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EFFECTS OF SURFACE PREPARATION ON LONG-TERM DURABILITY OF COMPOSITE ADHESIVE BONDS

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SUMMARY: The evaluation of adherend surface preparation techniques on the long-term durability of composite bonded joints is addressed. Several potential factors are evaluated, concentrating on the effects of peel plies, release fabrics, grit blasting, and paste versus film adhesive on the strength, durability, and failure modes of adhesively bonded composites. Materials and configurations studied are typical of commercial and general aviation (GA) aircraft. Alternate forms of the double cantilever beam (DCB) test and a travelling wedge test were developed and used for evaluating bond integrity and failure characteristics. The research is to aid the interpretation of a form of the static wedge test where the usual aluminum adherends are substituted by composite adherends.

DCB results have shown that adherends cured against coated nylon release fabrics become chemically contaminated, resulting in interfacial bond failure with low strain energy release rates (G_{Ic}). Adherends cured against PTFE vacuum bags had cohesive failures and higher G_{Ic} values. Grit blasting increased all joints' G_{Ic} values but did not change their modes of failure, even for joints with cohesive failures.

KEYWORDS: bonded joint durability, surface preparation, mode I fracture tests

INTRODUCTION

A common practice currently adopted in the GA industry for aircraft structural development is the use of full-scale structural articles. Unfortunately, this approach can limit the ability to evaluate the adequacy of critical structural details such as bonded joints. By implementing a tailored version of the building block test/analysis/fabrication philosophy that supports effective integrated product development and is used widely in the military and commercial aircraft community, valuable and key information can be obtained on bonded joint characteristics. It is suggested that the utilization of such approaches could serve to complement full-scale test information, providing aircraft developers with insight that would facilitate the design and manufacture of reliable bonded structures.

Advantages of bonding over mechanical means of fastening include higher stiffness, more uniform load distribution, cleaner lines for aerodynamic benefits, part consolidation, no holes drilled in adherends (with the resulting stress concentrations), and, generally, less labor.

Adherend surface preparation plays a critical role in the development and evaluation of bonded joints. GA tends to rely extensively on bonded joints, in part due to the lower loads typically found in smaller aircraft. Inadequate surface roughening, environmental effects, peel ply/release film chemical contamination [1], and other factors (both mechanical and chemical) can prevent adhesives from bonding properly to composites, resulting in interfacial failures. These failures occur at loads well below those of properly bonded joints that fail cohesively. Other failures can occur over time in service, as joints are exposed to harsh environments, including elevated temperature and humidity [2-11]. Basic and applied research such as that

reported herein can potentially provide greater insight and more extensive data to support increased application and confidence in bonded structures.

Initially, many possible factors that could affect an adhesive bond's durability were amassed (table 1). This research focuses on the effects of peel plies, release fabrics, and grit blasting, which all have significant effects on bond integrity and are relevant to the aviation industry.

Table 1 Potential bonding durability factors

Factor	Variables
Adherend layup	0° _[n] , quasi-isotropic, other; orientation of ply on bonding surface
Adherend material	Fiber, matrix, composite vs. metal, typical aviation materials
Adhesive filler material	Type of filler, percentage of filler
Adhesive preparation	Hand-mixed, machine-mixed, apply vacuum to remove trapped air
Bondline thickness control	Glass microbeads/silane treatment, wires, tabs/tape, applied pressure
Compressed air blowing	Pressure, gas used, bottle vs. compressor, exposure time
Environmental exposure	Temperature, humidity, exposure time, pre-bond, post-bond, under load
Grit blasting	Pressure, grit size, grit media, number of passes, speed of passes
Hand sanding	Grit size, number of passes, pressure applied
Peel ply / release fabric	Nylon/polyester/none, release agents/calendering/untreated
Solvent wiping	Acetone, isopropyl alcohol, number of wipes, applicator type

Bonds were evaluated with a test method combining appropriate features of ASTM D3433 Standard Test Method for Fracture Strength in Cleavage of Adhesives in Bonded Joints and ASTM D5528 Standard Test Method for Mode I Interlaminar Fracture Toughness of Unidirectional Fiber-Reinforced Polymer Matrix Composites. A variant on ASTM D3762 Standard Test Method for Adhesive-Bonded Surface Durability of Aluminum (Wedge Test), with a travelling, rather than static, wedge, was also used. Analytical models of test methods were performed to analyze test data and specimen configurations. Materials and processes typical of those used for aircraft are studied to quantify the relative importance of each factor's contribution to bond strength. Results can be used to provide manufacturers with bonding guidance and to assist the FAA with interpreting data for certification procedures.

TEST SPECIMEN PREPARATION

Tests were performed on IM7/8552 22-ply unidirectional adherends bonded with Hysol EA9394 two-part epoxy adhesive mixed with 0.127 mm (0.005 in) diameter glass microbeads (2.5% by weight). Adherends were cured with Chemfab VB-3 PTFE vacuum bag (VB) film on the bottom surface (tool side) and a nylon release fabric (RF) with silicone and siloxane release agents on the top side (figure 1). This layup process creates panels with different surface properties on each side. Samples were bonded in one of two configurations: RF to RF or VB to VB, with half of each group of samples grit-blasted before bonding, creating four different types of specimens. Although this sort of layup and bagging procedure, with different surfaces on both faces, is not typical of a component used in production, it was well-suited to research and evaluation. In doing so, bonds made to different surface types can be compared against each other more reliably, as all of the specimens are derived from the same panel, removing possible differences resulting from variations in specimen production.

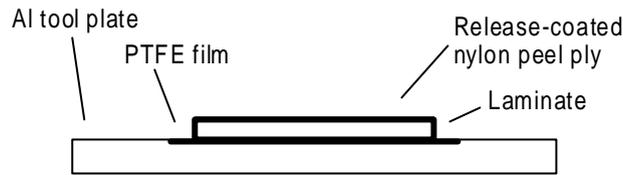


Fig. 1 Schematic layup of composite adherends

The composite samples that were grit-blasted (with Mil-A-2222B grit) were done so with a regulator line pressure of 413.7 kPa (60 psi). The hinges used to hold the DCB samples in the test machine grips were made from 0.102 mm (0.04 in) thick continuous hinge, cut to 25.4 mm (1 in) lengths and grit-blasted at 689.5 kPa (100 psi). The surfaces to which the hinges were bonded on all samples were also blasted at 413.7 kPa (60 psi) prior to bonding the hinges. The hinges were bonded to the samples with the same adhesive and bonding process used for the samples themselves. All bonded surfaces (adherends and hinges) were cleaned thoroughly with de-ionized water, oven-dried, wiped with isopropyl alcohol, then air-dried before bonding. All crack initiators were created with 76.2 mm (3 in) of flashbreaker tape on the ends of the specimens. The sides of samples were painted white and tick marks were placed manually at 1.588 mm (0.0625 in) intervals for visual crack tip observation.

Because there is a lack of standard test procedures for measuring the bond strength of adhesively bonded composite materials, a new test specimen needed to be developed. ASTM test methods cover either adhesively bonded metals or interlaminar failures in composites. The new specimen used for the DCB tests and to be used for the static wedge tests is based on those of ASTM D3433, ASTM D3762, and ASTM D5528. It is approximately 152.4 mm (6 in) long by 25.4 mm (1 in) wide, with 3.18 mm (0.125 in) thick adherends.

After the sequence of DCB tests conducted and reported herein, it was determined that a longer specimen would be beneficial to testing, as more data points can be obtained for each specimen, especially those that exhibit “stick-slip” behavior and fracture quickly under low loads. The new specimens will be 304.8 mm (12 in) long and 12.7 mm (0.5 in) wide, half the width of the ones used in this study. Very small specimen widths can affect a sample’s fracture toughness, because a significantly larger portion of the crack front tends to exhibit plane stress behavior instead of plane strain. But work by Crosley and Ripling [6] and Kinloch and Shaw [19] demonstrated that specimen geometry of these new dimensions should not affect test results.

TEST METHODS OVERVIEW

There are several standard test methods designed to measure bond strength. Traditionally, lap shear tests have been used, since bonded joints are generally designed to carry shear loads. However, Davis and Bond recently echoed previous findings that the lap shear test only verifies the short-term bond strength, and that shear and peel strength tests are not sufficient to assure durability, while the ASTM wedge test is ideal for durability measurement [12]. Furthermore, Hart-Smith had previously noted that lap shear tests tell nothing about durability and that process control, monitored by wedge tests, can help ensure long-term durability [10]. Marceau et al also reported that, for metal adherends, lap shear tests as a function of temperature, peel tests as a function of temperature, and unstressed lap shear tests under environmental exposure, do not duplicate the disbond behavior of bonded joints in service [3].

In summary, shear tests are not as good an indicator of bond durability as mode I cleavage tests. Additionally, because it is impossible to construct a purely shear-loaded mode II joint in practice (there always exists some peel component from eccentric load paths near joint edges [3]), mode I DCB tests are an appropriate test for the durability of bonded joints.

Literature research and a review of standard test methods revealed that the DCB and static wedge tests (figure 2) are well-suited to evaluating the short- and long-term durability of adhesive bonds [6, 10, 12-18]. In the DCB test, a bonded sample is pulled apart at a constant test machine crosshead velocity by fixtures (hinges or pinned blocks) at the end of the beams. The specimen is loaded and unloaded under displacement control until the crack has propagated entirely through the sample (or the specimen can be tested in one continuous load cycle without any unloading). For the wedge test, which can be performed with the same specimen configuration, a tapered metal wedge is driven into the crack opening to stimulate crack growth. Then, the sample is observed (typically in an environmental exposure chamber) while the wedge remains in its initial position, and the crack tip propagation, due to environmentally induced degradation of the crack front/bond interface, is recorded over time.



Fig. 2 DCB and wedge test methods

DCB TEST PROCEDURE AND THEORY

Specimens were loaded into an Instron 8562 test machine by clamping the hinges in grips. The test machine was run under displacement control at a crosshead speed of 0.508 mm/min (0.02 in/min) while loading the sample and at a higher rate during unloading. After visible crack propagation, the sample was unloaded and then reloaded. This process repeated for approximately each 12.7 mm (0.5 in) of crack growth. Subsequent tests omitted the unload/reload portions of the test, providing one continuous load cycle.

The test machine recorded load and opening displacement (at the free ends of the cantilever beams) while the operator noted the crack tip location visually with a telescope. There are several methods to calculate critical strain energy release rates from this load-displacement and crack length data. Blackman et al compare four such methods: the area, compliance, simple beam theory, and modified beam theory (MBT) or “displacement” methods [20]. The MBT method was used in this study, as specified by Blackman et al, Whitney et al [21], Johnson et al [22], and detailed in the ASTM D5528 test method. For comparison, the data was reduced with the area method also. The average G_c values calculated with the area method were within 9% those from the MBT method, while the standard deviations obtained with the MBT method were somewhat greater than those calculated by the area method.

The calculation methods used do not account for the adhesive layer and its interfaces. From Fernlund and Spelt's experimental findings, calculations for tests with specimens with long crack lengths relative to beam height minus bond thickness do not need to account for the adhesive [23]. The dimensions of the specimens used in this study satisfy this criterion.

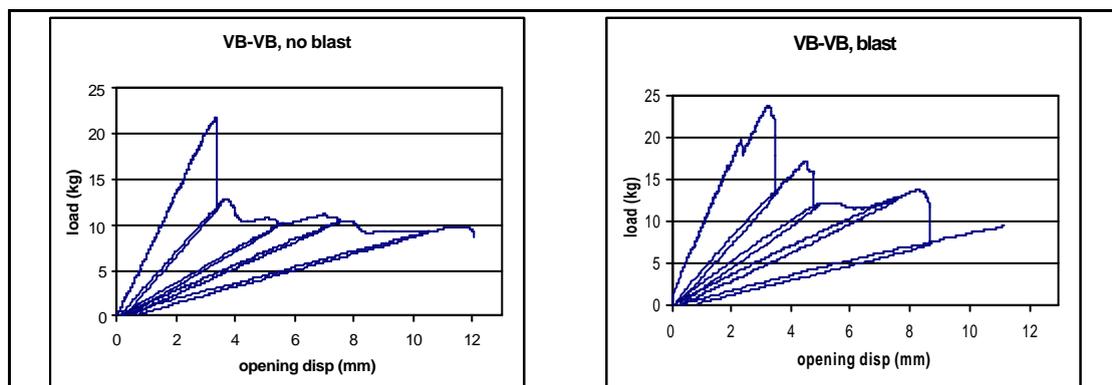
Basic beam theory describes the strain energy release rate of the specimen, assumed to be two linear elastic cantilever beams clamped at their ends (the crack tip). The assumption of rigid clamping is incorrect, resulting in inflated strain energy release values. Because the cantilever beams' constraint actually allows some rotation (at the crack tip) a plot of compliance versus crack length does not go through the origin, the position at which zero crack length correlates to zero compliance. The crack length must be offset to correct the error. This offset is determined experimentally for each specimen by plotting compliance^{1/3} versus crack length, performing a linear least squares fit, and finding the x-axis intercept. Excellent curve fits obtained in this study confirm the consistency of using optical tick-marks for determining crack tip location. Later x-ray photos (figure 6) confirmed that viewing the crack tip from one side was adequate, as the crack front was nearly straight.

DCB TEST RESULTS

The results of the DCB tests come in many forms, each of which is an indicator of the quality of the bond. The load-displacement curves generated by the test machine can be compared visually to determine maximum loads and displacements. Additionally, the path defined by the crack propagation can be smooth, indicating continuous crack growth, or jagged, indicating stick-slip behavior. The post-failure fracture surfaces, with adhesive (at the adhesive-adherend interface) or cohesive (within the adhesive) failure modes, show whether or not the adhesive bonded properly to the adherend. Finally, the calculated G_{Ic} values are indications of bond durability, by quantitatively evaluating how much energy must be put into the specimen to create fracture surfaces.

From a sampling of four typical load-displacement curves, one from each category (figure 3), several clear trends emerged:

1. Bonds made to surfaces cured against nylon release fabric failed at lower maximum loads than bonds to surfaces cured against vacuum bag.
2. Bonds made to surfaces cured against nylon release fabric exhibited complete failure at lower opening displacements than bonds to surfaces cured against vacuum bag.
3. Cracks propagated continuously in bonds made to surfaces cured against vacuum bag, but in a stick-skip behavior in bonds to surfaces cured against release fabric.
4. Grit blasting resulted in an increase in the initial failure load.



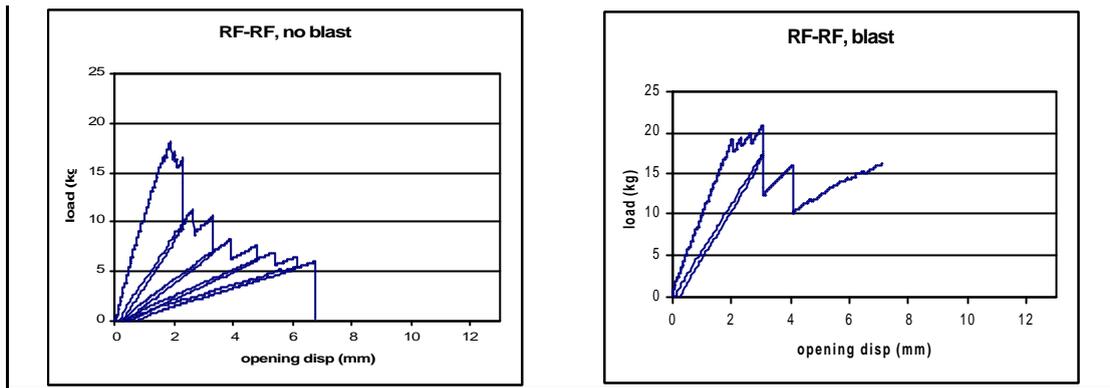


Fig. 3 Sample load-displacement curves for bonded composite DCB tests

Just as important as quantitative values such as loads and displacements is the more qualitative analysis of modes of failure. Well-bonded composite joints should fail cohesively or interlaminarly (within the adherends) when broken apart. Interfacial failure generally indicates that the bond was not performed properly, likely as a result of the silicone and siloxane release agents that were deposited onto the adherend surface from the release fabric during cure. From a sample of four typical failure surfaces, one from each of the four main groups of the set of samples (figure 4), a few more trends were clear:

1. Bonds made to surfaces cured against nylon release fabric failed interfacially.
2. Bonds made to surfaces cured against vacuum bag material failed cohesively and interlaminarly.
3. Grit blasting surfaces did not change the mode of failure.

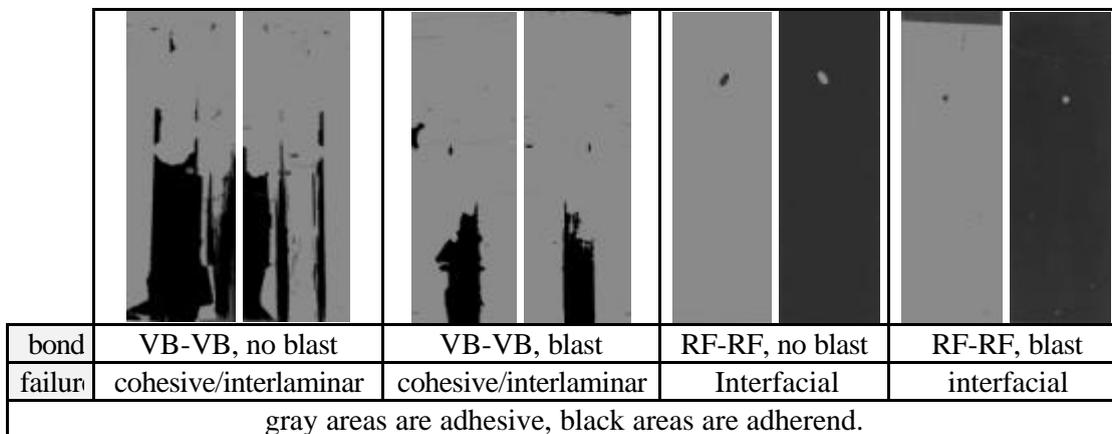


Fig. 4 Computer-enhanced pairs of fracture surfaces of bonded composite DCB specimens

The critical strain energy release rates calculated for the DCB test specimens (table 2 and figure 5) follow the observed trends in the load-displacement curves (figure 3) and the fracture surfaces (figure 4). Area method calculations are included for comparison to the MBT method. Hysol documents $G_{Ic} = 1.02 \text{ kJ/m}^2$ (5.83 in-lb/in^2) for EA9394 adhesive in a technical service laboratory report, tested on phosphoric acid anodized and etched aluminum DCB specimens with a 0.127 mm (0.005 in) bondline thickness controlled by glass beads.

Table 2 DCB critical strain energy release rate test results

Preparation of bonded surface	RF-RF no blast	RF-RF blast	VB-VB no blast	VB-VB blast
G_{Ic} : kJ/m ² (in-lb/in ²)	0.205 (1.174)	0.407 (2.328)	0.448 (2.560)	0.497 (2.843)

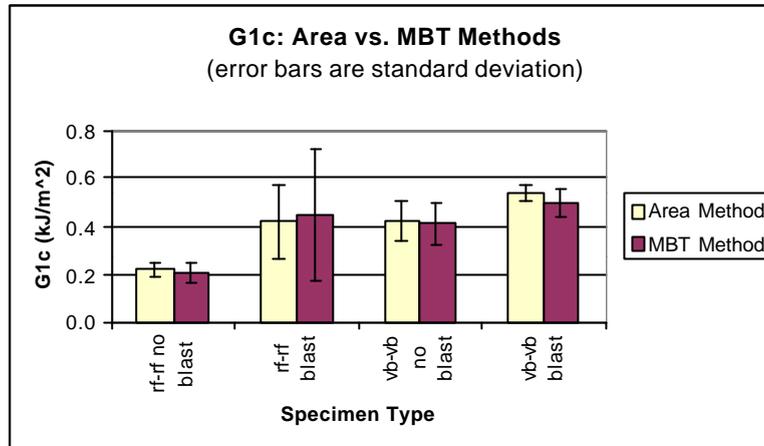


Fig. 5 DCB Critical strain energy release rate test results

The two key G_{Ic} trends seen in the tests were:

1. Bonds made to surfaces cured against vacuum bag material produced higher G_c values than bonds to surfaces cured against nylon release fabric.
2. Bonds made to grit-blasted surfaces produced higher G_c values than their non-blasted counterparts, regardless of other surface preparation. This correlates with partial removal of the silicone and siloxane peel ply release agents—enough to improve the bond strength, but not enough to change the mode of failure. Note that the RF-RF bonds benefited more noticeably from blasting than the VB-VB ones.

TRAVELLING WEDGE TEST PROCEDURE AND THEORY

An alternate version of the wedge test with a travelling wedge, rather than a static one, has undergone preliminary research as a viable counterpart to the DCB test [24,25]. The test has been run in two different configurations. Initial travelling wedge tests used an apparatus consisting of a 0.67 mm (0.0264 in) thick razor blade driven by a servomotor through a micrometer mechanism. The blade was forced into the horizontal specimen (supported against a vertical surface at the end opposite the wedge) at 3×10^{-3} mm/min (1.18×10^{-4} in/min), while crack length measurements were taken. Measurements taken at several locations over the length of the sample were averaged to provide fracture toughness data.

The apparatus could accommodate specimens up to only 71.1 mm (2.8 in) in length, and some specimens exhibited stick-slip behavior with crack lengths on the order of 32 mm (1.34 in), providing few data points per sample. The second configuration involved placing a longer 165 mm (6.5 in) specimen vertically in an Instron 8562 machine with a 101.6 mm (4 in) crosshead travel range. The wedge was thickened to 3.03 mm (0.119 in) and the specimen was notched and placed on a dowel pin to support the end opposite the wedge.

G_{Ic} for both travelling wedge test configurations can be determined from simple beam theory. This gives a good approximation for cracks that are long relative to the beam thickness, but it overestimates G_{Ic} for the shorter crack lengths typical of strong interfaces. A better

approximation uses a beam on elastic foundation analysis. The analysis reduced to equation 1 when the specimen is symmetric [24, 25].

$$G_{I_{travwedge}} = \frac{3}{16} \frac{d^2 E h^3}{a^4} \frac{1}{(1 + 0.64 \frac{h}{a})^4} \quad (1)$$

where d = opening displacement, E = longitudinal Young's modulus, h = beam height of the sample, and a = crack length, measured from the crack tip to the point of contact between the specimen and the wedge.

TRAVELLING WEDGE TEST RESULTS

To evaluate the travelling wedge test method, a series of DCB and travelling wedge samples were cut from the same panels. These tests were conducted with the initial travelling wedge apparatus described above. As seen in table 3, the travelling wedge test gave higher average and standard deviation for measured G_{Ic} values. This is a result of a handful of tests that produced unnaturally high values, possibly a result of the visual crack measurement method and fourth power of this crack length a in equation 1. Clearly, this method is more sensitive to accurate crack measurement than the DCB test. Also note that, due to a processing error, the DCB G_{Ic} values were somewhat lower than those seen in table 2, and all of the failure modes were interfacial, even for the VB-VB bonds. Using a revised travelling wedge test method better suited to the materials and specimens in this study (longer specimens, more accurate visual crack measurement), a second series of six VB-VB specimens were tested. The resulting G_{Ic} values matched the DCB tests' almost exactly (see table 3).

Table 3 Comparison of DCB and travelling wedge test methods

Test Method	Bond type	# of samples tested	G_{Ic} average kJ/m ² (in-lb/in ²)	G_{Ic} standard deviation kJ/m ² (in-lb/in ²)
DCB	RF-RF	6	0.142 (0.815)	0.0245 (0.140)
Travelling wedge	RF-RF	5	0.397 (2.27)	0.222 (1.27)
DCB	VB-VB	2	0.350 (2.00)	0.0552 (0.316)
Travelling wedge	VB-VB	9	0.645 (3.69)	0.496 (2.84)
Travelling wedge (second method)	VB-VB	6	0.331 (1.894)	0.161 (0.922)

ADDITIONAL TEST METHOD NOTES

One concern of the visual measurement of crack position against a hand-drawn set of tick marks was that only one side of the specimen can be monitored. Consistency in test-derived G_{Ic} values, which rely upon consistent crack measurement within a test and from test to test, have proven that the current method of crack tip measurement is adequate. But, to better understand the crack front for the specimens used, X-ray photography was employed. Specimens were wedged open to advance their cracks and a zinc iodide solution was injected into the crack tip and X-ray photos were taken (figure 6). The crack fronts in figures 6(a) and 6(b) are relatively linear but slightly diagonal. The crack front in 6(a) on one side is 03.45 mm (0.136 in) ahead of the other side across its 25 mm (0.983 in) width, while 6(b)'s crack varies

by 1.35 mm (0.053) across the sample's 10.8 mm (0.425 in) width. The crack front in 6(c) shows the effects of natural environmental exposure over one week—the crack advanced noticeably on the edges, where water (humidity in the air) was attacking the interface. Note that this was a sample with an interfacial bond failure, more susceptible to environmental exposure than a bond with a cohesive failure.

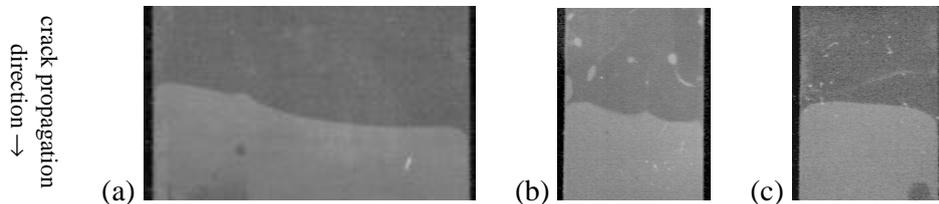


Fig. 6 X-ray photographs of crack fronts: crack propagation direction from top to bottom:
 (a) 25 mm (0.983 in) wide specimen, freshly cracked
 (b) 10.8 mm (0.425 in) wide specimen, freshly cracked
 (c) 10.8 mm (0.425 in) wide specimen, one week of exposure to natural environment

The DCB and travelling wedge tests are intended to provide a foundation upon which to extend the investigation of future static wedge testing on similar geometrical configurations. The static wedge test samples themselves will be the same as those used in the tests discussed herein to make data correlation between the tests straightforward, as they are essentially the same test (figure 2). In the static wedge test, the specimens are to be subjected to elevated humidity, which is a reliable short-term method to predict bond integrity of a joint over long periods of time in service, as detailed in the literature [3-11].

Additionally, there are plans to use profilometry and atomic force microscopy to attempt to study the effects of abrasion on the surface of the adherends. These tools should provide quantitative data to determine optimal grit blasting methods and parameters.

Planned future use of x-ray photoelectron spectroscopy will not only provide the chemical makeup of peel plies and contaminants, but it will indicate the transmissibility of these chemicals to the adherend surface before bonding.

CONCLUSIONS

Because DCB and travelling wedge tests accurately predict short-term strength and static wedge tests predict long-term adhesive bond durability in service, surface preparation methods that affect bond strength are evaluated with these methods. DCB testing shows that grit blasting surfaces prior to bonding led to higher G_{Ic} values, though the mode of failure (interfacial or cohesive) is unchanged from a non-blasted sample. Adhesive bonding to composite surfaces that were cured against a nylon release fabric rather than a PTFE vacuum bag film showed the following trends:

1. Failure at lower loads and corresponding lower opening displacements.
2. Stick-slip crack propagation.
3. Lower G_{Ic} values.
4. Interfacial, not cohesive failure.

Static wedge tests in an elevated humidity environment, with the same bonded composite specimen, will utilize DCB or travelling wedge test results to predict joint failure in the

projected research.

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