment times was investigated and scanning electron microscopy and x-ray photoelectron spectroscopy was carried out on the treated surfaces.

The plasma unit employed was a 'Plasmaprep 100 Plasma Chemistry unit'. The principal features of this unit were: the aluminium barrel reactor chamber, the rotary vacuum pump, the 220/240V, 50/60Hz electrical supply, the RF power amplifier, the oxygen cylinder, the valve HT transformer and the fixed inductor and variable capacitors (60pF, 200pF). The substrate materials were firstly abraded and cleaned with acetone and then treated in the plasma unit for periods between 2 and 20 minutes, at a forward power of 85w.

RESULTS

Figure 2 shows that a plasma treatment time of only three minutes was sufficient to achieve the high energy, cohesive mode of failure when APC-2 was bonded with the epoxy-film adhesive. However, when this material was bonded with the epoxy-paste adhesive, Figure 3 shows that a treatment time of 7.5 minutes was necessary to achieve cohesive failure and the high energy plateau was attained after a treatment time of 12.5 minutes.

Surface analysis by X-ray photoelectron spectroscopy (XPS) clearly indicated that the plasma treatment had greatly increased the atomic % of O (1S) relative to C (1S) on the surface, as was shown for corona treated joints [5]. In addition, the surface ablation which occurred during the treatment was shown to have reduced the surface concentration of releasing agents and scanning electron microscopy (SEM) indicated a marked increase in surface roughness, resulting from the plasma treatment.
HIGH RATE TESTS

EXPERIMENTAL

Adhesively bonded DCB specimens were tested in mode I over a wide range of opening rates. At displacement rates from $10^{-6}$ to $10^{-2}$ m/s, a screw driven tensile testing machine was employed with a static load cell. At rates between $10^{-2}$ and 10 m/s, a high speed, servo-hydraulic tensile testing machine was employed with a piezo-electric crystal load cell. A high speed camera was used to monitor the beam opening displacement. Severe dynamic effects were experienced at the higher test rates, and for this reason it was not possible to measure load values at rates of over 2 m/s. The APC-2 was corona treated.

RESULTS

The adhesive fracture energy, $G_c$, for the joint systems was calculated using eqns 1-3. Because eqns 1-2 require the load to be measured, only eqn. 3 was used above 2 m/s. The contribution of kinetic energy to the energy balance at crack initiation was less than 2% at 10 m/s and was therefore not considered. Values of $G_c$ at crack initiation have been calculated.

The values of $G_c$ calculated via eqns 1-3 show excellent agreement at slow rate. However, at higher test rates it is interesting to note that low values of $G_c$ were obtained when either eqn. 1 or 2 were used. Figure 4 shows that for the APC-2 joints bonded with the epoxy-film adhesive, then low values of $G_c$ were obtained from eqns 1-2 at rates above $10^{-1}$ m/s. However, when eqn. 3 was used to calculate $G_c$, which is independent of the load value, then the value of $G_c$ remained almost constant for this joint system from low to high rate. It was concluded that the fall off in the $G_c$ values given by eqns 1-2 were invalid and were an artifact of the test, caused by the dynamic effects introducing an error into the load measurements. Accurate and valid values of $G_c$ at high rate were given by eqn. 3, which was independent of the load measurements. Similar results were obtained for joints made with either the thermoplastic or thermosetting substrates. All failure was cohesive.

When the epoxy-paste adhesive was employed, then eqn. 3 showed that there was a genuine reduction in the value of $G_c$ at rates above 0.5 m/s. At 10 m/s, joints prepared with this adhesive suffered a 40% reduction in $G_c$, relative to the static value, as shown in Figure 5. This behaviour was again observed for either substrate materials.

Visual inspection and scanning electron microscopy of the fracture surfaces revealed that the high $T_g$, epoxy-film adhesive remained ductile at all test rates. Extensive cavitation of the rubber particle sites had occurred at even the highest rate and there was visual evidence of shear yielding. However, the fracture surfaces obtained from the low $T_g$, epoxy-paste adhesive showed that there had been a distinct change in the micromechanisms of fracture operating within the adhesive at high rates. Evidence of shear yielding and particle cavitation was observed at slow rates, but at high rates this occurred to a significantly lesser extent.

ENVIRONMENTAL TESTS

EXPERIMENTAL

An environmental chamber was set up in order to examine the durability of adhesive joints exposed to hostile environmental conditions. APC-2 and U/C epoxy substrates were prepared and bonded with either the hot curing epoxy-film or the cold curing epoxy-paste
adhesive. The APC-2 was corona treated. The hot-wet environmental chamber contained distilled water at 50°C. Each month, two joints of each of the four systems were removed and tested in mode I at 2mm/min. Joints were exposed for up to one year. To investigate the effect of the chamber temperature on the fracture energy of each adhesive, joints were exposed to in a hot (50°C), dry chamber.

RESULTS

The results obtained from the hot-dry exposure indicated that, at 50°C, the cold curing epoxy-paste adhesive exhibited a reduction in fracture energy of between 35-40% of the unaged value within one month. It was apparent that the elevated temperature had post cured this adhesive, producing a more crosslinked and thus more brittle adhesive, with a correspondingly lower joint fracture energy. The epoxy-film adhesive did not exhibit this effect. Its cure temperature is 125°C, i.e. well above the the chamber temperature, so no further curing took place.

The results of the hot-wet exposure are shown in Figures 6-7. It was particularly interesting to note the significance of the substrate material to the long term durability of the joints. Figure 6 shows that when the U/C epoxy was bonded with the epoxy-film adhesive, then the value of $G_c$ initially increased with ageing time and then decreased significantly. All the failure in this joint system was cohesive, in the adhesive. However, when corona treated APC-2 was bonded with the epoxy-film adhesive, then the value of $G_c$ immediately decreased and continued to fall until a lower plateau was reached. This decline was associated with the progressive change in the locus of joint failure, from cohesive to apparently interfacial. Water diffusion and bulk adhesive tests have been carried out on the two adhesives in order to gain a further insight into possible mechanisms [6] by which water has weakened the joints.

When the epoxy-paste adhesive was employed it was found that interfacial failure was not observed with this adhesive (except for very small amounts in the APC-2 joints after a long period of exposure). However, it was clear from a visual inspection that water had attacked this adhesive, resulting in discolouration and a change in fracture surface roughness. It can be seen in Figure 7 that the joints prepared with the APC-2 substrates were somewhat more durable than the joints prepared with the U/C epoxy substrates using the same adhesive. Because the failure was always cohesive, it was concluded that this difference was due to the effect of stress on the adhesive, caused by different amounts of swelling of each substrate material.

ACKNOWLEDGEMENTS

The authors would like to thank Dr Peter Davies and Dr Wesley Cantwell at the EPFL (Lausanne, Switzerland) for kindly allowing us the use of their plasma unit, Dr John Watts at the University of Surrey for use of surface analysis facilities and finally the United States Army, through their European Research Office, for financially supporting this work.
REFERENCES


FIGURE 1. The Adhesively bonded Double Cantilever Beam (DCB) Specimen
FIGURE 2. $G_C$ versus plasma treatment time for the APC-2 substrates bonded with the epoxy-film adhesive. (Open points: Interfacial; Closed points: Cohesive). Substrates were abraded and solvent cleaned prior to plasma treatment.

FIGURE 3. $G_C$ versus plasma treatment time for the APC-2 substrates bonded with the epoxy-paste adhesive. (Open points: Interfacial; Closed points: Cohesive). Substrates were abraded and solvent cleaned prior to plasma treatment.
FIGURE 4. $G_C$ at crack initiation versus displacement rate for the APC-2 substrates bonded with the epoxy-film adhesive. The APC-2 was corona treated to 10J/mm$^2$. All failure was cohesive. Stick-slip crack growth was observed.

FIGURE 5. $G_C$ at crack initiation versus displacement rate for the APC-2 substrates bonded with the epoxy-paste adhesive. The APC-2 was corona treated to 20J/mm$^2$. All failure was cohesive. Stick-slip crack growth was observed.
FIGURE 6. Retention of the 50°C dry $G_C$-values versus exposure time for joints bonded with the epoxy-film adhesive. Square points indicate U/C epoxy substrates; Diamond points indicate APC-2 substrates. The APC-2 was corona treated to 10J/mm². The numbers next to diamond data points show the % of cohesive failure.

FIGURE 7. Retention of the 50°C dry $G_C$-values versus exposure time for joints bonded with the epoxy-paste adhesive. Square points indicate U/C epoxy substrates; Diamond points indicate APC-2 substrates. The APC-2 was corona treated to 20J/mm². All failure was predominantly cohesive.
Introduction and Summary

Fiber metal laminates are a family of advanced hybrid aerospace structural materials consisting of thin aluminum alloy sheets adhesively bonded to alternating layers of fiber/epoxy prepregs. Two classes of laminates are available: aramid fiber reinforced ARALL and GLARE, which incorporates high-strength glass fibers. Fiber metal laminates have high specific tensile strength and excellent fatigue crack growth resistance, while retaining the good workshop properties of monolithic aluminum alloys. ARALL 3, the aramid/epoxy/7475-T76 laminate, is currently in production and flight test as the skin of the C-17 aft cargo door.

A series of one-sided bonded patch repairs was carried out on 220 mm wide ARALL 3 panels. (Incidental damage was simulated by cutting a 38 mm diameter hole in the center of the panels.) In-field bonded repair techniques involving vacuum bags and heat blankets were applied. Both elevated temperature curing epoxy films and room temperature curing two-part paste adhesives were evaluated, as well as monolithic aluminum and fiber metal laminate patch materials. The best combination of patch material and bonding techniques restored the panel to more than 90% of its undamaged strength, after extensive fatigue testing. The results showed a significant influence of patch stiffness on the overall residual strength of the repaired panel: the stiffer the patch, the weaker the repair.

Further investigation into the failure modes of the bonded repair panels showed that fatigue damage in the ARALL panel just beyond the tip of the repair patch typically resulted in a small static delamination in the first prepreg layer of the ARALL. Upon residual strength testing, controlled delamination growth occurred during which the repair patch and the first aluminum layer of the laminate peeled, followed by final failure of the laminate across the reduced net section of the cutout. A failure model for cracked laminates based on an energy release rate criterion has been proposed which reasonably predicts the observed failure behavior.

Bonded Repairs to ARALL 3

Bonded GLARE 3 and 2024-T3 patches were evaluated using simulated in-field techniques. The 3M adhesive AF163-2 was used most extensively in this program. Its mechanical properties and durability have proven to be excellent when cured with only vacuum pressure at 120°C for an hour. A resistive "heat blanket" accomplished the cure. Two-part epoxy paste adhesives (Ciba Geigy Araldit AW106 resin with HV9534...
hardener and 3M EC 9323) were used as well. The Araldit specimen was "rivet-bonded," that is, non-structural pop rivets held a monolithic aluminum patch in place during the cure. After one day at room temperature, the cure was accelerated by heating the panel at 100°C for an hour. In another test, a GLARE 3 patch was held in place with vacuum pressure during curing of the 3M EC 9323 two-part paste epoxy adhesive (with 1% by weight glass microballoons for bond line thickness control) and cured at 60°C for 2.5 hours.

Patch thicknesses were chosen from available standard gauges to provide static strength roughly equivalent to the tensile strength of 3/2 ARALL 3. The chosen patch thicknesses were 1.8 mm for 2024-T3, 1.4 mm for GLARE 3 bonded with AF-163-2 adhesive, and 1.1 mm for GLARE 3 with glass fiber/epoxy prepreg in the bond line. Given the relatively moderate elastic modulus of ARALL 3, this resulted in patch stiffness values (elastic modulus times average thickness) which varied by a factor of approximately two.

Constant amplitude fatigue tests \((S_{\text{max}} = 120 \text{ MPa}, R = 0.05, f = 10 \text{ Hz})\) were run on 3/2 layups at room temperature in lab air. (All stress levels referred to in this report are gross stress levels in the unnotched skin, i.e., force divided by skin area.) Based on previous experience with laminates, the repaired specimens were tested for 270,000 cycles (representing three lifetimes of a typical narrow body commercial airliner), followed by a static residual strength test to failure.

To establish a baseline strength value, an unrepaired 220 mm wide open hole specimen was tested to static failure. The baseline specimen was not fatigued prior to the static test. Failure occurred at the minimum net section at a gross stress of 388 MPa.

**Test Results**

The results are summarized in figure 1. The patch material, patch thickness and adhesive appear on the respective bars. Fatigue cracking was noted in the upper layer of the ARALL skin, just outside the patch tip, in the case of the 1.8 mm thick 2024-T3 patch. Failure of the bonded repair specimens typically initiated by interlaminar peeling of the top layer of the ARALL skin. The peeling proceeded in the rough shape of the patch from the tip to the minimum net section, where final overload occurred. This failure mode demonstrated that the unreinforced adhesive which bonded this patch to the skin had a higher peel strength than the (aramid fiber reinforced) prepreg within the laminate itself. Although unreinforced adhesives generally resist peel better than prepregs, this result shows that the vacuum bag/heat blanket bonding of the patch performed quite well in comparison with "factory-made" (autoclave bonded) ARALL.

Several interesting trends can be noted from the results. First, the stiffest patches gave the lowest overall strength values. Extensive tapering by chemical milling of a monolithic 2024-T3 patch produced only a marginal improvement in overall strength. The use of a fiber-reinforced prepreg in the repair bond line caused a reduction in overall panel strength and was accompanied by a change in failure mode to stable delamination (peeling) of the bonded patch from the ARALL panel. An identical patch tested without fibers in the bond line resulted in an 18% higher ultimate strength.

The rivet bonded/paste adhesive specimen failed in tension at the rivet hole nearest the tip of the patch. Nonetheless, its residual strength performance was comparable with the similar 2024-T3 patch bonded with film adhesive. When the two 1.4 mm GLARE 3
patches are compared, the higher performance of the film adhesive is apparent: the specimen bonded with AF-163-2 film outperformed the EC 9323 paste by 20%.

When identical bonding processes are compared, a striking reverse correlation between patch stiffness (elastic modulus times average thickness) and overall repair strength becomes evident. Figure 2 shows that, as patch stiffness increases, there is an approximately linear reduction in the overall strength of the repaired panels. (For the tapered 2024-T3 patch, its average stiffness was calculated.) This trend can be better explained if one considers the load transfer into and around the patch. A very stiff patch tends to attract a higher portion of the overall load. In addition, a thicker patch induces more secondary bending in the sheet. Both factors result in a large stress concentration in the repaired panel immediately beyond the tip of the patch. In ARALL, this results in fatigue damage in the uppermost aluminum layer, followed by controlled delamination between the now cracked aluminum layer and the first prepreg layer.

Once the fatigue damage has initiated delamination in the first prepreg layer of the base laminate, the controlling failure mechanism becomes the energy release rate between the upper aluminum sheet and the prepreg layer. The case of a bonded doubler which extends across the entire width of the base laminate without tapering has been derived in [2]. The controlling relationship for stable delamination is:

\[ R = G \]

where \( R \) = delamination resistance of the laminate, (typically 1.0 to 2.3 N/mm for ARALL, depending on loading mode),

and \( G \) = delamination driving force, equivalent to the energy release rate.

The derivation will not be presented here due to space limitations; however, the result of the derivation is that the delamination driving force can be expressed as:
Figure 2. Correlation of 3/2 ARALL 3 bonded repair residual strength after fatigue with patch stiffness. All patches were bonded with AF-163-2 adhesive cured with vacuum at 120°C.

\[ G = \frac{1}{2b} P^2 \frac{1}{AE} \left( \frac{1}{B} - 1 \right) \]

where \( b \) is the specimen width,
\( P \) is the axial tension load,
\( A \) is the cross-sectional area of thicker part of the specimen,
\( E \) is the elastic modulus of the thicker part of the specimen and
\( B \) is the ratio \( (AE)_{thick}/(AE)_{thick} \).

This relationship can predict the failure stresses of bonded doubler configurations relatively accurately when fatigue damage exists in the upper layer of the laminate near the thickness step. Reference [2] shows that this relationship applies in general to thickness steps ("doublers") in fiber metal laminate structures. If fatigue cracks have not yet developed, the prediction will be conservative. Further work is needed to measure more accurately the crack growth resistance (R) of ARALL and GLARE fiber metal laminates, and to extend the model to complex geometries involving width-tapered doublers.

References


PART ONE: THE CONCEPTS OF REPAIR

INTRODUCTION

Composite materials are different from many conventional materials in two major ways. 1) The material and component are often made in one operation. 2) The material is often inhomogeneous on a macro-scale. In general, composites are reinforced by fibres, in either chopped or continuous form. When damage occurs to the composite, often fibres are fractured, as well as the matrix material.

In one sense, it is impossible to restore the integrity of the material, as the broken fibres cannot be mended. However, it is more usual to take a more pragmatic approach: the aim of a repair is to restore the structural integrity of the component as a whole. This discussion concentrates on bonded repair patches, made of composite materials.

TYPICAL BONDED REPAIRS

The design of a bonded repair depends on: 1) the loading of the structure; 2) the thickness of the structure; 3) the extent of the damage. Various types of repair are shown in Fig. 1.

The simplest of bonded repairs are cosmetic, and carry no load at all. These would cover scratches to prevent contamination by fluids in the environment. One step further along, is a simple external patch. This may be very basic (e.g. a patch on a fibre-glass canoe), or more technologically advanced (e.g. an emergency repair to a fighter-plane wing).

More complex repairs may use stepped lap or scarfed joints. These are especially useful in aircraft applications as the original surface contour of the component can be reproduced more closely than is possible with an external patch. The patch may be thicker than the original component, and additional plies are usually cut to different lengths to taper the edge. Ply ends may also be serrated. Both these measures reduce the peel stresses at the edge of the patch.

When using a pre-cured patch, an adhesive is essential. When the patch is made from prepreg material, a film adhesive is normally used. The prepreg patch and the adhesive are then cured in the same operation.

FIG. 1: Bonded Composite Repairs.

(a) cosmetic repair; (b) external patch repair; (c) flush patch repair with scarf joint; (d) flush patch with double scarf joint; (e) flush patch with stepped lap joint; (f) overlapping repair plies, showing one ply end serrated.
PROPERTIES OF REPAIRS

Repairs are often made in non-ideal environments, so it is hardly surprising that the properties of repair laminates may be different from laminates of the same material made in a tightly controlled manufacturing process. And it is not only the immediate mechanical properties of the repair joint that are important. A permanent repair should last as long as the original component. Thus the long-term properties of the repair should also be considered, and investigated, before a repair can be certified as “safe”.

Meaningful research into the properties of repairs is not, however, very straightforward. The only guaranteed reliable method to measure their properties is to test the whole repaired component, but this is often not practical. Results from the use of simple, idealised joints may not be relevant, and fracture mechanics approaches may not give the whole story. Thus coupon joints of a realistic geometry were used in the research described below.

PART TWO: THE REPAIR OF XAS/914 PARENT WITH XAS/914, USING BSL 319 ADHESIVE

THE JOINT

The joint is typical of those used for the permanent repair of secondary aircraft structure, and is shown in Fig. 2. The parent structure is of quasi-isotropic XAS/914 carbon fibre-reinforced epoxy resin, and panels are 2mm thick (16 unidirectional plies). The joint is a scarf joint with a slope of 1:30 (−2°). The repair has 18 plies. The central plies are in the same orientation as the parent material, and the two extra plies are both aligned with the load. The extra plies overlap the ends of the scarf joint by 12.5mm. A coupon containing one such joint is used in the tests.

FIG. 2: The Design of the Repair Joint Studied

PERFORMANCE IN FATIGUE

There are two main ways in which to assess the fatigue performance of a joint or material: 1) by the relationship between the maximum fatigue stress and the number of cycles to failure (the S-N curve); 2) by the relationship between the number of cycles of fatigue and the residual strength after fatigue, at a given maximum fatigue stress. Both these investigations are complemented by an examination of the developing fatigue damage.

The S-N and residual strength curves for the repair of an XAS/914 parent with XAS/914, using BSL 319 adhesive, are shown in Fig. 3, and an illustration of the fatigue damage at 5 × 10⁵ cycles is shown in Fig. 4. The fatigue life of the repair is much lower than for the parent material at a given stress level, but the two curves are of roughly the same shape. The residual strength shows no significant drop before 10⁴ cycles, but drops rapidly as the fatigue life at that stress level is approached.
The main fatigue damage that develops is delamination and ply cracking. Cracks occur in the 90° plies, parallel to the fibres, from very early in the fatigue process. Delaminations occur at 0/90 interfaces close to the centre of the laminate, due to Poisson's ratio mismatch. The other main damage is cracking initiated at the ends of the overlapping repair plies. The crack at the shoulder of the scarf is more critical as it is the propagation of this crack, parallel to the scarf slope, which causes failure of the joint.

The fatigue performance of this repair joint is governed by damage initiation and propagation in the composite adherends. The adhesive plays no significant part in the fatigue failures.

FIG. 3: Fatigue Behaviour of Repair of XAS/914 Parent with XAS/914
(a) fatigue life (S-N) curve; (b) residual strength curve.

FIG. 4: Fatigue Damage at 5 x 10⁵ Cycles

Crack Under Delaminations at Overlapping Ply

Delaminations at 0/90 Interface

Ply Cracks

THE EFFECT OF A HOT/WET ENVIRONMENT

Exposure to a hot/wet environment is often detrimental to adhesive properties and to the matrix-dominated properties of composites. Humid environments are often encountered by composites in service, and so it is important to assess their effect on repair joints. The environment chosen was 84% relative humidity (RH) and 50°C. A composite exposed to 84%RH will absorb as much moisture at saturation as one exposed to the worst world-wide real environment.

The change in strength with exposure time for the repair of an XAS/914 parent with XAS/914, using BSL 319 adhesive, is shown in Fig. 5, and the change in failure mode is shown in Fig. 6. The repair of XAS/914 with XAS/914 shows an increase in strength over the first month of exposure. This increase is maintained until about 6 months, and then lost. The strength stabilises after about 1 year of exposure. An increasing amount of adhesive failure is
observed with increasing exposure time. It is thought that the initial increase in strength is due to the relief of residual curing stresses by hygrothermal swelling of the laminates. The subsequent drop in strength seen in this case is probably due to the adhesive’s plasticisation.

**FIG. 5:** The Change in Strength on Exposure to a Hot/Wet Environment
Repair of an XAS/914 parent with XAS/914, using BSL 319 adhesive. Environment : 50°C, 84%RH.

![Graph showing the change in strength over time](image)

**FIG. 6:** The Change in Failure Mode on Exposure to a Hot/Wet Environment
Repair of an XAS/914 parent with XAS/914, using BSL 319 adhesive. Environment : 50°C, 84%RH.
The thick line indicates the failure path.

![Failure mode before and after exposure](image)

**CONCLUSIONS**
The properties of a repair joint depend on the properties of both the parent and repair laminates, and on the adhesive used to bond them. The individual joint components may be susceptible to different stresses or environments. Thus, while the adhesive properties may dominate the properties of the joint in one situation, they may have no effect on the joint properties in another situation. Also, some “damage” may occur to one of the joint components, without affecting the strength of the joint as a whole.
ADHESIVE BONDING TECHNIQUES FOR USE WITH PARTICULATE REINFORCED METAL MATRIX COMPOSITES.

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Bonding techniques for use with particulate metal matrix composite materials have been developed and assessed using a variety of test methods. Due to the presence of SiC particles at the interface, anodizing processes, which would normally be used to obtain the most durable joints are unreliable. The oxide formed on anodizing in various electrolytes has been examined, and joint strengths reported. These compare favourably in some cases with joints made using conventional aluminium alloy adherends. An alternative, more consistent approach has been developed using an acid etch followed by treating the surfaces with an appropriate silane coupling agent. Results are presented for each of the pretreatments, using unstressed joints and future work proposals such as impact and continuous stress testing are discussed.

EXPERIMENTAL:

The structural use of MMCs in commercial ventures has been restricted by several factors one of these being the difficulty of joining these materials. We have examined the potential for the adhesive bonding of 8090 particulate MMC, when bonded using conventional and non-conventional pretreatment techniques.

As a first approach we looked at the structures formed during conventional chromic acid anodizing (CAA) and phosphoric acid anodizing (PAA). The material, which is lithium (8090) alloyed, with 25% SiC reinforcement, appears at first sight to anodize in a reasonably satisfactory manner, although the voltage / current relationships during anodizing are rather unusual with increasingly large currents being drawn as anodizing proceeds. An oxide is nevertheless formed, which it was felt might be satisfactory for bonding purposes. However, examination of the oxides under the electron microscope revealed an anodic structure which clearly was not ideally suited to adhesive bonding. Whilst the oxide thicknesses obtained were as expected, 0.5 µm for PAA and 3-5 µm for CAA, the actual structures were quite different. The presence of the non-conducting particles has clearly disrupted the current distribution at the electrolyte / oxide boundary resulting in retarded barrier layer growth and, most importantly, a non-porous cell structure.
It is now widely accepted that in order to create good durability when bonding aluminium it is essential to achieve good penetration of the porous oxide by the adhesive and total surface wetting of the adhesive onto the substrate. These factors can be controlled by care in adhesive selection and good bonding technique thereafter.

The adhesives we used in these experiments were chosen because we had previous experience with these adhesives and their abilities to fully wet and penetrate an anodic oxide. In the case of MMCs, however, no porous oxide results from anodizing and so adhesive penetration into the oxide to create a micro-composite of adhesive in oxide is impossible.

Because of these observations, and despite some promising initial joint strengths, we did not consider it acceptable to assume that good durability would inevitably follow from the initial joint strengths. Alternative methods of joint preparation were investigated which were intended to make use of the non-conducting asperities at the surface rather than to persist in the use of electrochemical processes which are disrupted by their presence.

In the first durability experiments anodized pretreatments were not therefore evaluated. These early experiments were designed to investigate the possible use of chemical coupling agents and hydration inhibitors with MMCs when used in conjunction with a simple chromate free acid etch (Oakite 33). Joints were assembled using two types of adhesive, a two-part rubber toughened acrylic and a single-part rubber toughened epoxy, chosen for the reasons outlined earlier. Six joints were tested in each case.

Having established that good initial joint strength and reasonable durability can be achieved from a variety of surface preparation techniques, further testing of a more specific nature is being carried out. For all other tests in the program the appropriate silane treated etch was used as the reference against which other pretreatments were evaluated.

These other tests, which have recently begun, include high speed impact testing and steady state stress rupture testing.

**RESULTS/DISCUSSION:**

Electron micrographs showing typical surface topography and, in appropriate cases, the cross-section through the oxide are shown in figure 1. The pretreatments considered are:

- a) a phosphoric acid based, chromate free etch
- b) a phosphoric acid anodize
- c) a chromic acid anodize

The reinforcing particles are evident, in many cases breaking the surface after the pretreatment. The surface topography of each of the three pretreatments looks normal. Cross-sections of the anodized samples shows that the oxide structure is, however, abnormal, with no evidence for pore formation.
Joints made using these pretreatments, and an experimental pretreatment which uses a phosphoric acid etch following chromic acid anodizing, gave the initial joints strengths shown in figure 2. These showed that in most cases un-aged joints with comparable strengths to conventional alloys can be made, but the experimental CAA+PAD pretreatment performed less favourably.

This result, together with the electron micrographs, indicated a problem with the anodized pretreatments. To investigate the possible use of non-electrochemical pretreatments, joints were made up using the acid etch followed by treatment with a suitably compatible silane coupling agent, gamma-Glycidoxypropyl-trimethoxy-silane (A187) for epoxy joints and gamma-Methacryloxypropyl-trimethoxy-silane, (A174) for acrylic joints, which was chosen to match the chemistry of the adhesive. Also, an organo-phosphonic acid, Nitrilotris-methyleneephosphonic acid (NTMP), was used for some joints, in this case to inhibit hydration of the anodic oxide. The joint strengths obtained are shown in figure 3, and it can be seen that there may be some advantage in using a carefully chosen silane, but at present no advantage in using the hydration inhibiting agents. Durability testing was performed by immersing unstressed, undrilled joints in distilled water at 50° C and measuring the failure strength as a function of exposure time.

The advantage of the silane can now be seen with joint strength remaining reasonably good even after 3000 hours of accelerated ageing. The hydration retarder has performed badly and there is clearly no advantage in its use in this application.

A much more severe test for an adhesive joint is to subject that joint to the combined effects of temperature and stress. This test is particularly discriminating towards the effectiveness of the surface pretreatment. This work has barely begun, but we are able to report encouragingly high times to failure, almost certainly as a result of the very high stiffness of the adherends and the consequent reduction in peel forces within the joint. High modulus epoxy adhesives are likely to produce the strongest joints under these conditions.

Similarly, the effects of a very high strain rate, such as might be experienced during an impact loading, are being examined and compared with more conventional materials. First indications from the unaged tests are that the test is extremely sensitive to type of adhesive used. It is probable that when used with the more common toughened epoxy systems, MMCs will exhibit similar performance to conventional aluminium alloys. When used with high performance acrylic adhesives, it is unlikely that the MMCs will show such good impact behavior, because the apparently weaker oxide formed on MMCs is likely to fail under impact at the high energies that acrylic adhesives can absorb.
CONCLUSION:

Bonded joints using conventional adhesive bonding pretreatments such as anodizing and chemical etching have been made and tested. The oxide formed on MMCs is unusual in that it does not appear to have a cell and pore structure. However, joint strengths have been high, probably due to the stiff nature of the adherends. In an attempt to produce a durable joint without anodizing an etch / silane treatment has been used, which has resulted in consistent joint strengths.

The unstressed, undrilled durability tests using the etch / silane pretreatment has produced joints which have retained 70% (epoxy) and 55% (acrylic) of their initial strength after ageing at 50°C for 3000 hours. This compares with unstressed, drilled anodized joints made from conventional aluminium alloys which typically retain 70% (epoxy) and 90% (acrylic) of initial strength after 1000 hours accelerated ageing. We estimate that drilled joints will degrade four times as quickly as undrilled joints.

Further testing is in hand to evaluate performance under the influence of a steady load at elevated temperatures and also to assess the effects of high speed impact on the performance of bonded MMC joints. Initial findings are that high strength and durable joints will be possible, but care will be needed to ensure that a suitable adhesive / pretreatment system is selected for a particular application.

Metal Matrix Composite Pretreatment Surface Structures.

Figure 1(a) MMC etched topography x 5400

Figure 1(b) MMC phosphoric acid anodized topography, X 10000 and cross-section, X 30000

Figure 1(c) MMC chromic acid anodized topography, x 10000 and cross-section, x 15000
Figure 2. Showing the relative strengths of bonded aluminium alloy joints compared with MMC joints for two adhesive systems.

Adhesive Bonding MMCs

Bowditch & Moth
Figure 3 Showing the effects of pretreatment on the joint strengths of adhesively bonded particulate reinforced metal matrix composites.
The Study of Strain Distributions in Stressed Composite Bondlines

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4 March 1993, London

Introduction

In order to obtain quantitative shear strain and modulus data for structural adhesives much effort has been expended in developing zero gauge length extensometry (AEA, Althof and Krieger). Most designs are quite delicate so that the extensometer must usually be removed before failure of the joint, so as to prevent damage to the gauge. In all gauge designs deformations occurring in the adherends need to be corrected for shear and rotation effects. They all produce average values of deformation across the bond line thickness at discrete points near the centre of the bond as a function of load from which a shear stress strain curve can be obtained. However, even in the thick adherend shear test (TAST) geometry, where the adhesive might appear to be subjected only to shear displacements, there are always associated peel deformations which vary along the bond length. It is not possible using conventional extensometry to measure these effects, nor to determine how or whether the deformations vary across the bond thickness.

High Resolution Laser Moiré Interferometry

The experimental determination of deformation fields in both stressed bond lines and adjacent adherends is now possible using a high resolution Laser Moiré Interferometric (LMI) method. The quantitative measurement is made more difficult because generally structural bond thicknesses are typically less than 0.5 mm.

The practical value of LMI is that full field deformations in orthogonal directions can be made simultaneously. The sensitivity of ~0.42 µm per fringe order allows such measurements to be used to:

- quantitatively measure the stiffness of structural adhesives in joints,
- quantitatively observe effects which are impossible to model,
- gain an improved understanding of the mechanisms which control failure,
- verify and refine theoretical models,
- improve test specimen geometries,
- investigate quantitatively creep and fatigue effects in stressed bonds.

Moiré measurements on modified TAST specimens have been taken using metal adherends and structural epoxide adhesives on both thin and thick bondlines.
AEA Technology Adhesive Joint Design Codes

Computer models to predict the stiffness and strength of adhesive joints based on both continuum mechanics and on finite element techniques have been developed at AEA Technology, Harwell. The prediction of stresses and strains in adhesively bonded joints can be achieved efficiently and for simple geometries using closed form solutions as used in BISEPS-LOCO developed at Harwell in the mid-eighties. Such codes are generally restricted to simple geometries and can not easily be extended to more complicated geometries or loading conditions. In order to model the behaviour of joints of arbitrary complexity, a purpose written code, FELOCO (Finite element lap joint in combined loading), has been formulated. The program is run on a PC and written to be user friendly with automatic 2D mesh generation around the bonded joint. The following features can be accommodated:

- single and double lap shear geometries
- profiled adherends
- profiled adhesive layer
- spot welds allowed in the bond line
- stepped bond analysis
- linear orthotropic materials allowed for adherends
- linear and non-linear isotropic materials for both adhesive and adherends
- thermal expansion effects in both adherends and adhesives
- prediction of shear, peel and longitudinal stresses and strains in both adhesive and adherends
- pressure forces allowed on top and bottom surfaces of the joint prescribed displacements allowed.

Results may be requested along defined lines inside the adhesive layer and at discrete layers within the adherends to obtain axial, peel and shear stresses and strains and horizontal (u) and vertical (v) displacements. The number of increments at which the output is given is determined by the user and is restricted by the limits of machine memory.

Moiré Tests on Thin Bondline TAST Specimens

Tests, to failure, on TAST specimens made using steel adherends with bond thicknesses of 0.5 mm have been studied using LMI. Fringe densities in the adhesive become very high for loads close to failure initiation, however, by altering the spacing of the virtual reference grating it is possible to introduce a carrier pattern in order to coarsen the moiré fringes. The shear stiffness of the adhesive can be accurately quantified and was found to be in the region of 1 GPa for the Ciba-Geigy 2007 adhesive. Also it is clear that the shear deformations are uniform across the bondline thickness except in regions near the ends of the joint. The equivalent peel strains are compressive at the centre of the joint and tensile at the ends. As shown in Figure 1, these experimental observations are in good agreement with the strain distributions predicted by BISEPS-LOCO. Moiré fringes indicate that the peel strain is non-uniform through the thickness of the joint and this is especially marked close to the ends. Failure initiated close to
the interface near the joint ends where the peel strains and shear strains are the highest. Crack growth is stable. The maximum peel strain at the crack tip remains constant at 0.8% as the crack grows. The corresponding shear strains are supported by the uncracked adhesive.

**Moiré Tests on Thick Bondline TAST Specimens**

In order to observe more clearly the though thickness effects occurring, a specimen with an adhesive thickness of 2.85 mm and a bond length of 8.61 mm between aluminium adherends was studied. The results of the u and v displacement fields are shown in Figure 2. The equivalent shear and peel strain distributions are shown in Figure 3-5. Figure 3 shows the shear strains fall to zero at the free edges, falling most rapidly close to the interface and least rapidly at the centre of the bond. In all cases the effected region is within a bondline thickness of the free edge. Figure 4 shows the peel strain through the bond thickness at the free edge. The strain is tensile ~1.8% at the left interface, compressive ~1.4% at the right-hand interface and close to zero at the centre. The moiré data on the thick bond specimen has been compared with predicted solutions using FELOCO. The points (■) in Figure 5 show the predicted peel strains at the centre of the bond and at the interfaces. An adhesive shear modulus of 0.97 GPa, obtained from the moiré fringe measurements has been used in the calculation. Figures 5 and 6 show the excellent agreement between experiment and predictions for the peel strains and shear strains along the bond length at the interfaces and at the middle of the bond. In this case the experimental moiré has served as a tool to test a complex analytical approach albeit in a relatively simple loading geometry.

**Moiré Tests on Composite to Steel TAST Specimens**

Recent experiments on stressed joints between CFRP cross ply laminates and mild steel have been performed and studied using LMI. Initial findings will be reported at the meeting and results compared with predictions.

**Conclusions**

Laser moiré interferometry is a relatively new technique, and has proved to be very powerful for use in the detailed study of in-plane deformation fields, and hence strain fields in adhesive bonds. In particular:

- There is good agreement with the predicted strain distributions measured using non-linear continuum mechanics analysis programs (BISEPS-LOCO) developed at AEA Harwell.

- Moiré interferometry shows that the peel strain is non-uniform through the thickness of the joint and this is especially marked close to the joint ends. It is highly localised peel strains close to the interface near the joint ends which initiate initial failure of the adhesive. Quantified through-thickness measurements using LMI show excellent agreement with predictions using FELOCO.
Figure 1 Average shear and peel strain as a function of position in bonded lap joint

Figure 2 Moire displacement fringes in a thick bondline
Figure 3 Shear strain as a function of position along the bond line for various layers

Figure 4 Experimental peel strains through bond thickness near the free edge

Figure 5 Comparison of predicted and experimental peel strains along bond length
Figure 6 Comparison between FELOCO and LMI results
Aspects of Adhesively Bonded Joints

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1. Introduction

The excellent performance record which has been established by composites over the last 30-40 years is acknowledged by an ever-increasing commitment to the creation of larger structures which must function effectively for prolonged periods.

Adhesive bonding is one method which can be used to assemble the parts and it is known to be very successful. However, in a world where production costs are forever under pressure it is essential to know the limiting parameters eg. design and long term performance. The presentation will discuss these basic issues in relation to bonding pipes and pultrusions.

In the case of steel components, the welded joint can be easily checked by non-destructive methods, ultrasonics or dye penetrants for example.

Thermoplastic components can be welded, using a range of available techniques, including hot plate, friction, ultrasonic, and resistance welding.

In the case of thermosetting plastics, however, where welding is not possible, the parts must be adhesively bonded together. There is then no recognised non-destructive method for checking the quality of the joint. This is a considerable disadvantage, which is also experienced with composite materials.

In addition, the long term performance of adhesively bonded joints can often lead the design engineer to avoid the use of adhesives. Thus, information is required to persuade the designer that the long term durability of the adhesively bonded joint is adequate. But how can one determine the ageing behaviour of a bonded joint?

This presentation will highlight the potential for using adhesives by reference to good design principles, composite pipes, and then extend these concepts to pultrusions.

2. Composite Pipes

The absence of ageing experience for adhesively bonded joints which operate permanently at high temperature is holding up the development of composite pipes for heating systems.
Table 1. The advantages and disadvantages of a welded joint on a steel pipe with those of a bonded joint on a composite pipe.

<table>
<thead>
<tr>
<th></th>
<th>STEEL PIPE WELDING</th>
<th>COMPOSITE PIPE BONDING</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>ADVANTAGES</strong></td>
<td>Absence of pre-treatment</td>
<td>Absence of corrosion problems</td>
</tr>
<tr>
<td></td>
<td>Butt jointing</td>
<td>Joints possible with pipes of other materials</td>
</tr>
<tr>
<td></td>
<td>Automatic welding possible</td>
<td>Manipulation still possible after application of the adhesive</td>
</tr>
<tr>
<td></td>
<td>X ray examination of the welded joint</td>
<td></td>
</tr>
<tr>
<td><strong>DISADVANTAGES</strong></td>
<td>Post welding: varnishing of the welded joints</td>
<td>Pre-treatment necessary</td>
</tr>
<tr>
<td></td>
<td>Welding specialists needed</td>
<td>Curing time slightly longer than welding time for some adhesives</td>
</tr>
<tr>
<td></td>
<td>Risk of corrosion at the welded joint</td>
<td>Absence of recognised NDT method</td>
</tr>
<tr>
<td><strong>AGEING EXPERIENCE</strong></td>
<td>Available</td>
<td>Lack of ageing experience</td>
</tr>
</tbody>
</table>

Under a given water pressure, at a fixed temperature, the time required to obtain a crack can be measured. The extrapolation of such data, to longer time periods, is not possible, because the scatter of the data is too great. The presentation contemplates a method which appears to provide an answer to the problems raised and thus gets around this difficulty. The adhesively bonded joint was tested at constant temperature with the pressure increased in stages, at fixed times.

In the data, the pressure and times are given in logarithmic scale, the crack observations are indicated. It can be seen that a law appears, allowing extrapolation of the results to extremely long periods.

Of course, heat is not the only enemy of adhesives, extreme cold can also be encountered. Construction of the Alaskan pipeline required an adhesive that could withstand the stresses of Arctic conditions while bonding integral parts of the pipeline. The adhesive is required to join steel sheets to fibreglass, the major elements of the insulation panels. The specific manufacturing demands include:

* Application by automatic spraying onto only one surface, the porous fibreglass.
* Consistency that would ensure the retention of the adhesive on the glass fibres rather than settling in between them.

* Bond development within four minutes.

* Immiscibility with water.

Conventional insulation adhesives were inadequate and a rubber based solvent borne adhesive was developed.

3. Pultrusions

Pultrusions are predominantly axially reinforced and have transverse and shear properties that are poor in comparison with their axial properties. Consequently, load diffusion into these materials requires care, and adhesively bonded joints should be considered, to minimise stress concentrations. This reduces the tendency towards shear failures that can occur when using mechanically fastened joints for joining composites. In spite of this, many examples of bolted pultrusions are visible, and the presentation will highlight some of these. Alternative joining methods will be considered, with reference to specific examples.

4. Concluding Remarks

Large composite structures, particularly those based on thermoset resins can be readily and durably bonded, both to themselves and associated metal structures.