

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



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**FAA**



# Outline

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- **Industry collaboration and acknowledgements**
- **Motivation**
- **Surface preparation considerations**
- **Double cantilever beam (DCB) tests: preparation, analysis, results**
- **Travelling wedge tests: verification, preparation, analysis, results**
- **Work in progress**
- **Planned future work: static wedge, further travelling wedge, SEM, EDS/XPS**
- **Summary**

# **Industry Collaboration and Acknowledgements**

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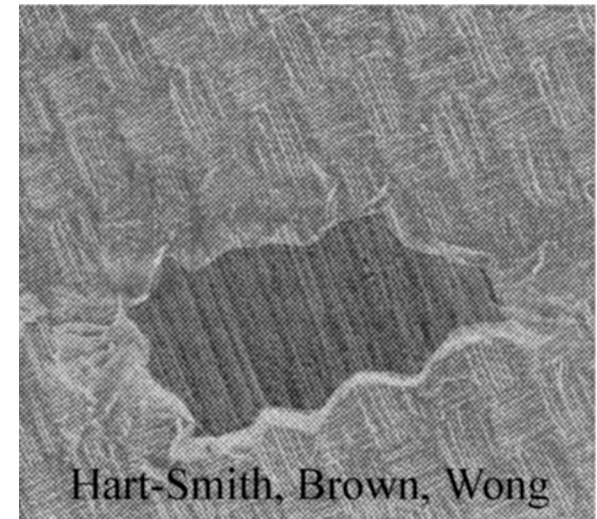
- **The Boeing Company/Cirrus Design/Lancair: industry experience**
- **Composite Optics, Inc.: fabrication, paste adhesive experience**
- **Cytec Industries: film adhesive**
- **FAA: funding, industry application**
- **Hexcel: prepreg composite material**
- **Precision Fabrics Group: peel plies and release fabrics**
- **Richmond Aircraft Products: processing supplies and tools**
- **UCSB: all research, testing, experiments, equipment**
- **WSU: materials and specimens, early funding**

# Motivation

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- **Minimization or *prevention* of interfacial failure in adhesive bonds**
- **Characterization of the effects of the following on bond durability:**
  - **Chemical contamination from release fabric or other source**
  - **Surface preparation**
  - **Environmental effects (temperature, moisture, chemicals, etc.)**
- **Develop recommended practices for fabrication to ensure *consistent* bonds**
- **Provide *quantitative* technical foundations for certification of bonding procedures**

“Surface Preparations for Ensuring that the Glue Will Stick in Bonded Composite Structures”



## Surface Preparation Variable Details

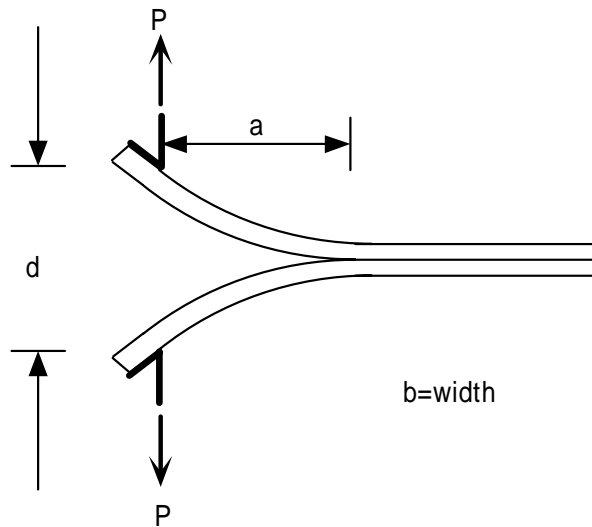
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- **Release Fabric/Peel Ply: chemical contamination possibility**
- **Abrasion: hand abrade (operator inconsistency, “folding” [Davis & Bond 1999]), grit blast (media, pressure, cleanliness)**
- **Time: adherend drying before bonding, between release fabric removal & adhesive application, between adhesive application & assembly (amine carbonate formation)**
- **Order of operations: cleaning before/after abrasion [Caldwell 1999, Harris & Beevers 1999]**
- **Do chemical or mechanical factors affect bond strength & durability? chemical [Davis & Bond 1999, Harris & Beevers 1999]**

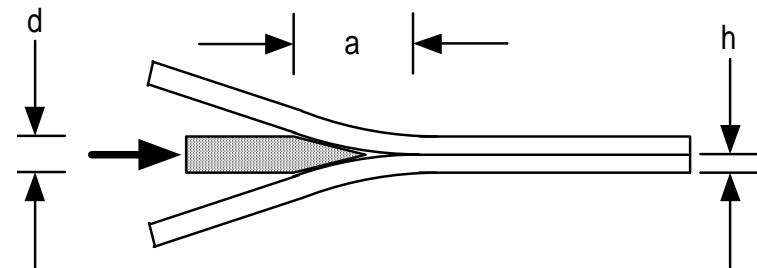
## Test Methods to Determine Adhesive Bond Durability

- Bonded joints designed to carry *mode II* shear load, not *mode I*
- Carpenter & O'Donnell 1988: floating roller test more sensitive to & more affected by surface prep & aging than lap shear test
- Davis & Bond 1999: “lap shear test...only verify the short-term strength,...not durability”, “shear & peel strength...not sufficient to assure durability”, “[static] wedge test is ideal for ...durability”
- Hart-Smith 1997: “lap shear test...tells...nothing”, “process control...[static] wedge-crack tests...ensure long-term durability”
- Marceau 1976: “lap shear  $f(T)$ ...peel  $f(T)$ ...unstressed lap shear  $f(\text{environment})$ ...not duplicate...service disbonds”

# Preliminary DCB and Wedge Tests



Double Cantilever Beam

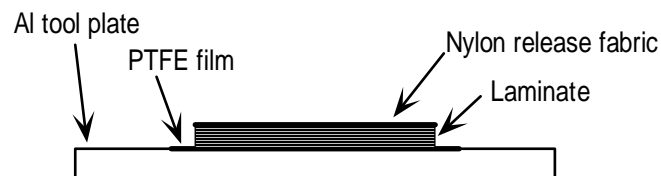


Wedge (static or traveling)

- **Similar test geometries—can compare results between the two**
- **DCB: pull apart adherends at a constant, slow velocity**
- **Static wedge: insert wedge, place in environment, observe crack**
- **Travelling wedge: force wedge through bondline at slow velocity**

## DCB Test Specimen Details (Paste Adhesive)

- **IM7/8552 22-ply unidirectional adherend ×2**
- **Adherend panels cured with Chemfab VB-3 PTFE vacuum bag film (vb) on tool side and nylon release fabric (rf) on other side to create 2 different surfaces on panel (for rf-rf or vb-vb bonds)**
- **Blasted & unblasted specimens scrubbed with Scotch-Brite pads before bonding**
- **Adherends bonded with Hysol EA9394 2-part epoxy paste adhesive with 0.005” glass beads mixed in 2.5% by weight**
- **1” mild steel piano hinge segments bonded with Hysol EA9394 with 0.005” glass beads mixed in 2.5% by weight**



# DCB Test Specimen Details (Paste Adhesive)

- Data from literature

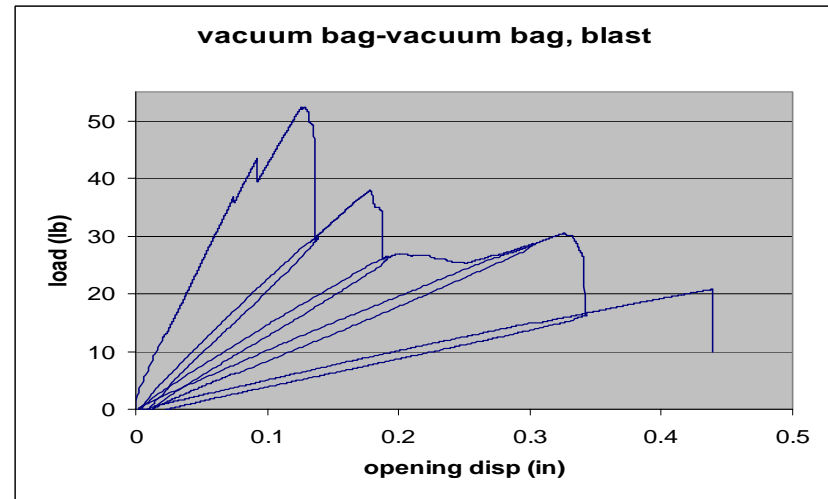
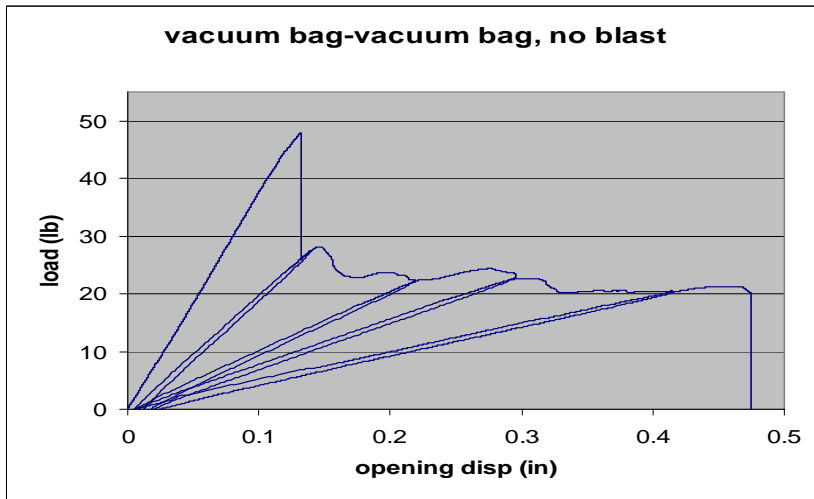
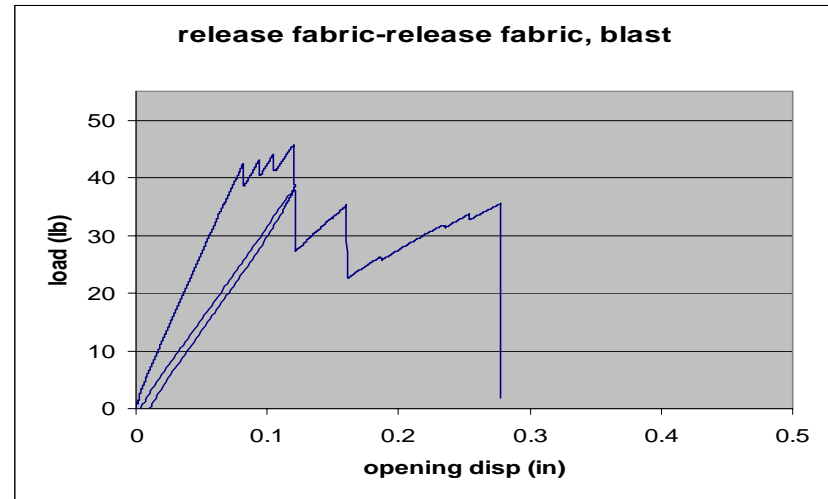
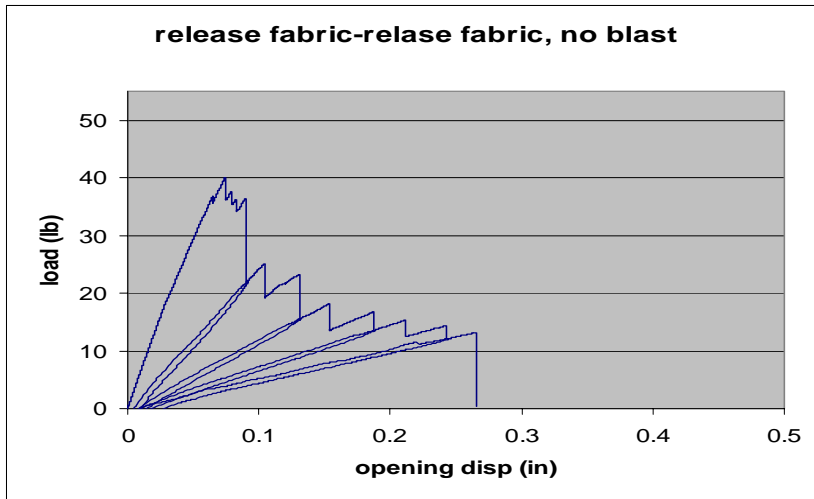
<u>IM7/8552 Unidirectional Ply</u>	<u>IM7/8552 Laminate</u>	<u>EA9394 Adhesive</u>
$E_1 = 23.8 \times 10^6$ psi [164 GPa] $E_2 = 1.7 \times 10^6$ psi [11.7 GPa] $G_{12} = 1.0 \times 10^6$ psi [6.89 GPa] $\nu_{12} = 0.28$ $\nu_{21} = 0.0163$ source: Hercules	$G_{Ic} = 1.33$ in-lb/in <sup>2</sup> [0.233 kJ/m <sup>2</sup> ] source: Cairns / Hercules	$G_{Ic} = 5.83$ in-lb/in <sup>2</sup> [1.02 kJ/m <sup>2</sup> ]  (DCB test with Al, phosphoric acid anodize etch, 0.005" glass bead bondline) source: Hysol

- Process variables

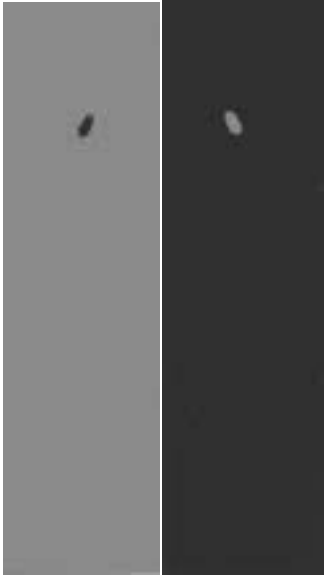
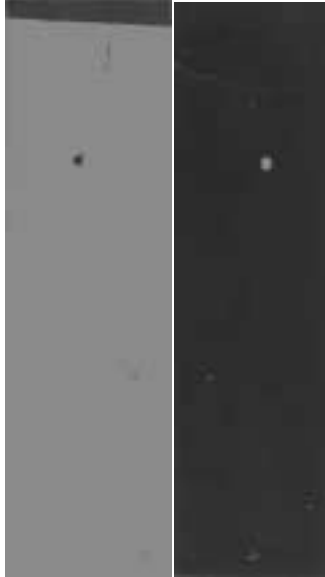

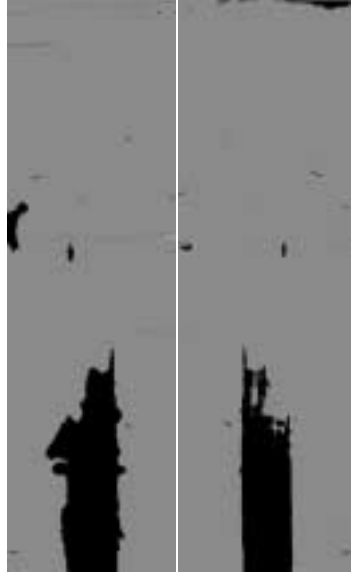
<u>Bonded IM7/8552 UCSB Specimens</u>	
Grit blast (#120 Garnet, Mil-A-2222B)	60 psi
Steel hinge grit blast	100 psi
Composite adherend hinge area grit blast	60 psi
Pre-bond cleaning	De-ionized H <sub>2</sub> O + Alkanox soap scrub with Scotch-Brite pad, oven dry, isopropyl alcohol wipe, air dry
Adherend cure	12 hr @ room temp
Hinge cure (before handling)	4 hr @ 180°F
Crack starter	3" flashbreaker tape

- Hysol recommended cure: 5-7 days @ 77°F or 1 hr @ 150°F**

# DCB Test Sample Data (Paste Adhesive)



# DCB Test Sample Fracture Surfaces (Paste Adhesive)

crack travel ↓				
	<b>Bond</b>	release fabric - release fabric	release fabric – release fabric	vac bag – vac bag
<b>Blast</b>	no	Yes	no	yes
<b>Failure</b>	purely interfacial	purely interfacial	cohesive / interlaminar	cohesive / interlaminar

(Black is IM7/8552 adherend, gray is EA9394 adhesive)

- Grit blasting does not change mode of failure

## DCB $G_{Ic}$ Calculations

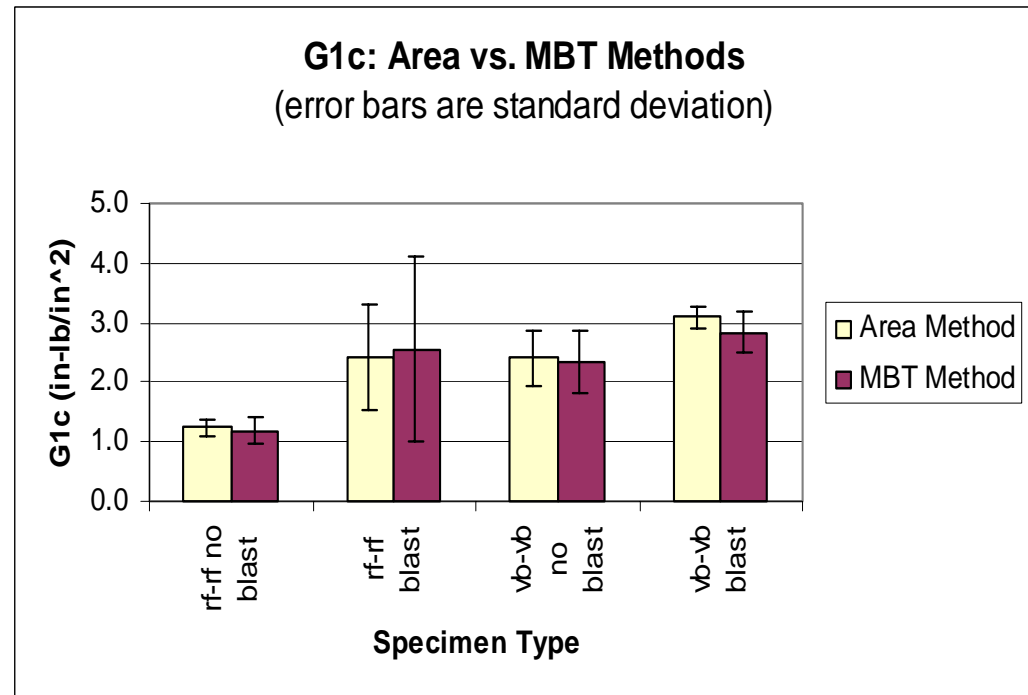
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- **Area Method**: based on change in strain energy (from change in compliance) with crack length, computed from area between loading & unloading curves
- **Modified Beam Theory Method**: based on simple theory with correction for incorrect assumption of cantilever beams rigidly supported at crack tip (which overestimates  $G_I$ ), beam actually rotates at “clamped” end—correct with offset from  $C^{1/3}$  vs. a plot
- **Adhesive Layer Considerations**: Fernlund & Spelt showed, for homogenous DCB data and beam theory/BOEF, OK to ignore adhesive for long cracks

## DCB Test Results (Paste Adhesive)

	rf - rf, no blast	rf - rf, blast	vb - vb, no blast	vb - vb, blast
<u>AREA <math>G_{Ic}</math>: in-lb/in<sup>2</sup></u>	1.244 ± 0.144	2.412 ± 0.896	2.410 ± 0.464	3.086 ± 0.198
<u>MBT <math>G_{Ic}</math>: in-lb/in<sup>2</sup></u>	1.174 ± 0.224	2.560 ± 1.565	2.328 ± 0.516	2.843 ± 0.326

- **Release fabric vs. vac bag:**  
vacuum bag → higher  $G_{Ic}$
- **Grit blasting vs. not: blast**  
→ higher load &  $G_{Ic}$  (esp. on release fabric surfaces)
- $G_{Ic}$  of EA9394 w/ Al adherend is 5.83 &  $G_{Ic}$  of



**IM7/8552 interlaminar is 1.33, bracketing UCSB's test results**

# Travelling Wedge Tests Introductory Notes

- Variant on static wedge test where wedge is forced slowly (0.125 in/min quasi-static loading condition) through specimen
- Simple specimen fixturing and testing (no hinges/alignment)
- Analysis based on fracture mechanics / beam on elastic foundation
- Test results extremely sensitive to crack length measurement (4<sup>th</sup> power term)

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

(Tada, Creton et al., Jiao et al.)

## Preliminary Travelling Wedge Tests (Paste Adhesive)

- Set of specimens tested with travelling wedge & DCB tests
- Instron, SST wedge (0.1191”), telescope

Test Type	Bond type	# of samples tested	$G_{Ic}$ average in-lb/in <sup>2</sup>	$G_{Ic}$ standard deviation in-lb/in <sup>2</sup>
DCB (from previous tests)	vacuum bag-vacuum bag, no blast	4	2.328	0.516
DCB	vacuum bag-vacuum bag, no blast	2	2.00	0.316
Travelling wedge	vacuum bag-vacuum bag, no blast	17	1.89	0.922
DCB (from previous tests)	release fabric-release fabric, no blast	3	1.174	0.224
Travelling wedge	release fabric-release fabric, no blast	4	1.675	0.150

## **Travelling Wedge Tests: Materials (Film Adhesive)**

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- **Adherend:**

- **[0]<sub>11</sub> double-thickness IM7/M73 prepreg → beam height ≈ 0.12”**
- **Cure: 355° F, 6 hours, ~95 psi**
- **Cured against A5000 FEP release film, 60001 NAT polyester peel ply, 60001 SRB polyester release fabric, or 60001 VLP calendered polyester peel ply**

- **Adhesive:**





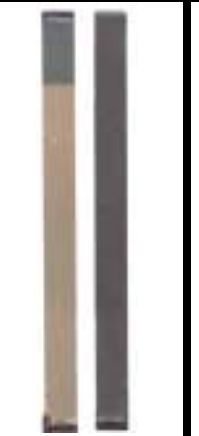



- **FM300-2K film adhesive → bond thickness ≈ 0.011”**
- **Cure: 250° F, 1.5 hours, 40 psi**

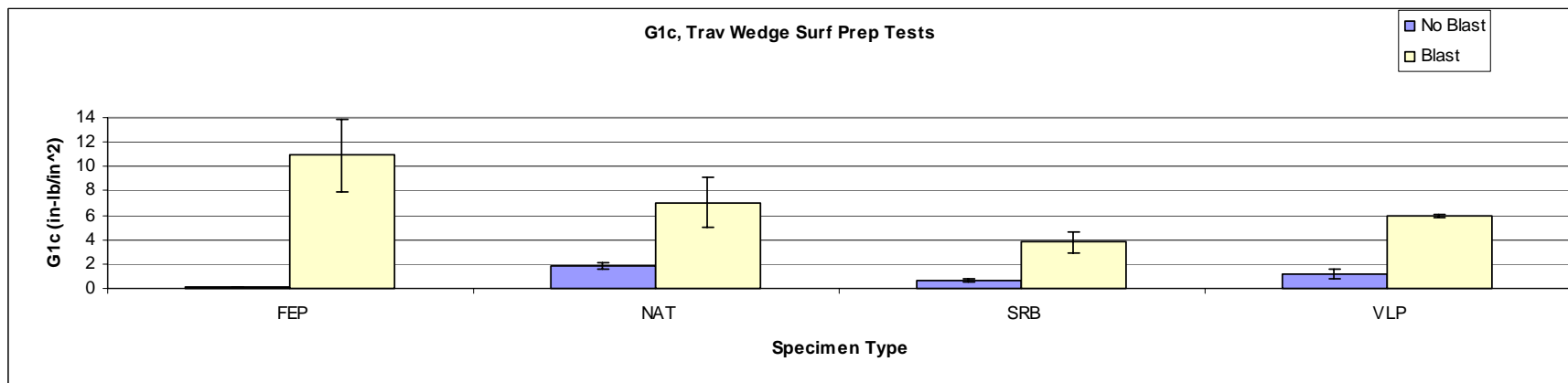
## Travelling Wedge Tests: Surface Prep (Film Adhesive)

Non-Blasted Specimens	Blasted Specimens
Keep peel ply/release fabric/release film on panel surface	
Cut panels with abrasive circular saw with deionized H <sub>2</sub> O	
Dry panels for > 12 hours in 180° F oven with exhaust	
Remove peel ply/release fabric/release film	
-	Acetone wipe
-	Blast: 40psi, ~5-7" away, angle 80-90° from surface, 2 passes at ~2 in/sec, Al <sub>2</sub> O <sub>3</sub> media, 180 grit/100-400 mesh size
Blow clean with 100psi dry N <sub>2</sub> blast	
Stack adhesive (thawed for ~16 hrs) between adherends, vacuum bag, cure	

- Specimens handled only with disposable rubber gloves
- ~1 hour between assembly and cure
- For each bond type, ≥ 3 specimens each from 2 identically prepared panels were tested → ≥ 6 specimens tested per group

# Travelling Wedge Tests: Results (Film Adhesive)

crack travel ↓  ↓ un-spoilt frac. surf.								
	<b>Bond</b>	<b>FEP, no blast</b>	<b>FEP, blast</b>	<b>NAT, no blast</b>	<b>NAT, blast</b>	<b>SRB, no blast</b>	<b>SRB, blast</b>	<b>VLP, no blast</b>
<b>Failure</b>	interfacial	cohesive/ interlaminar	interfacial/ interlaminar	cohesive/ interlaminar	interfacial	interfacial/ interlaminar	interfacial	cohesive/ interlaminar



## Travelling Wedge Tests: $G_{Ic}$ Results (Film Adhesive)

Surf Prep	$G_{Ic}$ [in-lb/in <sup>2</sup> ]	Failure Mode	Notes
FEP, no blast	< 0.086	interfacial	crack length > 5.5" specimen length
FEP, blast	13.4 ± 1.86 8.47 ± 1.24	cohesive/interlaminar	some cracks moved from cohesive to interlaminar
NAT, no blast	1.82 ± 0.254	interfacial/interlaminar	mostly interfacial
NAT, blast	7.06 ± 2.07	cohesive/interlaminar	
SRB, no blast	0.678 ± 0.0936	interfacial	white side paint usually seeped into bond
SRB, blast	3.79 ± 0.853	interfacial/interlaminar	
VLP, no blast	1.20 ± 0.351	interfacial	
VLP, blast	5.97 ± 0.129	cohesive/interlaminar	

- Data points from interlaminar fracture portions not used in  $G_{Ic}$  calculations (lowers  $G_{Ic}$  & raises standard deviation)

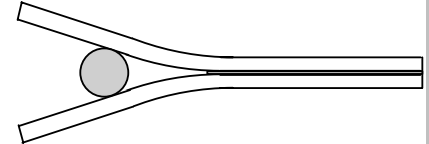
## **Travelling Wedge Test Discussion (Film Adhesive)**

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- **Blasting increased  $G_{Ic}$  significantly (& usually its standard deviation, perhaps from manual blasting technique, perhaps from randomness of texture and mechanical interlocking)**
- **Unblasted specimens tended to fail interfacially**
- **All blasted specimens failed partially interlaminarly (either interlaminarly/interfacially or interlaminarly/cohesively)**
- **Wedge disturbs fracture surface for post-test visual examination**
- **Test appears to be sensitive to preparation variations**

## Static Wedge Test (Film Adhesive) \*\*\*in progress!\*\*\*

- Insert 1/8"-dia steel dowel pin with hammer/razor blade/wedge
- 4 samples per group, results reflect 3-9 days of exposure
- Samples submerged in room temperature (71° F) de-ionized H<sub>2</sub>O (pH 7.3)
- Crack growth usually stabilized within 5 hours



Surf Prep	Avg Init Crack	Crack Advance	Notes
FEP, no blast	> 5.5"	N/A in 3/4, no in 1/4	3/4 split at start, 1 razor
FEP, blast	2.06"	0.22" in 1/4	no growth in 3/4
NAT, no blast	2.79"	0.016", 0.031" in 2/4	no growth in 2/4
NAT, blast	2.05"	0.031-0.13" in 3/4	1/4 mis-wedged (interlam)
SRB, no blast	3.84"	N/A in 3/4, total in 1/4	3/4 split at insertion
SRB, blast	2.51"	0.031-0.078" in 4/4	growth in 4/4
VLP, no blast	3.25"	N/A in 1/4, 0.13-0.31" in 3/4	1/4 split at start
VLP, blast	2.15"	0.047" in 2/4, no in 2/4	no growth in 2/4

## **Future Work (Film Adhesive)**

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- **Continue static wedge tests immersed in acidic & basic, or elevated temperature solutions**
- **Lap shear tests**
- **Physical comparison of surface treatments by surface morphology characterization (Scanning Electron Microscopy)**
- **Chemical evaluation of all 8 surface preparation types plus interlaminar surface: Energy Dispersive Spectroscopy (EDS), X-ray Photoelectron Spectroscopy (XPS)**

## **Summary (Paste Adhesive)**

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- **DCB tests:**
  - **Blasting increased  $G_{Ic}$  test values (more for rf-rf than vb-vb)**
  - **Blasting did not change mode of failure**
  - **Vacuum bag-cured surface bonds produced cohesive failures**
  - **Nylon release fabric surface bonds produced adhesive failures**
- **Paste posed difficulties in processing: absolute bond thickness, bond thickness consistency within & between specimens, alignment/fixturing**
- **DCB specimen prep. proved tedious: bonding/aligning hinges**
- **Initial set of travelling wedge tests matched DCB's  $G_{Ic}$  values well**

## Summary (Film Adhesive)

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- **Travelling wedge tests:**
  - **Grit blasting increased  $G_{Ic}$  test values greatly (and usually  $\sigma$ )**
  - **Grit blasting *did* change mode of failure (except for SRB)**
  - **All unblasted surfaces produced 100% interfacial failures**
  - **FEP  $G_{Ic}$  went from lowest to highest with blasting**
  - **Grit blasted VLP had tightest  $\sigma$  out of all blasted prep methods**
- **Static wedge tests: in progress—too soon to summarize**
- **Film adhesive's processing characteristics & results were excellent**
- **Travelling wedge specimen & test proved to be straightforward & reliable—minor upgrades**