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Surface Preparation Effects on Mode I Testing of Adhesively Bonded Composite Joints*

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ABSTRACT: The determination and significance of surface preparation on the long-term durability of bonded composite joints is addressed. Several potential factors are evaluated, including the effects of release fabric (sometimes incorrectly referred to as a peel ply) usage and grit blasting on the fracture toughness and failure modes of bonded assemblies. The double cantilever beam (DCB) test is adapted with the intent of developing a quantitative interpretation of a composite version of the wedge test. This well-established wedge test is used for quality and durability assessments of metal bonded systems.

In conjunction with conventional lap shear and joint subcomponent testing, this research is directed at enhancing the reliability of composite bonded assemblies that are subjected to long-term loading and varying environmental exposure.

DCB tests show that curing nylon release fabrics against adherend surfaces leads to interfacial failures and intermittent crack propagation, with reduced loads and crack opening displacements, giving critical strain energy release rates (G_{Ic}) lower than bonds produced with equivalent PTFE vacuum bag surfaces. Grit-blasted adherends have higher failure load and G_{Ic} values than nonblasted ones, though the mode of failure does not change.

KEYWORDS: structural adhesive, bonded joint durability, surface preparation, mode I failure tests, joint failure, peel ply, release fabric, grit blast

Introduction

Purpose

A common practice currently adopted in the general aviation (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 an appropriately 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 durability characteristics of bonded joint regions. 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 aerodynamic lines, part consolidation, no holes drilled in adherends (with the resulting stress concentrations), and, potentially, reduced labor.

Adherend surface preparation plays a critical role in the development and evaluation of bonded joints. However, GA tends to rely more extensively on bonded joints than commercial aviation does, in part due to the lower load intensities typically found in smaller aircraft. Inadequate surface roughening, environmental effects, release fabric chemical contamination, and other factors (both mechanical and chemical) can prevent adhesives from bonding properly to composites, resulting in premature interfacial failures [*1*]. 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 [*2–10,19*]. 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.

Surface Preparation Variables

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

Test Methods for Adhesively Bonded Joints

Attempts to adapt the ASTM Standard Test Method for Floating Roller Peel Resistance of Adhesives (D 3167) to bonded composites were undertaken. A combination of the following was also employed: ASTM Standard Test Method for Fracture Strength in Cleavage of Adhesives in Bonded Joints (D 3433), ASTM Standard Test Method for Mode I Interlaminar Fracture Toughness of Unidirectional Fiber-Reinforced Polymer Matrix Composites (D 5528), and ASTM Standard Test Method for Adhesive-Bonded

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* In memory of Dr. Don Oplinger

TABLE 1—*Potential bonding factors.*

Factor	Variables
Adherend Lay-up	$O^{\circ}_{[n]}$, 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, carrier cloth, applied pressure
Compressed "Shop Air" Blast	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/Release Fabric	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

Surface Durability of Aluminum (Wedge Test) (D 3762). Materials and processes typical of those used for aircraft manufacture 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 regulating agencies, such as the FAA, with interpreting data related to certification and evaluation procedures. The above testing is intended to supplement the conventional lap shear testing (ASTM D1002) and related joint subcomponent development testing for the assessment of short-term structural integrity.

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, previous researchers have established that the lap shear tests provide limited verification of the general reliability of bonded assemblies where prolonged loading and variable environmental conditions are involved [*5,11–12*]. Confirmation of bond integrity under such conditions typically requires some form of peel-type (mode I-dominated) test configuration, such as the wedge test, used for durability and surface preparation/process quality evaluation for bonded metals. In addition, it was found that, for metal adherends, neither lap shear tests conducted over a range of temperatures nor unstressed lap shear tests under environmental exposure duplicate disbond behavior from bonded joints in service [*8*].

In summary of this review, shear tests are not as good an indicator of long-term bond durability as mode I cleavage tests. Additionally, the attainment of pure mode II-type loading conditions is rarely achieved in practice. There always exists some peel component from eccentric load paths near joint edges [*8*]; mode I DCB tests are, consequently, an appropriate test for evaluating surface preparation and durability-related phenomena of bonded joints.

Floating Roller Test

The initial approach utilized floating roller tests with woven fiberglass and carbon fiber/epoxy samples. The ASTM D 3167 test is designed for a moderately 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. It was recognized that the radius of curvature that developed tended to be so small that the thin adherend fractured before the bond could be broken (Fig. 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.

Double Cantilever Beam and Static Wedge Tests

Literature research and a review of standard test methods revealed that the DCB and wedge tests (Fig. 2) are well-suited to evaluating the short- and long-term characteristics of adhesive bonds [*4–5,11–18*]. In the DCB test, a bonded sample is pulled apart at a constant test machine crosshead displacement rate by fixtures (hinges or pinned blocks) at the end of the beams. The free rotation in these fixtures allows pure tensile loading on the specimen arms with no applied moment. The specimen is loaded and unloaded under displacement control until the crack has propagated entirely through the sample (alternately, the specimen can be tested in one continuous load cycle without unloading). The static wedge test can be performed with the same specimen, but a tapered wedge is driven into the crack opening to stimulate the crack growth. The sample is usually held at elevated temperature and humidity to advance aging effectively, allowing for reliable short-term predictions of bond integrity of a joint over long periods of time in service [*3–10,12,19*]. The crack tip propagation is observed as the interface degrades, while the wedge remains in its initial position.

The DCB tests performed in this study are intended to provide a foundation upon which to extend the investigation of future wedge testing on similar geometrical configurations. The wedge test samples 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 displacement tests of the same test specimen.

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 com-

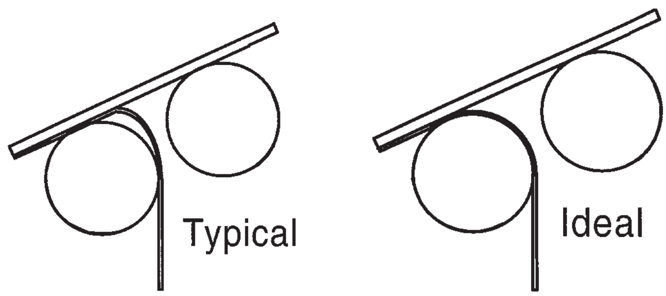


FIG. 1—*Typical composite adherend versus ideal metal adherend floating roller test.*

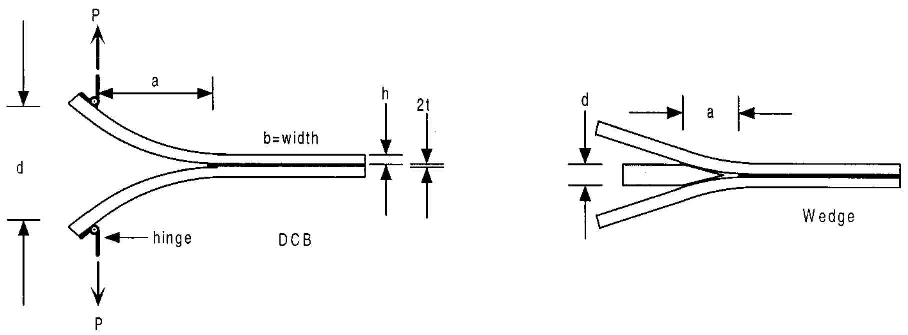


FIG. 2—*DCB and wedge tests.*

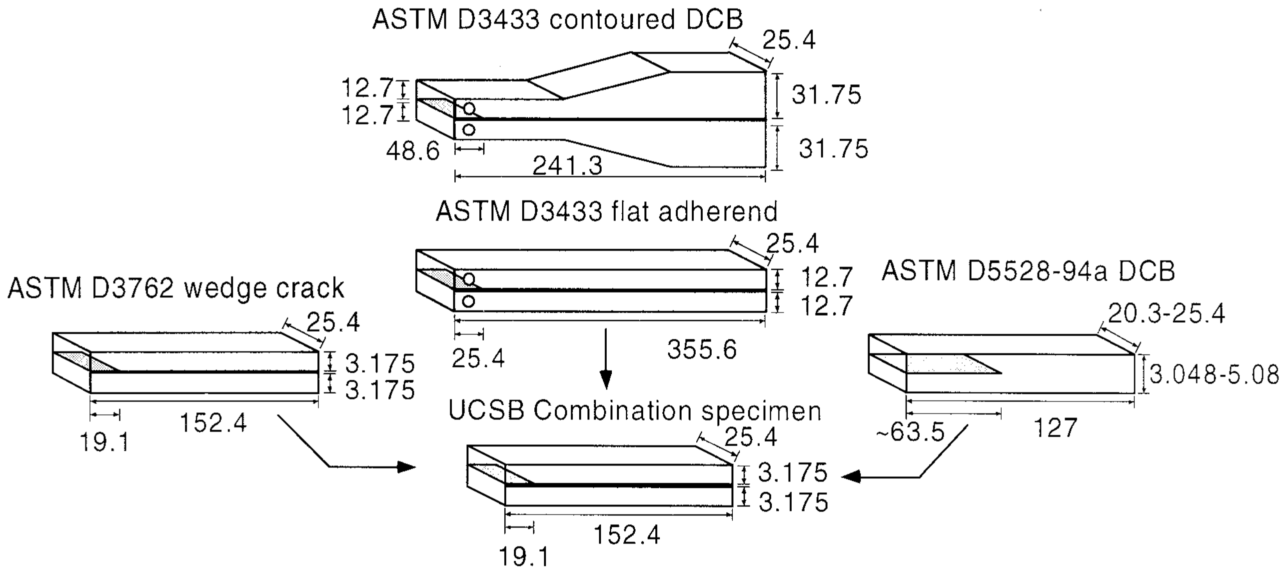


FIG. 3—*ASTM test specimens combined into one sample geometry. All dimensions are in mm.*

posites. The new specimen used for the DCB and wedge tests is based on those of ASTM D 3433, ASTM D 3762, and ASTM D 5528 (Fig. 3).

After completion of the sequence of tests conducted and reported herein, it was determined that a longer DCB specimen would be beneficial to testing, as more data points can be obtained for each test, especially with those samples that exhibit start-stop behavior and fracture quickly under low loads. The proposed new specimens are 304.8 mm (12 in.) long and 12.7 mm (0.5 in.) wide—half the width of the ones used in this study. Although very small widths can affect fracture toughness values of specimens (because they may tend to exhibit plane stress behavior instead of plane strain), previous work [*4,10,20*] shows that the specimen geometry of these new dimensions should not affect test results.

Specimen Preparation—DCB tests were performed on Hexcel® 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). 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, but relatively consistent bondline thicknesses suggest that they were. Despite the applied pressure (by piling on weights) during curing, bondlines never approached 0.127 mm (0.005 in.), while some bonds were as large as 0.508 mm (0.020 in.).

All adherends were cured with Chemfab® VB-3™ PTFE vacuum bag (VB) film on the bottom surface (tool side) and with a nylon release fabric (RF) with silicone and siloxane release agents on the top side (Fig. 4). This lay-up process creates panels with different surface properties on each side. Samples were bonded in one of two orientations: release fabric to release fabric (RF - RF) or vacuum bag to vacuum bag (VB - 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.

The composite samples that were grit-blasted (with Mil-A-2222B grit) were done so at 0.413 KPa (60 psi) regulator line pressure. The hinges used to hold the samples in the test machine grips were made from 1.016-mm (0.04-in.) thick continuous hinge, cut to 25.4-mm (1-in.) lengths and grit-blasted at 689 KPa (100 psi). The surfaces to which the hinges were bonded on all samples were also blasted at 0.413 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.

Before testing, the sides of samples were spray painted white and tick marks were penciled on manually at 1.588-mm (0.0625-in.) intervals for visual crack tip observation during the tests.

Testing and Data Reduction Methods—Specimens were loaded into an Instron 8562 test machine by clamping the hinges in grips. The test machine ran 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.

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: area, compliance, simple beam theory, and displacement methods [*21*].

The calculation methods discussed below do not take into account the effect of the adhesive layer and its interfaces. In tests conducted on adhesively bonded Aluminum DCB specimens, it was found that the adhesive layer need not be accounted for in beam theory-based G_I calculations, provided that certain geometrical conditions are met [*22*]. Using the more complex equation that included the adhesive layer, a consistent experimental 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 (Eq 1), 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 when

$$\frac{a}{h-t} > 8 \qquad (1)$$

where (see Fig. 2)
a = crack length
h = height of one DCB arm
t = half the thickness of the adhesive layer

For the specimens tested at UCSB, h = 3.175 mm (0.125 in.), t = 0.127 mm (0.005 in.), and the starter crack was 50.8 mm (2 in.) beyond the load point, putting the specimens well beyond the threshold value, so the G_{Ic} calculations herein need not include the adhesive layer.

The area method is based upon a change in the DCB sample's compliance (Eq 2) resulting from a change in crack length (Eq 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, Eq 3 gives the fracture toughness of the specimen [*23*]. When Eq 4 is used for multiple crack extensions per test, the specimen's calculated G_{Ic} value is the average of these individual calculations.

$$C = \frac{d}{P} \qquad (2)$$

$$G_I = \frac{1}{b}\frac{dU}{da} \qquad (3)$$

$$G_{Ic} = \frac{1}{2b\Delta a}(P_1 d_2 - P_2 d_1) \qquad (4)$$

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

The modified beam theory method is based on the "displacement" method [*21,23,24*] and detailed in the ASTM D5528 test method. From basic beam theory, Eq 5 describes the strain energy release rate of the specimen. This reduces to Eq 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 (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.

$$G_I = \frac{P^2}{2b}\frac{dC}{da} \qquad (5)$$

$$G_I = \frac{3Pd}{2ba} \qquad (6)$$

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.

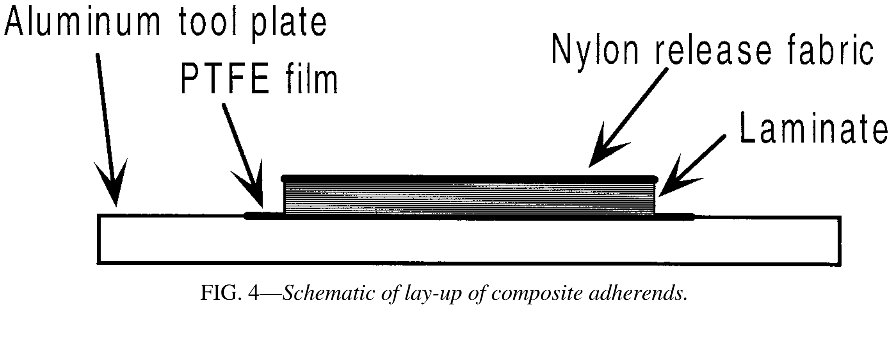


FIG. 4—*Schematic of lay-up of composite adherends.*

The crack length, *a,* must be offset by a value Δ to correct the error (Eq 7).

$$G_I = \frac{3Pd}{2b(a + \Delta)} \qquad (7)$$

This 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 (Fig. 5). The 1/3 power term in the plot is from the compliance-crack length relationship, which states that compliance$^{1/3}$ is proportional to crack length [*21*]. The excellent curve fits obtained in this study confirm the consistency of using a one-sided optical tick-mark measurement method for determining the crack tip location, a former concern.

Results—The results of the DCB tests come in many forms, each of which can indicate the bond quality. 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 start-stop behavior. The post-failure fracture surfaces, with adhesive and/or cohesive failure modes, show whether or not the adhesive bonded properly to the adherend. Finally, the calculated G_{Ic} values quantitatively show how much energy must be put into the specimen to create fracture surfaces. Although this is not a direct indication of bond durability, G_{Ic} is a parameter in the fatigue calculation for a crack, indicating some relation between G_{Ic} and durability.

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

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

Just as important as quantitative values, such as loads and displacements, is the more qualitative analysis of modes of failure. Well-bonded joints should fail within the adhesive (cohesive failure) and/or within the adherends (interlaminar failure) when broken apart. Failure at the adherend-adhesive interface (interfacial failure) generally indicates that the joint's surface preparation was not performed properly, likely a result of the silicone and siloxane release agents that were deposited onto the adherend surface from the release fabric 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 (Fig. 7), a few more trends were clear:

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

The critical strain energy release rates calculated for the DCB test specimens (Table 2 and Fig. 8) follow the observed trends in the load-displacement curves (Fig. 6) and the fracture surfaces (Fig. 7). Calculations were performed with both the area and MBT methods. 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, perhaps a result of choosing single data points rather than pairs when performing the MBT calculations.

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

1. Bonds made to vacuum bag surfaces produced higher G_{Ic} values than bonds to nylon release fabric surfaces.
2. Bonds made to grit-blasted surfaces produced higher G_{Ic} values than their non-blasted counterparts, regardless of previous surface preparation. This implies partial removal of the silicone and siloxane release fabric's chemical release agents and/or minor mechanical "keying" effects—enough to improve the bond strength, but not enough to change the mode of failure.

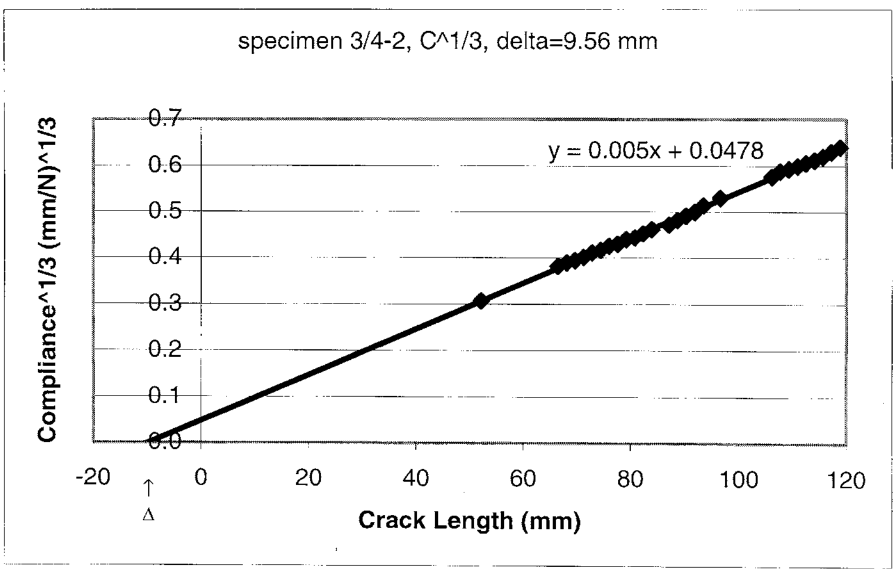


FIG. 5—*Sample compliance*$^{1/3}$ *versus crack length plot to determine crack offset for MBT method.*

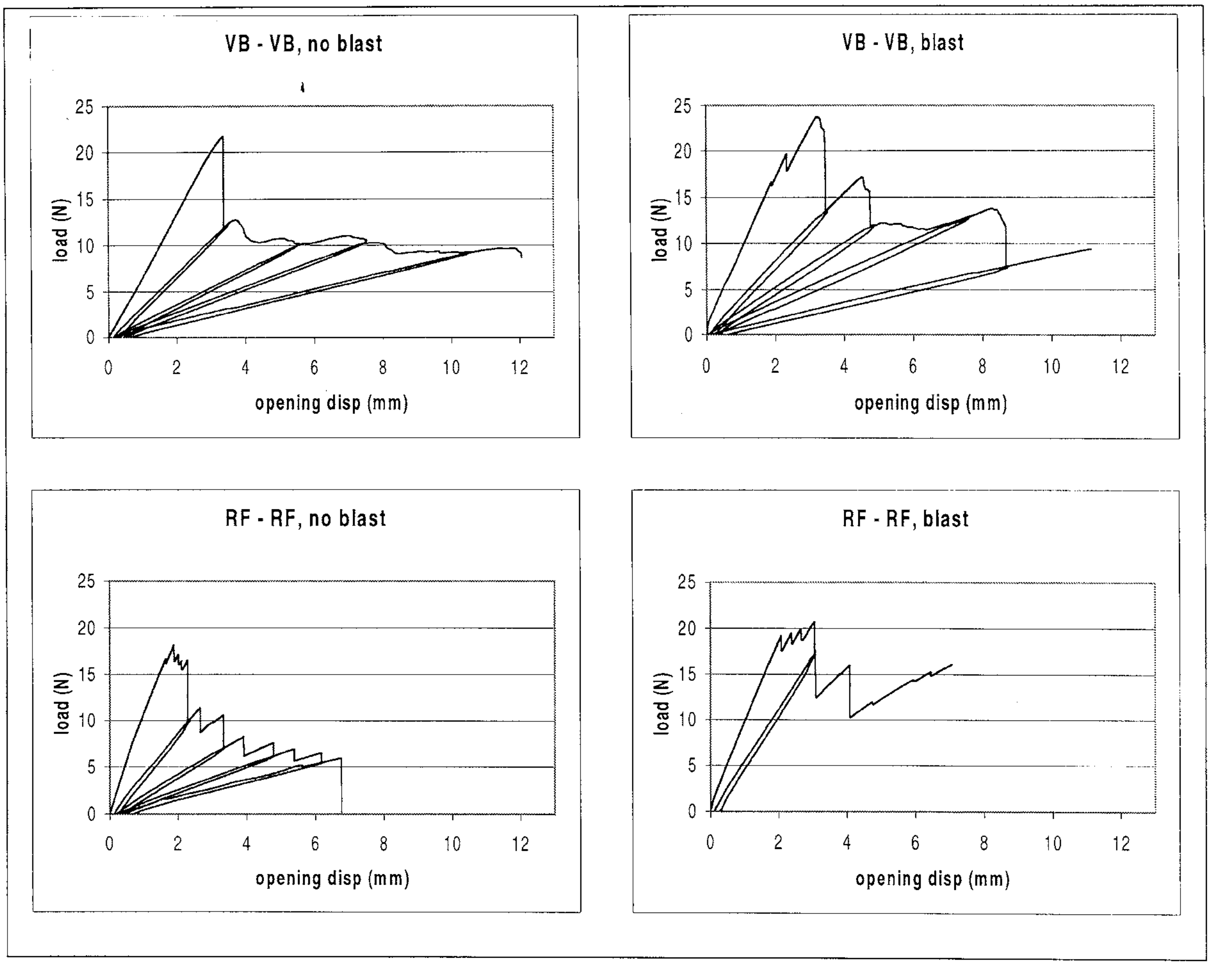
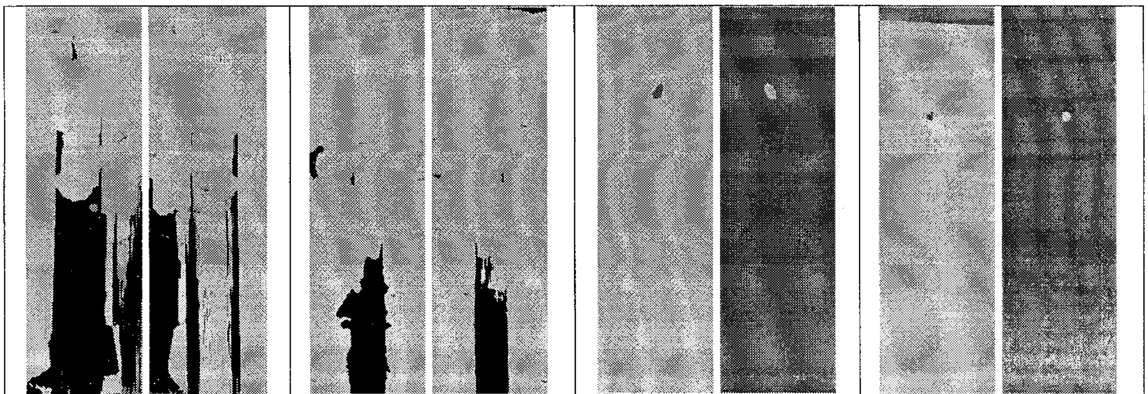


FIG. 6—*Sample load-displacement curves for bonded composite DCB tests.*



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

FIG. 7—*Computer-enhanced pairs of fracture surfaces of bonded composite DCB specimens.*

TABLE 2—*DCB critical strain energy release rate test results.*

Data Reduction Method	RF–RF, No Blast	RF–RF, Blast	VB–VB, No Blast	VB–VB, Blast
Area G_{Ic}: kJ/m^2	0.218	0.422	0.422	0.540
MBT G_{Ic}: kJ/m^2	0.205	0.448	0.407	0.497

Another point of interest is the absolute value of G_{Ic} for these bonds, relative to those in the literature. For well-prepared joints, in this case a VB-VB blasted joint, the average G_{Ic} is 0.540 kJ/m^2 (3.086 in.-lb/in.^{2}). The manufacturer of the EA9394 adhesive used in this research indicates a G_{Ic} of 1.02 kJ/m^2 (5.83 in.-lb/in.^{2}) in technical service laboratory report #90-06-31. This value was at-

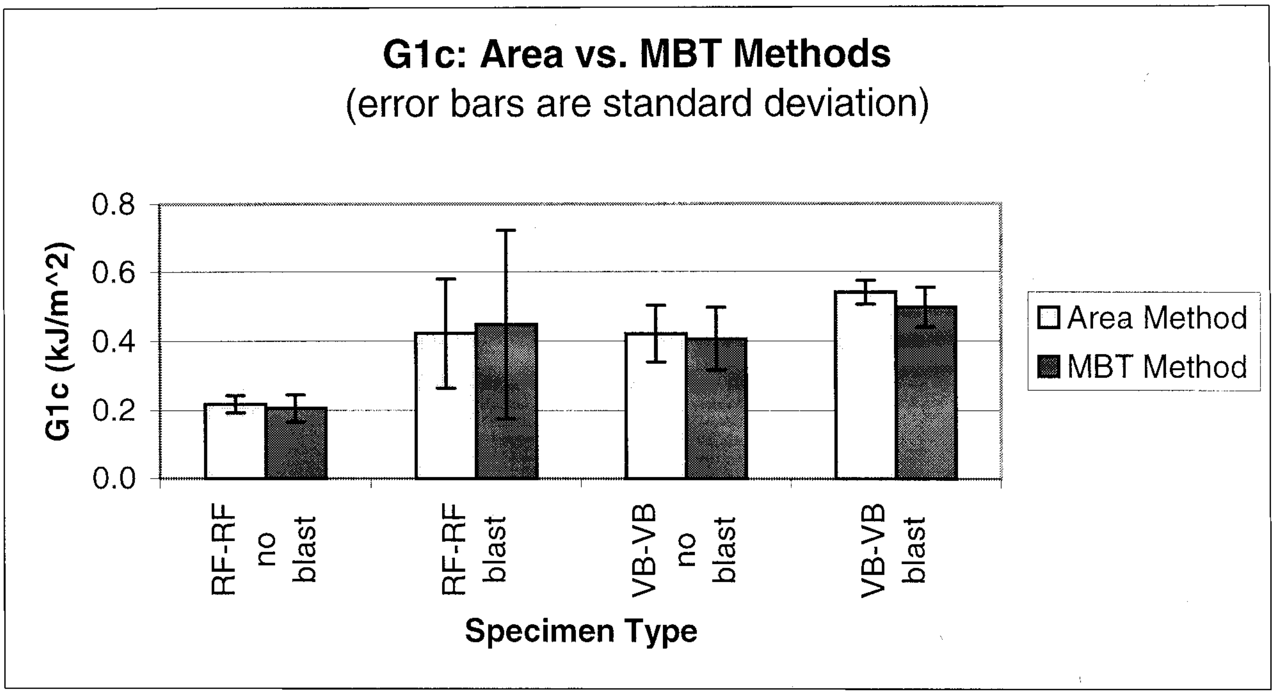


FIG. 8—*DCB critical strain energy release rate tests results.*

tained with aluminum 2024 T351 adherends, prepared with a phosphoric acid anodize etch, and with a bondline controlled by 0.127-mm (0.005-in.) glass beads. Additionally, the value for the interlaminar critical strain energy release rate of an IM7/8552 laminate is 0.233 kJ/m^2 (1.33 in.-lb/in.^{2}) [*25*]. The values obtained in this study fall between these two published values. It appears that the well-prepared bonded joints experienced interlaminar failure in addition to cohesive failure (see Fig. 7), because of the relatively low interlaminar G_{Ic} of the adherends, relative to the adhesive, never reaching the values obtained with metal adherends.

Conclusions

DCB and wedge test results accurately predict short-term strength and long-term adhesive bond durability in service, respectively, indicating that surface preparation methods that affect bond strength can be evaluated with these methods. DCB testing suggests 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 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 release fabric rather than a PTFE vacuum bag film showed the following trends:

1. Failure at lower loads and corresponding opening displacements.
2. Intermittent crack propagation.
3. Lower G_{Ic} values.
4. Totally interfacial, not cohesive, failure.

Wedge tests, in an elevated temperature and humidity environment, with the same custom bonded specimen (Fig. 3), will utilize DCB test results to predict joint failure in the projected research.

Due to geometric constraints and the low in-plane fracture strain of most contemporary composites materials, floating roller tests were found unsuitable for testing bonded composites for the thicknesses used herein.

Acknowledgments

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Dedication in Memory of Donald W. Oplinger

It is our wish to dedicate this contribution as a tribute to and in remembrance of *Donald W. Oplinger* for his support and guidance as sponsor, mentor, colleague, and friend. Until his passing on June 12, 2000, Don was R&D manager for Advanced Materials at the FAA Technical Center, New Jersey and was responsible for the organization of the workshop on "Bonded Joints and Assemblies in Aircraft" at which the reported research was originally presented. This workshop formed part of the American Society for Composites 15th annual conference held at Texas A&M University, September 26, 2000.

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