A98-31589

ICAS-98-4,4,3

THE EFFECT OF MATRIX MOISTURE CONTENT ON THE REPAIR OF CARBON FIBRE REINFORCED EPOXY

Flight Lieutenant David Bond & Flight Lieutenant Graeme Swan
ASI1-DGTA SCA(AF)
RAAF Williams
Victoria AUSTRALIA

Professor Mike Bader & Professor Paul Smith
Department of Material Science and Engineering
University of Surrey
Guildford SURREY UK

Abstract

Carbon fibre reinforced epoxy composites inevitably contain moisture absorbed from the atmosphere. Heating such composites to cure adhesively bonded repairs encourages diffusion of some of this moisture into the bonding adhesive. Here, the absorbed moisture may affect the integrity of the repair by causing; excessive adhesive voiding, adhesive plasticisation, interference to the adhesive cure and/or interference to the formation of an effective substrate-adhesive bondline. Thorough drying of composite substrates is usually recommended to avoid these possibilities. This research investigates the extent of drying required to restore substrate moisture free joint strengths. The strengths of joints constructed from substrates conditioned to various equilibrium moisture contents are compared to those of joints similarly conditioned, but which were dried for varying times prior to bonding. The pre-dried joints are shown to perform better (for a given substrate pre-join moisture content) than the undried joints. A model is developed to estimate the amount of moisture desorbed from the substrates to the joint adhesive and the strength of the joints is shown to be related to this quantity of moisture.

Introduction

Background

The fact that moisture levels of up to 1% weight to weight (w/w) may be found in CFRE composite structures exposed to the environment has been extensively (1, 2) reported, as well as the effect of the moisture on CFRE properties (1, 3). Should structures made from such materials be damaged, a more critical effect of this pre-join moisture is its effect on the 'repairability' of the structure. The moisture will diffuse out of the CFRE substrate should the activity of moisture in the surrounding environment be less than that within the CFRE. The heating of the repair area or joint succeeds in not only curing the adhesive but also in accelerating any diffusion of moisture out of the substrates by increasing the moisture diffusion coefficient of the composite.

Part of the moisture diffused out will exit to the atmosphere, some will be absorbed by the adhesive and some may locate itself at the adhesive/substrate interface. The adhesives used in repair applications are generally stored under 'cold-dry' conditions and consequently contain little moisture. They then readily absorb moisture from the substrate under cure conditions. The elevated temperature of cure initially reduces the viscosity of the adhesive to allow it to wet the substrate surface. This brings about an increased mobility of penetrant molecules within the adhesive and allows absorbed water molecules to move rapidly through the curing adhesive. The water emanating from the substrates may affect the adhesive in three ways:

(a) the moisture may cause the production of an excessive amount of voids (4, 5),

21st ICAS Congress
13-18 September 1998
Melbourne, Australia

(b) the cure reaction of the adhesive may be affected by the presence of the moisture (6) and,

(c) the moisture may cause plasticisation of the adhesive by interfering with the density of intra-molecular bonds formed in the cure process.

These mechanisms all reduce the load carrying capacity of the cured adhesive (7), although the latter mechanism may be able to be reversed by post cure drying of the adhesive/joint. The interface too may be affected by the moisture, obviously again through dissection of interfacial bonds but also through an interference with the actual formation of these interfacial links (8).

When CFRE substrates are heated for repair the pre-join moisture not only causes deterioration of the adhesive and the adhesive/substrate bond but it may also cause delamination and blistering of the substrates (9). This is the result of the moisture absorbed, vaporising as a consequence of the elevated cure temperatures. The increased vapour pressure being sufficient to cause internal stresses to reach sufficient magnitude to cause inter and intra lamina cracking in the substrate.

The common solution to the problems caused by the presence of this moisture is to thoroughly dry any CFRE components before repair. The recommended level to which CFRE components should be dried varies between 0.3% (10) and 0.5% w/w (11). As drying temperatures are normally kept below 100°C to avoid vaporisation and the components may be quite thick, drying to these low moisture levels may take considerable time. The moisture content of a component is usually unknown and typically a worst case estimate is used (9) contributing to the use of extended drying times.

The acceptance that a certain amount of moisture may be tolerated in substrates to be repaired is the result of empirical observations. The level of moisture being a function of many parameters including substrate and adhesive sorption characteristics, joint design, cure conditions and surface pre-treatment. Only recently however, has the notion been investigated that it is not the moisture content of the component that is the critical parameter rather, the concentration of moisture in the outer substrate plies (11, 12).

Joint Strength

The strength of adhesive joints manufactured from moisture conditioned substrates has been shown to decrease with increasing substrate equilibrium moisture content (8, 9, 11, 12). Typically, adhesives cured at 120°C exhibit more susceptibility to the effects of substrate moisture than those cured at 170°C (8, 9) as shown in Figure 1 . Note that the various adhesives investigated by Parker (8) all exhibited markedly different 0% laminate pre-bond moisture content strengths but similar reaction to the increasing presence of moisture.

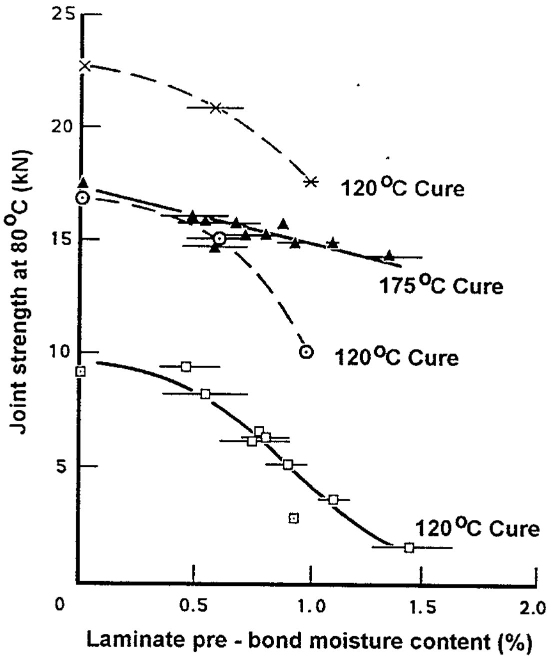

Figure 1 - The effect of substrate pre-bond moisture content on joint strength of differing temperature cured adhesives (8)

The reductions in joint strength of the adhesives shown in Figure 1 were reported to be contributed to by all of the possible mechanisms mentioned in the background of this paper. Some adhesives showed little or no increases in bond line void content or interfacial failure suggesting adhesive plasticisation as the dominant mechanism, while others exhibited increases in one or both of the former bondline defects.

Robson et al (11) incorporated the proposal of Parker (12) that the surface moisture content is the critical parameter affecting joint performance rather than the overall substrate moisture content. The results of Figure 2 and

Figure 3 show a marked reduction in scatter when joint strength is compared to the estimated surface moisture level (based upon diffusion calculations and pre-join storage environment). These results suggest that for the adhesive/substrate combination studied by Robson et al (11) the moisture content of the surface layers must be reduced to ≤ 1.2% to produce a joint of essentially dry substrate quality.

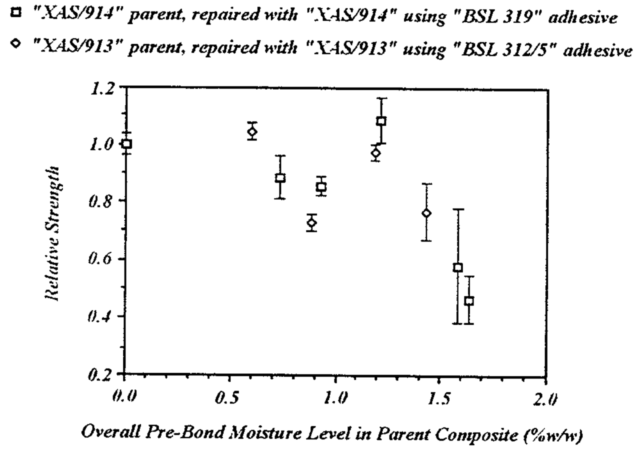

Figure 2 - The effect of substrate moisture content on scarf joint strength (11)

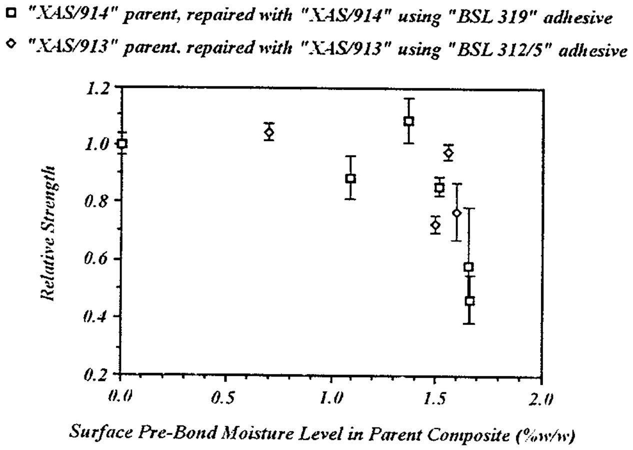

Figure 3 - The effect of substrate surface moisture level on scarf joint strength (11)

Modelling the effect of pre-join moisture

Modelling the effect of pre-join substrate moisture has typically been adopted from the methods developed to account for the effects of hygrothermal ageing on polymers. Currently such models are only capable of accounting for the plasticisation effects of the moisture as the effects of the moisture on adhesive cure and interfacial degradation have yet to be modelled. Augl (4) and Donnellan (5) have produced models based upon Fickian diffusion to predict void formation and growth albeit with considerable simplifying assumptions.

Materials, samples and test procedures

Torayca T800 carbon fibres embedded in Ciba 924 epoxy (T800/924) was used for all joint specimen substrates. The CFRE laminates were supplied by the Defence Research Agency (DRA-Farnborough) and were approximately 2.05 mm thick, of unidirectional $[0°]_{16}$ lay-up and had a fibre volume fraction of ≈ 0.58 (1).

Two adhesives were used to bond the substrates:

(a) Redux 312/5 film adhesive, manufactured by Ciba Composites and supplied courtesy of DRA (Farnborough) - a high strength 120°C cure modified Bisphenol-A epoxy resin intended for use in high temperature environments. It contains modified dicyandiamide and a woven nylon carrier cloth and has an expected gel time of 30 minutes (13).

(b) 3M Scotch-Weld AF-163-2 film adhesive, supplied by 3M United Kingdom PLC. (Manchester) - a variable temperature cure modified epoxy resin intended for use in high temperature and humidity environments. The form used in this research included a knitted nylon carrier cloth and at 120°C cure temperature had an expected gel time of 20.5 minutes (14).

Both adhesives were stored at room temperature in a 0% RH desiccator for approximately one week prior to use. This period of time was found to be sufficient to remove the 0.20%w/w (approx.) moisture inherent in the adhesives. The manufacturers of both adhesives specify that this time is well within acceptable room temperature shelf lives.

Dimensions and construction

Investigations into the effect of moisture on bonded repairs have typically used a form of modified scarf joint originally proposed by Myhre et al (15, 16). The modified scarf joint is typical of a depot-level aircraft repair scheme for a thick adherend and consequently performs well under even the most extreme conditions, frequently resulting in failure of the substrate as shown by Robson et al (17). Substrate failure is ideal for an aircraft repair however, it does hide the effect moisture exposure has upon the integrity of the adhesive bond. Consequently, a simple scarf joint was chosen as the specimen geometry for this research since it provides the best combination of reduced eccentricity, simplified stress analysis and ease of manufacture (See Figure 4). The dimensions of the scarf were chosen to be less than that required for substrate failure to ensure that all joints would fail in the region of the adhesive.

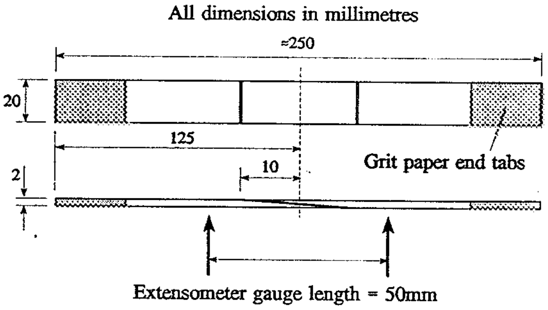

Figure 4 - Scarf joint dimensions

Specimen substrate moisture contents

The equilibrium moisture content of a CFRE is proportional to the relative humidity of the environment the composite is in (1,18). The specimen substrates were conditioned to various equilibrium levels by storing them in environments with various relative humidities (controlled by saturated salt solutions). Moisture content was established gravimetrically using a microbalance accurate to the nearest 10μg (typical substrate weight was approximately 12g). Each substrate was accompanied by a traveller coupon. The moisture content of the coupon was assumed to be representative of that of the substrate once the substrate had been used in a joint and was thus unable to be independently weighed. After bonding, all joints were stored in a 0%RH environment until the relevant traveller coupons indicated that the substrates were essentially 'dry' (ie. ≈ 0% moisture content) in which condition the joints were tested.

Surface pre-treatment and cure of joints

Prior to bonding the scarf surfaces were cleaned by:

(a) a light hand abrade with 1200 grade grit paper and then

(b) a thorough degrease with isopropanol.

Once cleaned, the joints were mounted in a jig to ensure stability during cure. The jig was then placed into a temperature controlled oven pre-heated to 60°C and the required adhesive cure pressure (185 kPa) applied. The oven was heated to 120°C at 3°C/min and held at that level for 5 hours to ensure full cure of the adhesive.

Tensile testing

Tensile testing of the manufactured scarf joints was conducted in accordance with ASTM STP D3528. The tests were conducted in Laboratory conditions using an INSTRON 1175 tensile testing machine to load the joints monotonically in tension. The rate of cross head movement was set to 0.5 mm/min. At failure the joints were immediately stored in re-sealable bags to preserve failure surfaces for future analysis.

The effect of pre-drying on joint strength

The principal aim of the research presented in this paper was to establish a protocol for the drying of CFRE substrates prior to repair by adhesive bonding. Before considering the effect of pre-drying, it is important to review the existing philosophy on the effect of substrate pre-join moisture content and the existing processes considered to alleviate the detrimental effects of this source of moisture. The RAF and the RAAF both suggest extensive substrate drying (at least 24 hours) to remove a significant proportion of the substrate moisture prior to repair. This solves the problem of the pre-join moisture content by removing it. However, Bond (1) has shown that in joints manufactured from T800/924 substrates bonded by either Redux 312/5 or Scotchguard AF-163, removal of all the moisture seems unwarranted as these adhesives are capable of demonstrating dry condition strength up to substrate equilibrium pre-bond moisture levels of around 1.2%w/w (which is higher than any reported in-service moisture content of a CFRE). This suggests that drying for twenty four hours or until the moisture content reaches some acceptable level may be excessive. It must be noted however, that the observations reported in this paper are based on the results of tensile testing joints stored in an ideal 0%RH environment. The effect of the substrate pre-bond moisture on joint durability in an operating environment would probably require a lower moisture level than that determined by the ideal tensile tests.

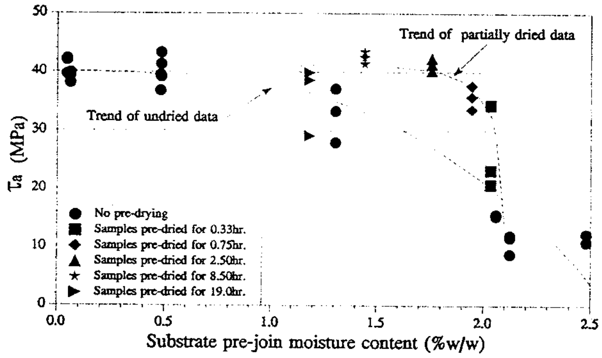

Figure 5 - Shear strength of joints using substrates with both uniform (un-dried) and non-uniform (dried) moisture distributions (Redux 312/5)

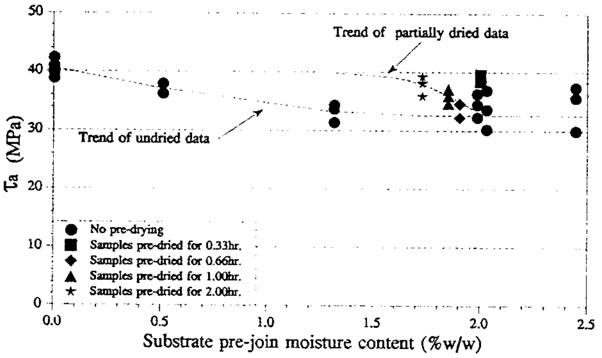

Figure 6 - Shear strength of joints using substrates with both uniform (un-dried) and non-uniform (dried) moisture distributions (Scotch-Weld AF-163)

Parker (12) and Robson et al (11) suggest that the effect of the pre-bond moisture is determined not so much by the overall moisture content of the substrates but by the moisture concentration near the substrate surface. Robson et al (11) investigated this proposal analytically however, there exist some significant inconsistencies with their results. Critically, they modelled the diffusion of moisture in the scarf with a series of one dimensional (through thickness) 'diffusion sections'. There is nothing wrong with this approach and it will be confirmed later in this paper that it is both simple and reasonably accurate, provided acceptable boundary conditions for the moisture diffusion equations are applied. The boundary conditions chosen by Robson et al (11) were unrealistic however, as they assumed that the moisture flux through the surface of the sample was zero once the samples were removed from the conditioning environment and stored in sealed bags. This resulted in the model calculating sample moisture distributions with non-constant surface moisture concentrations. This is quite clearly in opposition to the Fickian diffusion mechanics used to model the transport of moisture within the samples and suggests that the conclusions drawn from their theoretical investigation may not be totally accurate. The appropriate situation to model would have been one in which the moisture within the sample would flow either out to or in from the bagged environment to reach a condition where the entire surface would be at the same moisture concentration level as dictated by the sorption isotherm of the CFRE substrates and the equilibrium moisture activity within the bag.

With some doubt cast on the theoretical analysis of Robson et al (11), further evidence to invalidate the idea of a relationship between surface moisture content and joint performance may be obtained by considering joints initially conditioned to an equilibrium moisture level prior to being pre-dried for varying periods of time before bonding. The pre-drying reduces the surface moisture content of the substrates to zero so that the joint performance of these samples should be the same if the surface moisture content is the critical parameter. Figure 5 and Figure 6 show that this is not the case and that the joint strength varies with the amount of pre-drying applied to the substrates. Equally obvious from these figures is the fact that the joint strength is not related to the pre-join moisture content of the pre-dried joints. The pre-dried substrates were initially conditioned in an 85°C/96%RH environment and dried prior to bonding in an 85°C/0%RH environment for the times indicated in the two figures. In both the Redux 312/5 and the Scotch-Weld AF-163 bonded joints the dry substrate joint performance is shown to be regained by drying the substrates for as little as one hour.

Analysis of failure surfaces

The failure surfaces of the scarf joints were inspected using a Zeiss Axiophot photomicroscope. The quality of the adhesive was found to be affected significantly by the quantity of moisture desorbed from the substrates and appeared more 'crumbly' the higher the pre-join moisture content of the substrate (for the moisture equilibrated specimens). Interestingly, in the Scotch-Weld AF-163 adhesive, this degradation did not cause significant loss of joint strength. The extent of interfacial failure was also found to increase the higher the equilibrium level of pre-bond moisture in the substrates, leading to the samples immersed in water prior to bonding exhibiting almost total interfacial failure.

Examination of the adhesive void volume fraction from joint cross section photomicrographs showed an increasing fraction both for joints with higher initial substrate moisture levels and those subjected to shorter drying times. The quantity of through thickness void debonds was also found to increase with pre-bond substrate moisture content. The Redux 312/5 debonds were regularly arranged within the confines of the knitted carrier cloth and the larger voids were often the same size as the yarn spacing. The Scotch-Weld AF-163 through thickness voids were generally much smaller than those of the Redux 312/5. These observations confirm that the adhesive void volume fraction is affected by moisture desorbed from the substrates during the cure process.

Modelling moisture lost to the adhesive

This paper proposes that the performance of the specimen joints is affected by the amount of water absorbed by the adhesive up to its gel point when curing. With a 120°C cure oven and dry substrates the moisture absorbed would be effectively zero (the high temperature oven creating a very low activity environment). As the moisture content of the substrate increases, more moisture will be available for absorption by the adhesive. This process, while simplified by the knowledge that the moisture in the substrate will diffuse essentially in accordance with Fickian predictions (1), is extremely complex with many variables affecting the amount of water absorbed by the adhesive. Some of the more important variables include:

(a) the variation of adhesive sorption characteristics (most critically diffusion coefficient and sorption isotherm) as it melts and then begins to form cross-links while curing,

(b) the non-uniform thickness of the scarf and the effect that this has upon the moisture distribution and the consequential variation in moisture lost to the adhesive and

(c) the transport of moisture through an interface from one material to another with differing sorption characteristics. (This problem has been addressed in simple multi-material specimens by considering the variation of local to equilibrium moisture concentration as opposed to actual moisture concentrations and by varying the effective thicknesses of the materials to account for the different diffusion coefficients (19, 20).)

A review of available literature suggests that the concept of assessing the effectiveness of an adhesive joint by the quantity of moisture absorbed from the substrate has not previously been considered. Consequently attempts were made to overcome the difficulties presented in the previous paragraph to estimate an effective quantity of moisture absorbed by the adhesive under cure conditions. Insufficient knowledge of the sorption characteristics of the adhesive when curing has, so far, prevented such a technique being developed. What is considered in this research though, is a simplified model of the above process with the following assumptions made:

(a) the adhesive starts from an equilibrated moisture distribution of 0%,

(b) the adhesive is capable of absorbing all of the moisture supplied by the substrates at the substrate-adhesive interface,

(c) the adhesive absorbs the moisture at a rate sufficient to allow the desorption of moisture from the substrates to be considered well modelled by desorption to a 0%RH environment, and

(d) the amount of moisture desorbed by the substrates may be considered to be equal to the sum of one dimensional through thickness and lengthwise diffusion sections.

The first assumption is reasonably valid as the adhesives were stored in a 0% RH desiccator for approximately one week prior to bonding and in this time they were found to equilibrate to an apparent 0% moisture content. The second assumption is the most significant, for while it does allow calculation of a moisture content absorbed by the adhesive it has shortfalls that should eventually be eliminated. While not unreasonable to assume that the adhesive is capable of absorbing all of the moisture desorbed by the substrate in a time up to its gel point (a fact that will be supported later) ignoring the effect of the moisture distribution within the adhesive will, for higher moisture levels, lead to an over prediction of the moisture absorbed by the adhesive. This is explained by acknowledging that when the adhesive approaches its own equilibrium level (whatever that may be in a curing adhesive) the effective concentration gradient of local to equilibrium moisture content will reduce the amount of moisture flux through the adhesive/substrate interface and consequently the quantity of moisture absorbed by the adhesive. This effect must be born in mind when considering the results of the following analysis which utilises a corollary of assumption (b); that the total amount of moisture desorbed from the substrate up to the gel point of the adhesive is absorbed by the adhesive.

The third assumption has already been used by several researchers with some success to account for sorption performance of CFREs and polymers to determine edge diffusion correction factors (18) and moisture distributions (17, 21). The accuracy of assuming that the moisture desorbed by the substrates is equal to the sum of separate y and z axes one dimensional flow (as shown in Figure 7) may be considered by examining the rate of moisture loss in the substrates used for the pre-drying investigation.

The moisture lost in those samples may be represented by the one dimensional summation using the notation of Figure 7 as:

$$\Delta m(t) = \sum_{0}^{g} \delta m_z(D_{22}, V_y, t) + \sum_{0}^{h} \left(\delta m_y(D_{11}, V_z, t) + \delta m_x(D_{11}, V_y, t) \right) \quad (1)$$

where Δm represents the total moisture loss of the sample, g the width of the substrate, h the thickness of the substrate and $\delta m(D,V,t)$ the axial moisture losses in the small one dimensional 'diffusion section' shaded in Figure 7 . The axial moisture losses are related to the diffusion coefficients along these axes (D), the dimensions of the diffusion section (V) and the time of drying (t).

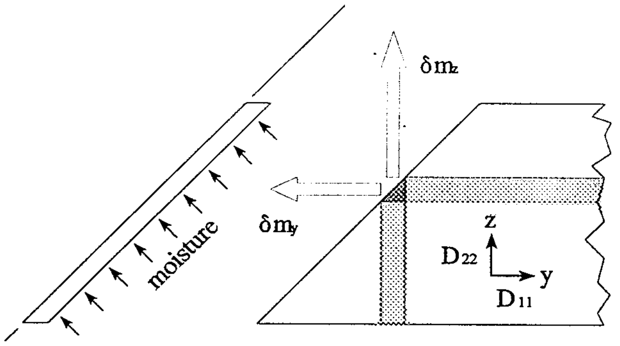

Figure 7 - Combined one dimensional analysis of the moisture desorbed by the CFRE substrates to the adhesive during cure

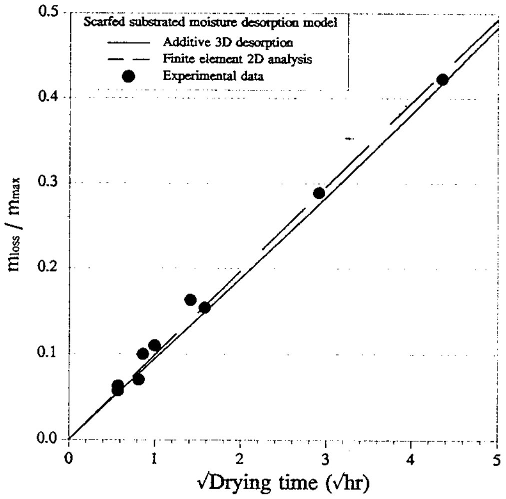

Figure 8 - Recorded and estimated moisture loss to equilibrium moisture content ratio for pre-dried substrate specimens

Figure 7 shows that the moisture desorbed to the adhesive from a single substrate is a sum of half the moisture lost along the y axis and half the moisture lost along the z axis in the region of the scarf (the other halves being lost to the atmosphere). Figure 8 shows that equation (1) is within acceptable limits of accuracy when compared to both a two dimensional linear exact solution obtained from FE analysis and the reported experimental moisture losses.

Within the accuracy of the assumptions considered, the moisture loss from a substrate was then determined for the exposure profile shown in Figure 9 . The profile includes a variable drying time (at 85°C/0%RH) and a surface preparation period of 20 minutes (at 25°C/50%RH) prior to the jigged joint being placed into the cure oven (pre-heated to 60°C) in which it is heated to 120°C (at 3°C/minute). The joint is held at this temperature for the gel time of the respective adhesive (30 minutes for Redux 312/5 - 20.5 minutes for Scotch-Weld AF-163 (13, 14)).

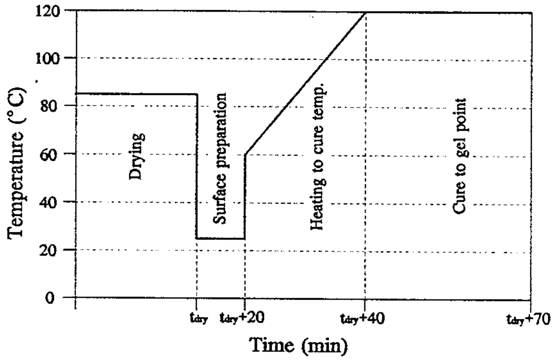

Figure 9 - Environmental exposure profile for theoretical estimation of substrate moisture loss to the adhesive (30 min gel time - Redux 312/5)

The model allows the moisture desorbed to the adhesive to be calculated for the three differing substrate combinations used in this research:

(a) identically equilibrated substrates,

(b) equilibrated substrates at different moisture contents and

(c) partially dried substrates with non-uniform moisture distributions.

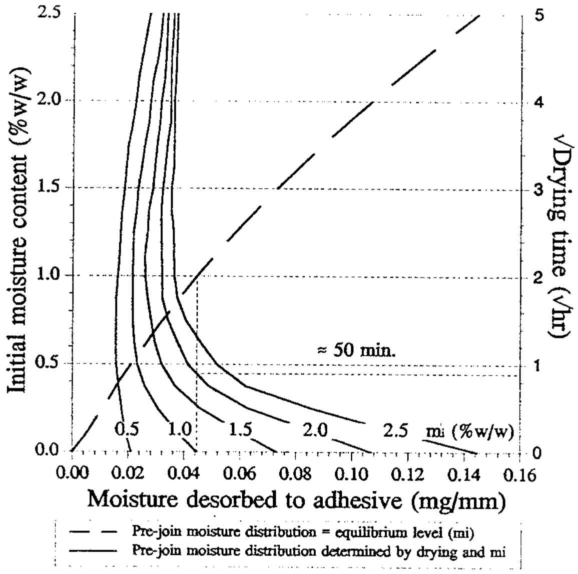

Figure 10 - Comparison between moisture desorbed to the adhesive from equilibrium conditioned samples with no pre-drying and those with varying pre-drying time

The results for the 30 minute gel time Redux 312/5 profile are used for the discussion of the results of the modelling. Figure 10 shows the moisture desorbed from varying initial equilibrated substrate moisture contents (dashed line - left hand axis for initial moisture content) and the moisture desorbed from substrates of specific initial moisture contents (m_i) pre-dried for varying times (solid lines - right hand axis for drying time).

Using the knowledge that Redux 312/5 bonded joint strengths are maintained up to a moisture content of at least 1%w/w (1), Figure 10 shows that for a substrate initially conditioned to 2.0%w/w a drying time of less than 1 hour is required to reduce the moisture desorbed to the adhesive to a similar level (≈ 0.045mg.mm). Using the proposal that it is this parameter which affects joint strength this suggests that the 50 min dried joint would perform to a similar level as the 1.0%w/w equilibrated substrate joint. This result is confirmed by Figure 5 and adds significant support to the theory proposed in this research. The results of Figure 10 highlight the ineffectiveness of drying samples for any longer than 4 hours (2 hrs$^{1/2}$). Additional drying acts only to reduce the moisture distribution in the sample to a level where the absorbtion of moisture from the surface preparation environment is facilitated. This moisture absorbed is located in the worst possible region, immediately at the surface of the sample, and is thus readily desorbed to the adhesive during cure. This suggests that there is an optimal drying time for all CFRE substrates about to undergo adhesive bonding and that longer drying times are not necessarily better. This is especially so in very thick substrates or in areas where the exposure to the surface preparation environment is for a long time or in hot wet conditions (ie. for repair of a wing component on a tarmac in a tropical region).

Joint performance as a function of moisture desorbed to the adhesive

Now that an assessment of the moisture desorbed to the adhesive in the joints (per unit width of joint) is available, the variation in joint strength may be examined in terms of this parameter. Figure 11 and Figure 12 suggest that the joint strength decreases proportional to the quantity of desorbed substrate moisture (ie. the moisture absorbed by the adhesive) until a levelling off; in the case of the Redux 312/5 adhesive at around 10 MPa and for the Scotch-Weld AF-163 at around 35 MPa. At this point, the concerns regarding the second and third assumptions made to develop this model must be considered. In the higher moisture content substrates it is unlikely that all the moisture these substrates are able to desorb is absorbed by the adhesive at the same rate that a 0%RH environment could absorb it. It would be expected that the higher moisture content results (ie. the higher quantities of moisture desorbed) over estimate the moisture absorbed by the adhesive. This suggests that a direct relationship may exist between the moisture absorbed by the adhesive from the substrates and the shear strength of the joints based on the lower moisture desorption data. Figure 11 and Figure 12 show excellent correlation between samples with moisture equilibrated substrates and substrates pre-dried from equilibrium levels around 2.1%w/w.

Examination of the quantities of moisture desorbed show that a loss of 0.12 mg/mm of moisture per joint substrate width corresponds to an absorbed moisture content for the adhesive of around 2.2% which is slightly above the recorded moisture content for the cured adhesive in hot/wet conditions (22). This is thought then to be a realistic expectation for the amount of moisture the adhesive is able to absorb considering that in the curing condition the adhesive is well above its glass transition temperature and that the molecular freedom in this condition would allowed increased penetrant uptake.

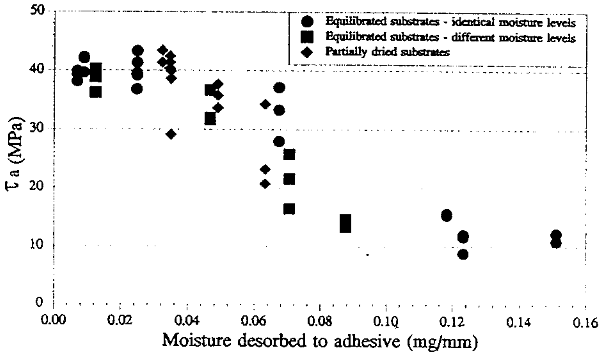

Figure 11 - Joint shear strength as a function of moisture desorbed from the substrates to the adhesive - Redux 312/5 (per unit width - mg/mm)

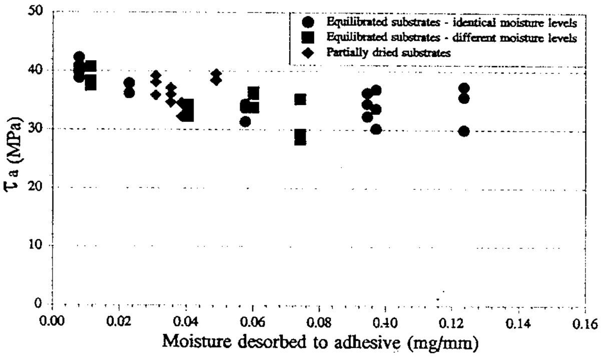

Figure 12 - Joint shear strength as a function of moisture desorbed from the substrates to the adhesive - Scotch-Weld AF-163 (per unit width - mg/mm)

Conclusions

The results of this research indicate that the presence of moisture in CFRE substrates has a significant effect on the integrity of joints formed with these materials by adhesive bonding techniques.

Examination of failure surfaces of joints manufactured from both adhesives showed that the quality of the adhesive was degraded by increasing substrate equilibrium pre-join moisture content. This corresponded with increases in adhesive porosity and interfacial failure of the joints. The results of the tensile testing and the failure surfaces suggest that the degradation mechanism most affecting the performance of the joints is associated with a reduction in the integrity of the substrate/adhesive interface.

Increasing levels of pre-join substrate moisture have a significant effect on the performance of Redux 312/5 bonded joints. Scarf joint strength is reduced from 40 MPa to around 10 MPa for a pre-join moisture content of around 2.3%w/w (obtained by water immersion). Scotch-Weld AF-163 adhesive is much less affected by pre-bond moisture than the Redux 312/5 and experiences a maximum of only 5-10 MPa reduction from the dry conditioned joint strength of 40 MPa (≈20%) for pre-join substrate moisture contents above 1.0%w/w.

Drying the substrates prior to bonding, was found to significantly improve performance of the joints of both adhesives. Pre-drying for as little as one hour in an 85°C/0%RH environment after conditioning in an 85°C/96%RH environment to an initial moisture content of approximately 2.1%w/w restores the strength of the scarf joint to the 40 MPa of the fully dry joints from the reduced 10-15 MPa (Redux 312/5) or 30-35 MPa (Scotch-Weld AF-163) shear strengths of un-dried joints. These results were seen to support the proposal that the critical parameter affecting the strength of joints bonded from moisture pre-conditioned substrates is the quantity of moisture absorbed by the adhesive not, as some previous research had proposed, the moisture content or surface concentration of the substrates.

A model was developed to estimate the amount of moisture desorbed from the substrates to the adhesive and allowed the time, temperature and humidity of the drying environment to be varied. Comparing the results of the experimental program with the prediction of the model and taking into account the limitations of the assumptions on which the model is based, the suggestion is made that the strength of an adhesive joint is directly related to the quantity of moisture desorbed from the substrates into the adhesive. The model is capable of considering substrates of differing moisture contents and distributions and is effective at reducing them all to a single relationship with joint strength. The results from the model also suggest that extended pre-drying of substrates is not beneficial to the performance of the joint. With the relevant substrate material sorption characteristics and the proposed geometry of a joint, the model is capable of predicting optimum substrate drying conditions/time to eliminate the effects of substrate pre-bond moisture.

References

1. Bond D.A., **PhD Thesis**, University of Surrey, Guildford, UK, 1996.

2. Hedrick I.G. & Whiteside. J.B., **Effects of environment on advanced composite structures**, AIAA Conference on Aircraft Composites: The emerging methodology for structural assurance, AIAA 77-463, 1977, pp 1-17.

3. Wright W.W., **The effect of diffusion of water into epoxy resins and their carbon-fibre reinforced composites**, Composites, July, 1981, pp 201-205.

4. Augl J.M., **Moisture Transport in composites during repair work**, 28th National SAMPE Symposium, Apr 1983, pp 273-286.

5. Donnellan T.M., Williams J.G. & Trabocco R.E., **Void formation in Adhesive Bonds**, 15th National SAMPE Technical Conference, Oct 1983, pp 478-488.

6. Paradis D.L., **The influence of moisture on adhesive film performance** Structural Adhesives and Bonding Conference, California, 1979, pp 110-118

7. Kinloch A.J., **Adhesion and Adhesives**, Chapman and Hall, 1987.

8. Parker B.M.. **Some effects of moisture on adhesive-bonded CFRP-CFRP joints**, Composite Structures, Vol 6, 1986, pp 123-139.

9. Myhre S.H., Labor J.D. & Aker S.C., **Moisture problems in advanced composite structural repair**, Composites, Vol 13, Jul 1982, pp 289-297.

10. Baker A.A., Chester R.J., Hugo G.R. & Radke T.C., **Scarf repairs to graphite/epoxy components**, AGARD-CP-550, Composite Repair of Military Aircraft Structures, 1994, pp 19(1)-19(12).

11. Robson J.E., Matthews F.L.,& Kinloch A.J., **The bonded repair of fibre composites:effect of composite moisture content**, Composite Science and Technology, Vol 52, 1994, pp 235-246.

12. Parker B.M., **The effect of composite pre-bond on adhesive-bonded CFRP-CFRP joints**, Composites, Vol 14, Jul 1983, pp 226-232.

13. Ciba-Geigy, **Redux®312**, Information Sheet No. RTA 312d, August 1989.

14. 3M Scotch-Weld, **Structural Adhesive Film AF-163-2**, Aerospace Technical Data, Issue No.1, March 1986.

15. Myhre S.H. & Beck C.E., **Repair concepts for advanced composite structures**, Journal of Aircraft, Vol 16. No. 10, 1979, pp 720-728.

16. Myhre S.H. & Labor J.D., **Repair of advanced composite structures**, Journal of Aircraft, Vol 18, No. 7, 1981. pp 546-552.

17. Springer G.S., **Numerical Procedures for the Solution of One Dimensional Fickian Diffusion Problems**, in Environmental Effects on Composite Materials, Chpt. 13, pp 166-199.

18. Miller R.K., **Multi-Material Model Moisture Analysis for Steady State Boundary Conditions**, Journal of Composite Materials, Vol 15, Mar 1981, pp 142-150.

19. Shen C.H. & Springer G.S., **Moisture absorption and desorption of composite materials**, Journal of Composite Materials, Vol 10 Jan 1976, pp 2-20.

20. Robson J.E., Matthews F.L. & Kinloch A.J., **The effect of a hot-wet environment on bonded composite repair joints**, ICCM 9, Vol 5, 1993, pp 453-460.

21. Robson J.E., **The repair of composite materials**, PhD thesis, University College of Science, Technology and Medicine, University of London, 1993.

22. DRA Technical Report, DRA/SMC/CR951184