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Effects of Surface Preparation on Long-Term Durability of Composite Adhesive Bonds

April 2001

Final Report

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16. Abstract

The long-term effects of surface preparation techniques for composite bonded joints are addressed. Several potential factors are evaluated, concentrating on the effects of peel plies and grit blasting on the fracture toughness and failure mode of adhesively bonded composites. An evaluation of the floating roller peel test configuration is described, where the intent was to extract quantitative data from this commonly used quality control test method. Subsequently, an alternate form of the double cantilever beam (DCB) test was developed and used for a sequence of test evaluations. The research will aid the interpretation of a form of the wedge test where the usual aluminum adherends are replaced by composite adherends.

DCB tests have shown that nylon peel ply* surfaces tend to precipitate interfacial failures and intermittent crack propagation, with reduced loads and crack opening displacements, hence, significantly lower critical strain energy release rates (G_{Ic}) than equivalent polytetraflouroethylene (PTFE) vacuum bag surfaces. Additionally, grit-blasted bonded joints tend to have higher failure load and G_{Ic} values than nonblasted ones, though the mode of failure (interfacial or cohesive) is unchanged. Several improvements to specimen preparation and testing, including a custom bonding jig, bondline thickness control methods, and an alternate version of the wedge crack test are also described.

*For this study, all discussions of a peel ply used in curing laminates refer to a nylon fabric coated with siloxane and silicone release agents. In industry, this material is often referred to as a release fabric, intended to release from the laminate without removing any material from the outer ply. A peel ply is generally a material that exposes a fresh, fractured surface on the laminate when it is peeled off.

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EXECUTIVE SUMMARY

The long-term durability of adhesively bonded composite joints is critical to modern aircraft structures, since the use of bonding is increasing as an alternative option to mechanical fastening. The effects of the surface preparation of the adherends are major durability factors that need to be characterized. In this study, several potential factors are evaluated, with concentrations on the effects of chemical contamination from peel ply release agents (silicone and siloxane in this work) and the chemical and mechanical effects of grit blasting on the fracture toughness and failure mode of adhesively bonded composites joints.

There are several test methods used to evaluate adhesive bonds, but the large majority tend to concentrate on bonded metals. An evaluation of the floating roller peel test configuration was performed, using composite in place of metallic adherends, with the objective of extracting quantitative data from this commonly used quality control test method. It was found that, for the range of available sample thicknesses, the brittle behavior and low failure strains of composites, relative to most metals, hampered this test.

Subsequently, an alternate form of the Double Cantilever Beam (DCB) test was developed. Based on three different American Society for Testing and Materials (ASTM) standards, this version was oriented toward adhesively bonded composites, a configuration not found in the test standards. This test method was used for a sequence of test evaluations. The results of this research are geared towards aiding the interpretation of a form of the wedge test where the usual aluminum adherends are substituted by composite adherends. This wedge test will utilize the same adherends as for the DCB test, thereby simplifying evaluation and comparison. An alternate version of the test, where the wedge is driven slowly by a motor to a series of fixed positions, instead of inserted and left in place, is also under evaluation.

Two different data reduction methods were used for analyses, one based on strain energy and the other on beam theory. Both techniques produced nearly identical results, and an investigation into cantilever beam compliance results confirmed the use of optical readings of hand-drawn tick marks as a consistent crack measurement method.

DCB test data showed that nylon peel ply* surfaces tended to precipitate interfacial failures and intermittent crack propagation, with reduced maximum loads and final crack opening displacements (before complete specimen failure), hence, significantly lower critical strain energy release rates (G_{Ic}) than equivalent polytetraflouroethylene (PTFE) vacuum bag surfaces. Additionally, grit-blasted bonded joints tended to have higher failure load and G_{Ic} values than nonblasted ones, though the mode of failure (interfacial or cohesive) was unchanged. Several improvements to specimen preparation and testing, including a custom bonding jig, bondline thickness control methods, and the alternate version of the wedge crack test are also described.

* For this study, all discussions of a peel ply used in curing laminates refer to a nylon fabric coated with siloxane and silicone release agents. In industry, this material is often referred to as a release fabric, intended to release from the laminate without removing any material from the outer ply. A peel ply is generally a material that exposes a fresh, fractured surface on the laminate when it is peeled off.

1. INTRODUCTION.

1.1 PURPOSE.

Adherend surface preparation plays a critical role in the development and evaluation of bonded joints. However, general aviation (GA) tends to rely more extensively on bonded joints, in part due to the lower load intensities typically found in smaller aircraft. Inadequate surface roughening, environmental effects, peel ply 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.

1.2 SURFACE PREPARATION VARIABLES.

Initially, many possible factors that could affect an adhesive bond's durability were amassed and evaluated (table 1). In this report, the focus is primarily on the effects of peel plies and grit blasting, both of which affect bond integrity greatly and are relevant to the aviation industry.

TABLE 1. POTENTIAL BONDING FACTORS

Factor	Variables
Adherend Lay-Up	$0°_{[n]}$, quasi-isotropic, other lay-up; orientation of ply on bonding surface
Adherend Material	Fiber, matrix, metal, 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 "Shop Air" Blowing	Pressure, exposure time
Grit Blast	Pressure, grit size, number of passes, speed of passes
Hand Sanding	Grit size, number of passes, pressure applied
Humidity Exposure	Humidity %, exposure time, prebond, postbond, under load
Peel Ply or Release Film	Nylon, polyester, none
Solvent Wiping	Acetone, isopropyl alcohol, number of wipes, applicator type
Temperature Exposure	Temperature, exposure time, prebond, postbond, under load
Water Bath	Temperature, exposure time, prebond, postbond, under load

1.3 TEST METHODS FOR ADHESIVELY BONDED JOINTS.

Specimens were evaluated with an adaptation of the American Society for Testing and Materials (ASTM) D3167 Standard Test Method for Floating Roller Peel Resistance of Adhesives. A combination of ASTM D3433 Standard Test Method for Fracture Strength in Cleavage of Adhesives in Bonded Joints and ASTM D3762 Standard Test Method for Adhesive-Bonded Surface Durability of Aluminum (Wedge Test), was also employed. Analytical and numerical models of test methods are performed to analyze test data and specimen configuration. Materials and processes typical of those used for aircraft were 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 Federal Aviation Administration (FAA) with interpreting data related to certification and evaluation procedures.

2. TEST METHODS.

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 reported that the lap shear test only verifies the short-term bond strength, not its long-term durability, and that shear and peel strength tests are not sufficient to assure durability, but the wedge test is ideal for durability measurement [12]. Furthermore, Hart-Smith noted that the lap shear test tells nothing about durability and that process control, monitored by wedge tests, can help to ensure long-term durability [10]. Finally, 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 disbond behavior from 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 never truly possible 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 double cantilever beam (DCB) tests are consequently an appropriate test for bonded joints.

2.1 FLOATING ROLLER TEST.

The initial approach utilized floating roller tests on woven fiberglass and carbon fiber/epoxy samples. The ASTM D3167 test is designed for a thick adherend bonded to a thin metal adherend that bends around the roller during peeling. Because composite-to-composite bonds are more typical of the type of aircraft being studied, single woven plies were used for the thin adherend instead of metal. However, these thin composite adherends did not conform to the fixture's roller as they were being pulled from the thick adherends. They bent at too tight a radius of curvature and fractured before the bond could be broken (figure 1). This test method was abandoned in favor of other bond strength tests that do not require such extreme strains on the adherends to fracture the bond.

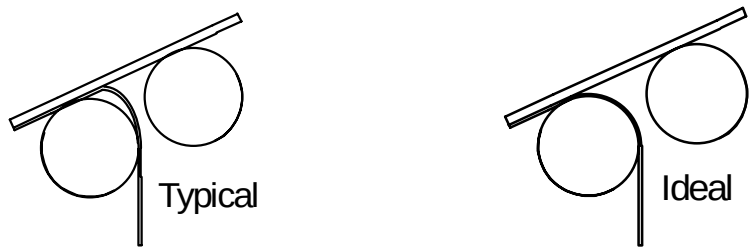


FIGURE 1. TYPICAL VERSUS IDEAL FLOATING ROLLER TEST

2.2 DOUBLE CANTILEVER BEAM AND WEDGE TESTS.

Literature research and a review of standard test methods revealed that the DCB and wedge tests (figure 2) are well-suited to evaluating the short- and long-term durability adhesive bonds [6, 10, 12 to 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 unloading). The wedge test can be performed with the same specimen, but a tapered wedge is driven into the crack opening to stimulate crack growth. Then, the sample is observed (often in an environmental exposure chamber) with the wedge remaining in its initial position, and the crack tip propagation is recorded.

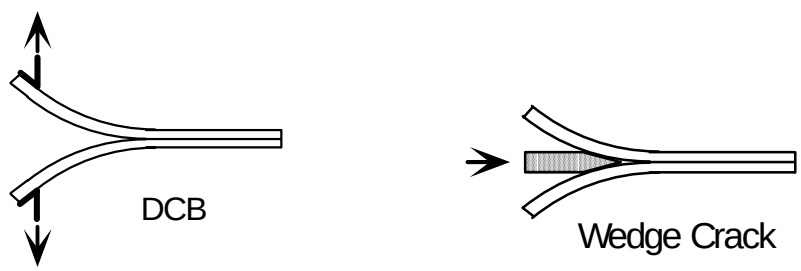


FIGURE 2. DOUBLE CANTILEVER BEAM AND WEDGE CRACK TESTS

2.2.1 Specimen Geometry.

Unfortunately, there is a lack of standard test procedures for measuring the bond strength of adhesively bonded composite materials. ASTM test methods researched cover either adhesively bonded metals or interlaminar failures in composites. The new specimen used for the DCB and wedge crack tests is based on those of ASTM D3433, ASTM D3762, and ASTM D5528-94a Standard Test Method for Mode I Interlaminar Fracture Toughness of Unidirectional Fiber-Reinforced Polymer Matrix Composites (figure 3).

After the sequence of 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 proposed new specimens are 12″ long and 0.5″ wide, half the width of the ones used in this

study. Although very small widths can affect fracture toughness values of specimens, because they exhibit a plane stress behavior instead of plane strain, work by Crosley and Ripling [6] and Kinloch and Shaw [19] demonstrated that specimen geometry of these new dimensions should not affect test results.

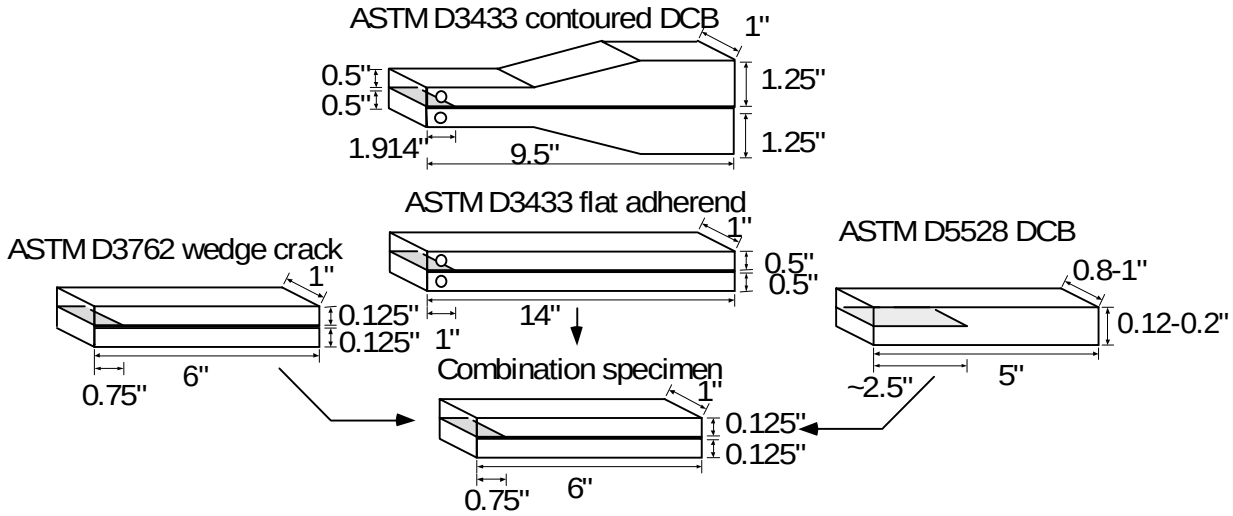


FIGURE 3. ASTM TEST SPECIMENS COMBINED INTO ONE SAMPLE GEOMETRY

2.2.2 Specimen Preparation.

DCB tests were performed on IM7/8552 22-ply unidirectional adherends bonded with Hysol EA9394 two-part epoxy adhesive mixed with 0.005 inch-diameter glass microbeads (2.5% by weight). All adherends were cured with Chemfab VB-3 polytetraflouroethylene (PTFE) vacuum bag (VB) film on the bottom surface (tool side) and with a nylon peel ply (PP) with silicone and siloxane release agents on the top side (figure 4). This lay-up process creates panels with different surface properties on each side. Samples were bonded in one of two orientations: PP to PP 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 lay-up and bagging procedure, with different surfaces on both faces, is not typical of a component used in production, it was well-suited as a research aid. 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 variances in specimen production.

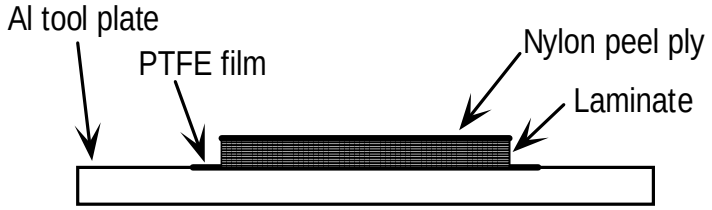


FIGURE 4. SCHEMATIC OF LAY-UP OF COMPOSITE ADHERENDS

The composite samples that were grit-blasted (with Mil-A-2222B grit) were done so at 60 psi (regulator line pressure). The hinges used to hold the samples in the test machine grips were made from 0.04″ thick continuous hinge, cut to 1″ lengths and grit-blasted at 100 psi. The surfaces to which the hinges were bonded on all samples were also blasted at 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 deionized water, oven-dried, wiped with isopropyl alcohol, then air-dried before bonding. All crack initiators were created with 3″ of flashbreaker tape on the ends of the specimens.

Before testing, the sides of samples were spray painted white and tick marks were penciled on manually at 1/16″ intervals for visual crack tip observation during the tests.

2.2.3 Testing Method and Data Reduction.

Specimens were loaded into an Instron 8562 test machine by clamping the hinges in test grips. The test machine ran under displacement control at a crosshead speed of 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 1/2″ of crack growth.

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 monoscope. There are several methods to calculate critical strain energy release rates from this load-displacement and crack length data. Blackman, et al. compared four such methods: the area, compliance, simple beam theory, and displacement methods [20].

The calculation methods discussed below do not take into account the effect of the adhesive layer and its interfaces. In Fernlund and Spelt's experiments on adhesively bonded aluminum DCB test specimens, it was found that the adhesive layer need not be accounted for in beam theory-based G_{Ic} calculations, provided that certain geometrical conditions are met [21]. Using the more complex equation that included the adhesive layer, a consistent G_{Ic} value was obtained for any crack lengths in a given specimen. They found that, at small crack lengths, the calculated G_{Ic} values approached zero if the adhesive layer effects were not incorporated. However, beyond a certain crack length threshold value, the calculated fracture toughness values matched exactly those from the more complex equation. From a plot of G_{Ic} versus crack length, the simpler beam theory equation provided valid results for

$$\frac{a}{h-t} > 8 \tag{1}$$

where a is the crack length, h is the height of one DCB arm, and t is half the thickness of the adhesive layer. For the specimens tested at University of California Santa Barbara (UCSB), $h \approx 0.125''$, $t \approx 0.005''$, and the starter crack was 2″ beyond the load point, putting the specimens well beyond the threshold value, so the G_{Ic} calculations need not include the adhesive layer.

2.2.3.1 Area Method.

The area method is based upon a change in the DCB sample's compliance (equation 2) resulting from a change in crack length (equation 3). Therefore, the strain energy lost due to crack extension for a linear elastic body is the area between the loading and unloading curves on a load-displacement plot. Assuming that the crack propagation portion of a load-displacement curve can be approximated with a straight line, equation 3 gives the fracture toughness of the specimen [22]. If equation 4 is used for each crack extension portion of a test, the specimen's G_{Ic} value is the average of individual calculations.

$$C = \frac{d}{P} \tag{2}$$

$$G_I = \frac{1}{b}\frac{dU}{da} \tag{3}$$

$$G_{Ic} = \frac{1}{2b\Delta a} P_1 d_2 \cdot P_2 d_1 \tag{4}$$

C is compliance, d is the deflection at load point, P is the applied load, U is the total strain energy stored in the test specimen, b is the specimen width, Δa is the crack length change from position 1 to position 2, P_1 and P_2 are the applied loads at positions 1 and 2, and d_1 and d_2 are the deflections at positions 1 and 2.

2.2.3.2 Modified Beam Theory (MBT) Method.

The MBT method is based on the "displacement" method discussed by Blackman, et al. [20] and Whitney, et al. [22], and detailed by Johnson, et al. [23] and in the ASTM D5528 test method. From basic beam theory, equation 5 describes the strain energy release rate of the specimen. This reduces to equation 6 when one assumes that the DCB sample consists of two linear elastic cantilever beams clamped at their ends (the crack tip). Note that these calculations require only one data point each, while the area method requires pairs, allowing for more data points obtained per specimen, and removing some of the subjectivity in determining which pairs of data points best represent the curve.

$$G_I = \frac{P^2}{2b}\frac{dC}{da} \tag{5}$$

$$G_I = \frac{3Pd}{2ba} \tag{6}$$

The assumption of rigid clamping is incorrect and it results 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, a, must be offset by a value Δ to correct the error (equation 7).

$$G_I = \frac{3Pd}{2b\left(a + |\Delta|\right)} \tag{7}$$

The offset Δ is determined experimentally for each specimen by plotting $C^{1/3}$ versus a, performing a linear least squares plot, and finding the x-axis intercept (figure 5). The 1/3 power term in the plot is from the compliance-crack length relationship, which states that $C^{1/3}$ is proportional to a [20]. The excellent curve fits obtained in this study confirm the consistency of using an optical tick-mark measurement method for determining the crack tip location.

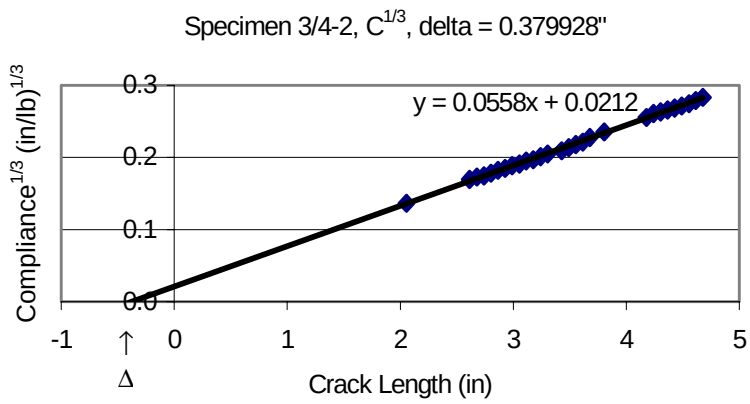


FIGURE 5. SAMPLE COMPLIANCE$^{1/3}$ VERSUS CRACK LENGTH PLOT TO DETERMINE CRACK OFFSET FOR MBT METHOD

2.2.4 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 postfailure fracture surfaces, with adhesive or cohesive 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 showing how much energy must be put into the specimen to create fracture surfaces.

2.2.4.1 Load and Displacement.

From a sampling of four typical load-displacement curves, one from each category (figure 6), several clear trends emerged.

- Bonds made to nylon peel ply surfaces held lower maximum loads than bonds to vacuum bag surfaces.

- Bonds made to nylon peel ply surfaces exhibited complete failure at lower opening displacements than bonds to vacuum bag surfaces.

- Cracks propagated continuously in bonds made to vacuum bag surfaces, but in a stick-skip behavior in bonds to nylon peel ply surfaces.

- Grit blasting resulted in an increase in the initial failure load.

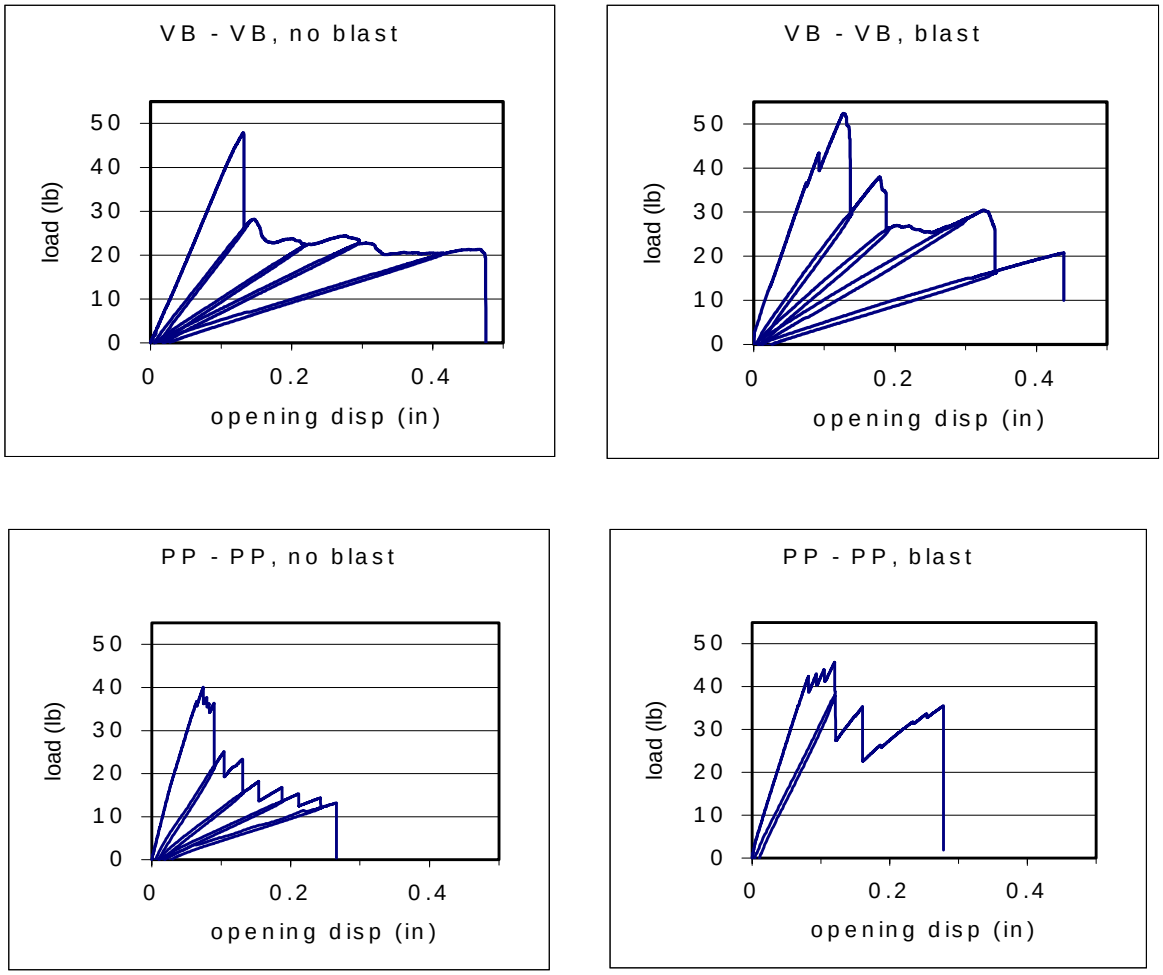


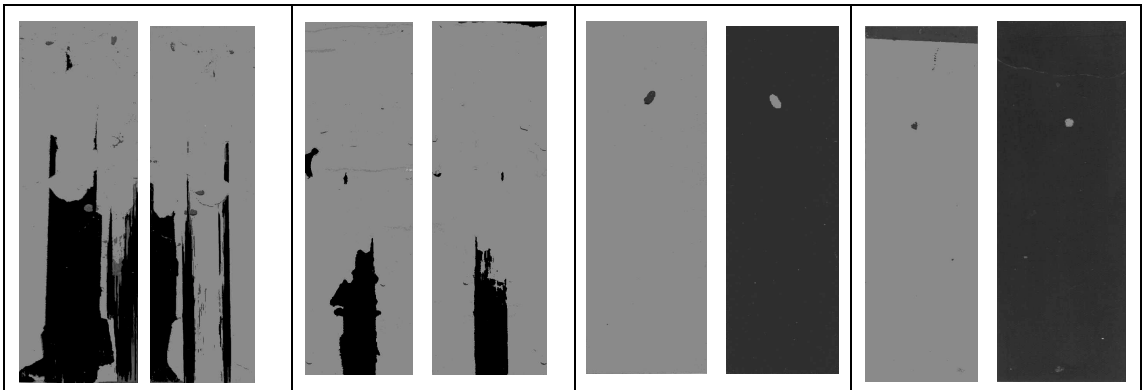
FIGURE 6. SAMPLE LOAD-DISPLACEMENT CURVES FOR BONDED COMPOSITE DCB TESTS

2.2.4.2 Failure Mode.

Just as important as quantitative values like loads and displacements is the more qualitative analysis of modes of failure. Well-bonded joints should fail within the adhesive (cohesive failure) or within the adherends (interlaminar failure) when broken apart. Failure at the adherend-adhesive interface (interfacial failure) generally indicates that the bond was not performed properly, a result of the silicone and siloxane release agents that were deposited onto the adherend surface from the peel ply during cure. From a sampling of scans of four typical

failure surfaces, one from each of the four main groups of the second set of samples (figure 7), a few more trends were clear.

- Bonds made to nylon peel ply surfaces failed interfacially.
- Bonds made to vacuum bag surfaces failed cohesively and interlaminarly. Test data from interlaminar failure portions of tests were not included in the analysis.
- Grit blasting surfaces did not change the mode of failure.



Bond	VB – VB, no blast	VB – VB, blast	PP – PP, no blast	PP – PP, blast
Failure	Cohesive/Interlaminar	Cohesive/Interlaminar	Interfacial	Interfacial
Gray areas are adhesive. Black areas are adherend.				

FIGURE 7. COMPUTER-ENHANCED PAIRS OF FRACTURE SURFACES OF BONDED COMPOSITE DCB SPECIMENS

2.2.4.3 Critical Strain Energy Release Rate.

The critical strain energy release rates calculated for the DCB test specimens (table 2 and figure 8) follow the observed trends in the load-displacement curves (figure 6) and the fracture surfaces (figure 7). Hysol documents a mode I critical strain energy release rate G_{Ic} = 5.83 in-lb/in^2 for their EA9394 adhesive (in their technical service laboratory report), tested on phosphoric acid anodized and etched aluminum DCB specimens with a 0.005″ bondline controlled by glass beads. Calculations were performed with both the area (section 2.2.3.1) and MBT methods (section 2.2.3.2). The average G_{Ic} values calculated with the area versus MBT method showed a correlation within 9%. The standard deviations obtained with the MBT method were somewhat greater than those calculated by the area method.

TABLE 2. DOUBLE CANTILEVER BEAM CRITICAL STRAIN ENERGY RELEASE RATE TEST RESULTS

	PP – PP, no blast	PP – PP, blast	VB - VB, no blast	VB - VB, blast
AREA G_{Ic}: in-lb/in^2 [kJ/m^2]	1.244 [0.217]	2.412 [0.422]	2.367 [0.414]	3.086 [0.540]
MBT G_{Ic}: in-lb/in^2 [kJ/m^2]	1.174 [0.205]	2.328 [0.407]	2.560 [0.448]	2.843 [0.497]

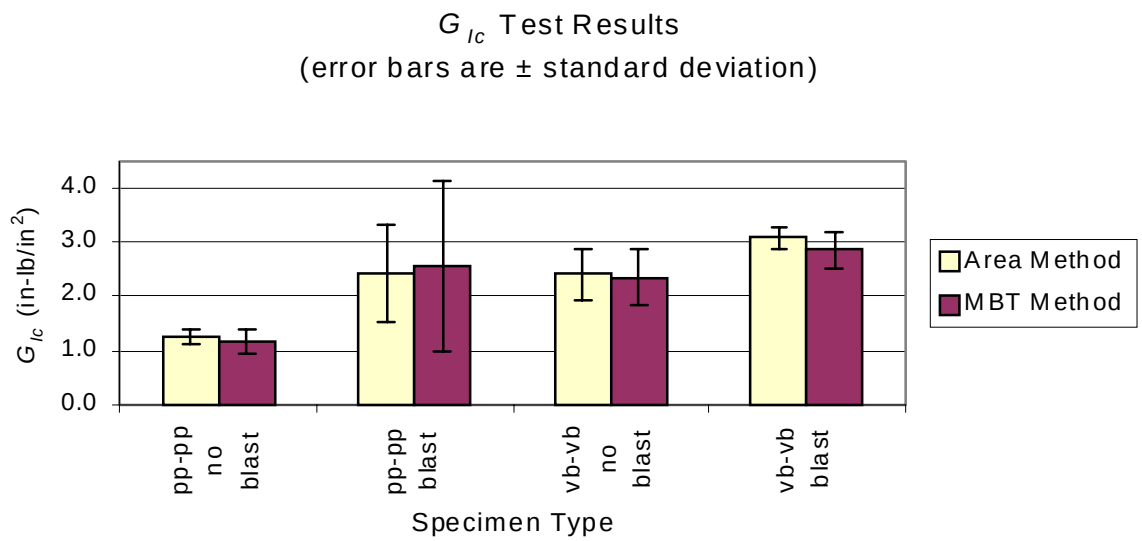


FIGURE 8. DOUBLE CANTILEVER BEAM CRITICAL STRAIN ENERGY RELEASE RATE TEST RESULTS

The two key G_{Ic} trends seen in UCSB's tests were:

- Bonds made to vacuum bag surfaces produced higher G_{Ic} values than bonds to nylon peel ply surfaces.

- Bonds made to grit-blasted surfaces produced higher G_{Ic} values than their nonblasted counterparts, regardless of previous surface preparation. This implies 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.

2.3 TRAVELLING WEDGE TEST.

An alternate version of the wedge test—with a travelling wedge, rather than a static one—is being considered for use in future research [24]. The apparatus consists of a razor blade driven by a servomotor through a micrometer mechanism. The blade is forced into the specimen at a very slow rate (1.18×10^{-4} in/min), then stopped, then crack length measurements are taken. The

process is repeated several times over the length of the sample, and these crack length values are averaged together to provide fracture toughness data.

3. UPGRADES AND REFINEMENTS.

Several upgrades to the DCB test method are under consideration for future tests. The specimen preparation itself is undergoing refinement to ensure consistency in bonds from sample to sample, in order to validate comparisons. Additionally, the use of glass microbeads for bondline thickness control is being evaluated.

The DCB tests are intended to provide a foundation upon which to extend the investigation of future wedge testing on similar geometrical configurations. The wedge test samples themselves will be the same as those used in the DCB test (minus the hinge hardware) to make data correlation between the two tests straightforward, as they are essentially displacement or load control versus fixed load of the same test specimen (figure 2). In the wedge test, the specimens are to be subjected to elevated temperature and 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.

Planned future use of x-ray photoelastic spectroscopy will not only allow the confirmation of the chemical makeup of peel plies and contaminants, but it will indicate how these chemicals are transmitted to the adherend surface before bonding.

3.1 DOUBLE CANTILEVER BEAM ISSUES.

Visual tracking with a monoscope is one method of measuring crack tip location. Others include foil resistance gauges and video camera setups. There were initial concerns over the accuracy and consistency of the employed method of scope observation of hand-drawn tick marks, but test results have proven the latter to be adequate (figure 5). The majority of the literature reviewed describes identical measurement methods.

One concern of the visual measurement of crack growth on a hand-drawn set of tick marks is that only one side of the specimen can be monitored. It is assumed that the crack front is perpendicular to the direction of crack growth and is relatively linear, but this has not been confirmed for these specimens. A second observer on the back side of the DCB sample can verify that the crack front is symmetric, but this cannot confirm linearity. A series of partially-cracked DCB samples may be C-scanned to determine the exact crack front shape in the specimen. Consistency in test-derived G_{Ic} values, which rely upon consistent crack growth measurement within a specimen and from test to test, suggests that the current method of crack tip measurement is adequate, though there are more elegant methods.

3.2 ALIGNMENT OF BONDED SPECIMENS AND FIXTURES.

After evaluating the manual alignment method for the first two sets of bonded DCB specimens and the hinges applied to them, it was decided to create a bonding jig (figure 9). The alignment of the bonded unidirectional laminates and the fixture hinges can affect the strain energy release rates considerably. If bonded panels are positioned by hand and then held under weight, there is an opportunity for misalignment.

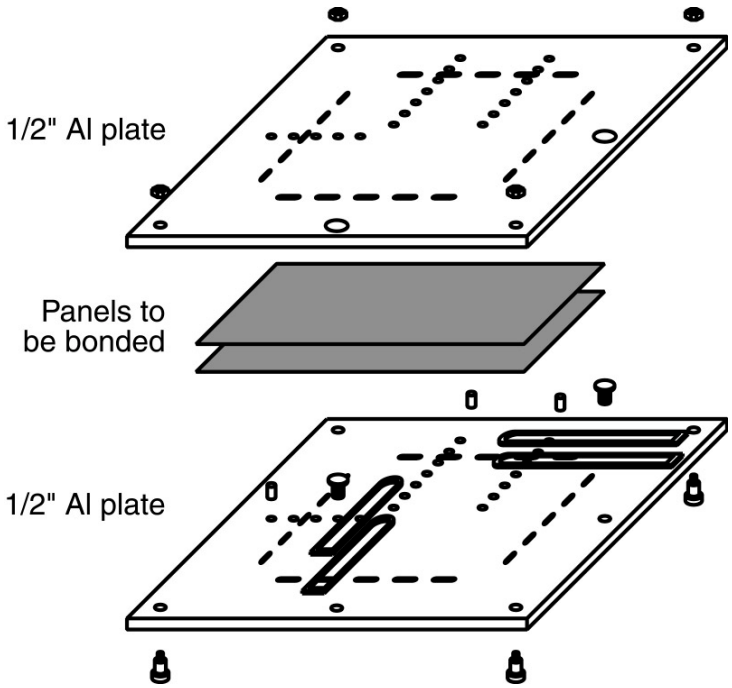


FIGURE 9. EXPLODED VIEW OF BONDING JIG

The bonding jig that was designed and constructed is essentially a large press made of two 18″ by 18″ by 1/2″ aluminum 6061 plates. Features include

- One-eight-inch-thick silicone sheets attached to the aluminum plates to distribute the press load evenly over local panel contours and prevent squeezed-out adhesive from bonding the jig together.

- A grid of peg holes to allow panels to be aligned to each other while centered in the jig.

- Two pairs of staggered clamping sliders to keep the panels against the alignment pegs, even with samples that are uneven or unequal in size.

- Twenty pairs of slots to accommodate the hinge pins for aligning hinges to DCB samples and for bonding both hinges to a specimen simultaneously.

3.3 BONDLINE THICKNESS CONTROL.

There are several methods of controlling the bondline thickness of adhesive joints. Users of paste adhesives, typical of GA, are left to determine the bond thickness of their joints by the insertion of spacers into the bondline. Users of film adhesives, typical of commercial transport, generally use products that come with built-in spacer schemes*.

3.3.1 Paste Adhesive.

The bond thickness for the first two sets of DCB samples was controlled by mixing in 0.005″ diameter glass microbeads (2.5% of the weight of the adhesive) into the epoxy before bonding. Because the two-part epoxy was mixed by hand and because the microbeads visually disappear once mixed in, it was impossible to determine if the beads were distributed evenly. Also, despite the diameter of the glass microbeads and the applied weight during curing, bondlines were never as small as 0.005″, and some practice bonds made in the bonding jig were as large as 0.020″.

While examining fracture surfaces with a scanning electron microscope (SEM), it was found that there were fine separation layers between the glass microbeads and the adhesive (figure 10). This indicates that the adhesive did not bond to the microbeads, creating possible crack nucleation points, potentially weakening the overall bond and lowering the measured G_{Ic} values. However, alternate theories propose that this lack of bond between the glass beads and the adhesive may actually lead to crack blunting, thereby increasing G_{Ic}.

Pretreatment of the glass beads with a silane adhesion promoter will ensure proper bonding between the beads and the adhesive, but it will not address the distribution issue. Alternate spacing methods are under consideration, including distributed wires, tabs, and layers of tape. These alternate methods allow more control over spacer distribution, but care must be taken to compensate for these large embedded features when cutting bonded panels into individual specimens. Additionally, the SEM photos revealed what appear to be very small cavities created by air trapped in the mixed adhesive, another potential crack initiation or blunting site like the are around the glass beads.

3.3.2 Film Adhesive.

Because of the difficulties and additional variables associated with paste adhesive, and because of a desire to expand this research to include the commercial transport industries, the focus of future studies will concentrate on film adhesive. The integral carrier cloth (or scrim cloth) embedded in film adhesives, coupled with a pressurized autoclave cure, will allow for much more accurate and thin bondlines.

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* It should be noted that for test samples in this study, the thickness can be more easily controlled than in larger structures.

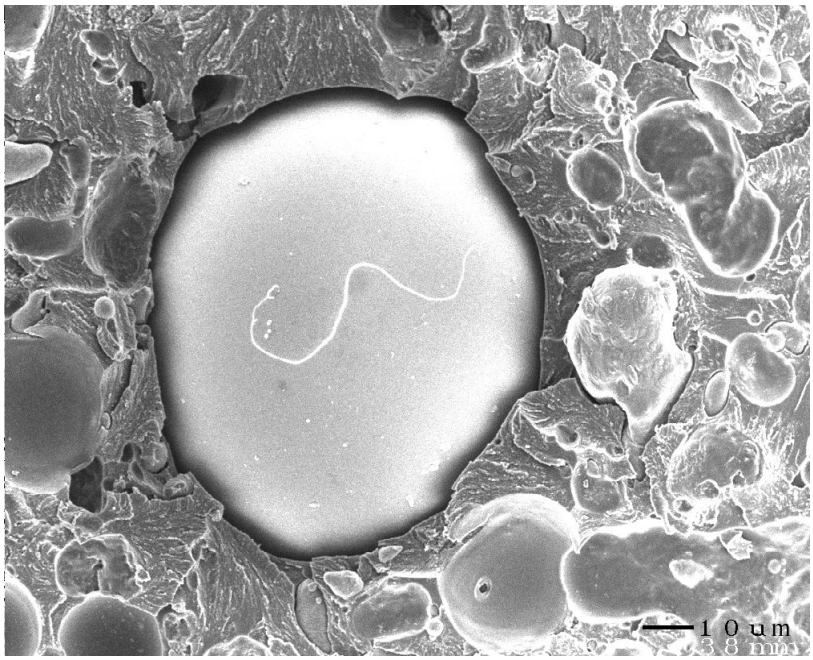

FIGURE 10. A 0.005 inch-DIAMETER GLASS BEAD IN ADHESIVE ON DCB FRACTURE SURFACE, 429× MAGNIFICATION

4. SUMMARY.

Because double cantilever beam (DCB) and wedge test results accurately predict short-term strength and long-term adhesive bond durability in service, respectively, surface preparation methods that affect bond strength were evaluated with these methods. The results of the DCB testing suggest that grit blasting surfaces prior to bonding led to higher G_{Ic} values, though the mode of failure (interfacial or cohesive) was unchanged from a nonblasted sample. Two different data reduction methods were demonstrated and found to be consistent with each other. Adhesive bonding to composite surfaces that were cured against a nylon peel ply rather than a polytetraflouroethylene (PTFE) vacuum bag film showed the following trends:

- Failure at lower loads and corresponding opening displacements.
- Intermittent crack propagation.
- Lower G_{Ic} values.
- Interfacial, not cohesive failure.

Due to geometric constraints and the low in-plane fracture strain of composites materials, floating roller tests were found to be unsuitable for testing bonded composites for the thicknesses used herein. A bonding jig was designed and constructed to ensure accurate alignment and bonding of specimens and DCB hinges.

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