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

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Outline

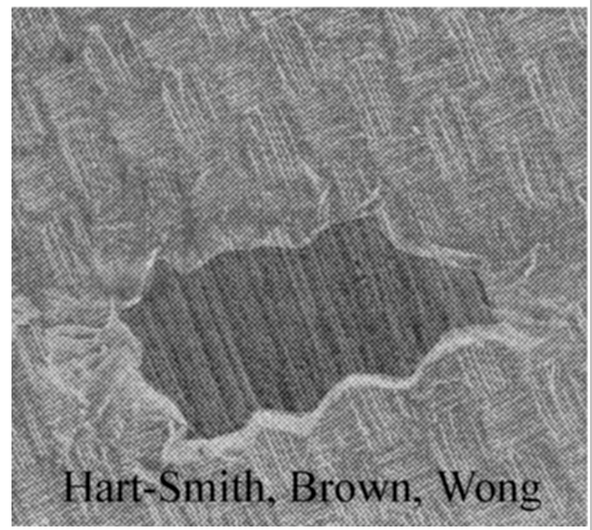
- **Industry collaboration and acknowledgements**
- **Motivation**
- **Surface preparation considerations**
- **Double cantilever beam (DCB) tests: preparation, analysis, results**
- **Travelling wedge tests: verification, preparation, analysis, results**
- **Work in progress**
- **Planned future work: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

Industry Collaboration and Acknowledgements

- 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

- **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

- **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? <u>chemical</u> [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***
- **<u>Carpenter & O'Donnell 1988</u>: floating roller test more sensitive to & more affected by surface prep & aging than lap shear test**
- **<u>Davis & Bond 1999</u>: "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"**
- **<u>Hart-Smith 1997</u>: "lap shear test…tells…nothing", "process control…[static] wedge-crack tests…ensure long-term durability"**
- **<u>Marceau 1976</u>: "lap shear *f*(T)…peel *f*(T)…unstressed lap shear *f*(environment)…not duplicate…service disbonds"**

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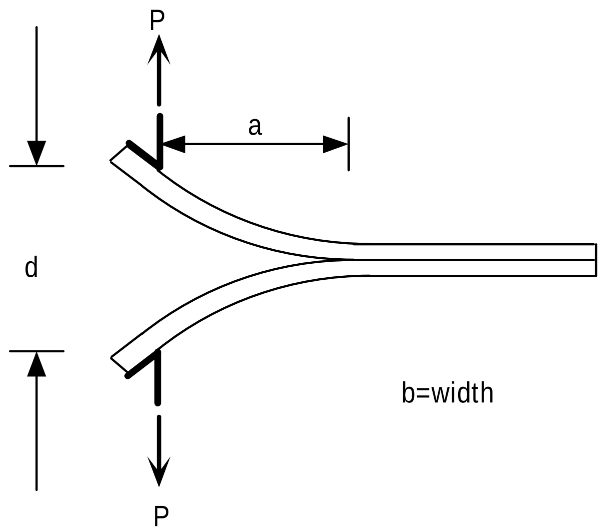
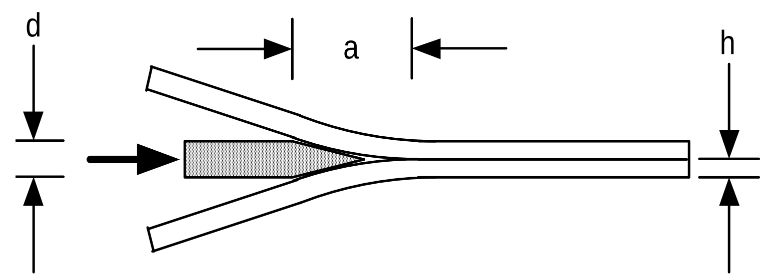
Preliminary DCB and Wedge Tests



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**

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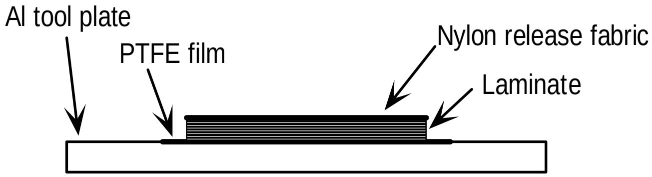
DCB Test Specimen Details (Paste Adhesive)

- **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**

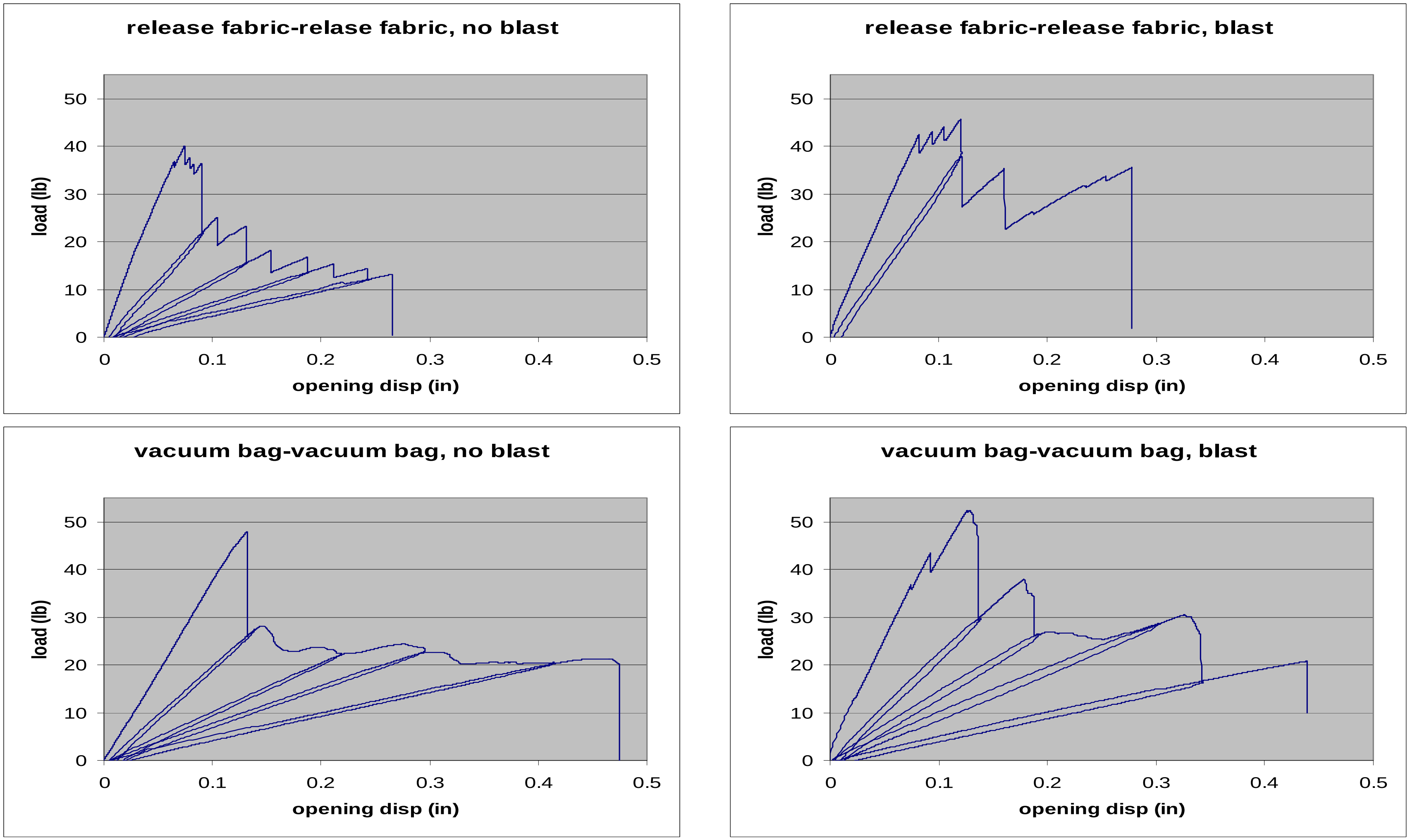
IM7/8552 Unidirectional Ply	IM7/8552 Laminate	EA9394 Adhesive
$E_1 = 23.8 \times 10^6$ psi [164 GPa] $E_2 = 1.7 \times 10^6$ psi [11.7 GPa] $G_{12} = 1.0 \times 10^6$ psi [6.89 GPa] $\nu_{12} = 0.28$ $\nu_{21} = 0.0163$ source: Hercules	G_{Ic} = 1.33 in-lb/in^2 [0.233 kJ/m^2] source: Cairns / Hercules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

- **Process variables**

	Bonded IM7/8552 UCSB Specimens
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_2O + 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)



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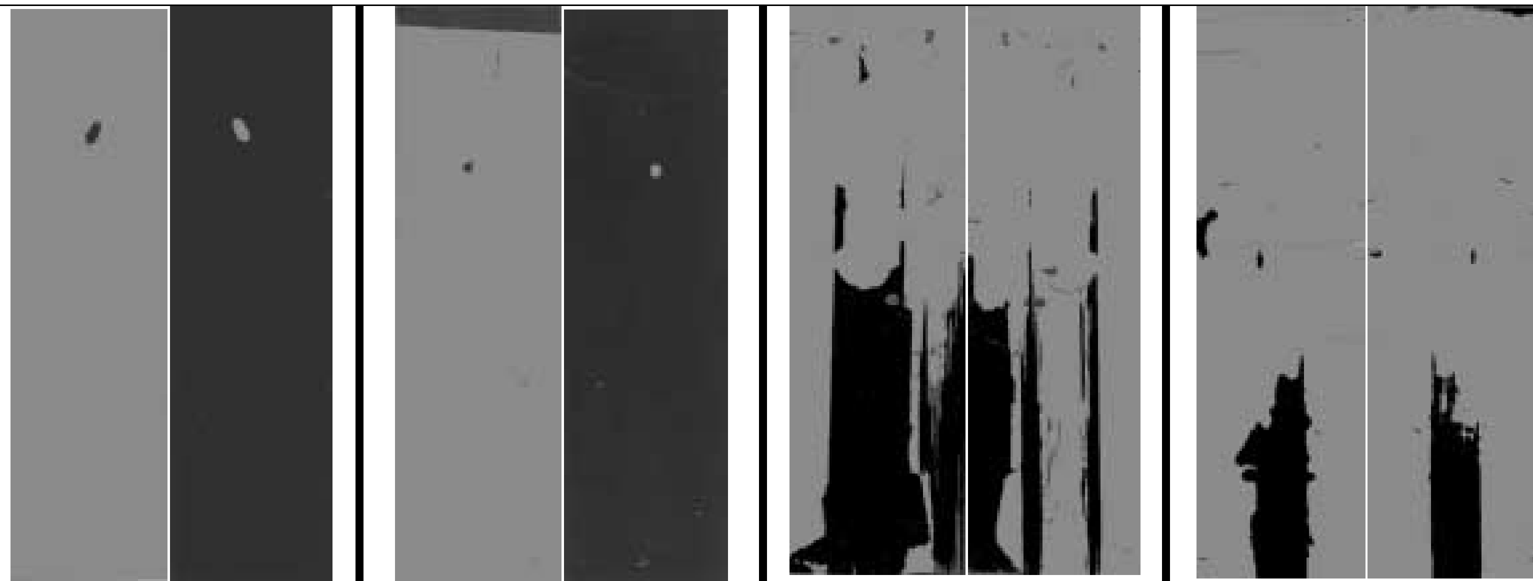
DCB Test Sample Fracture Surfaces (Paste Adhesive)

crack travel ↓



Bond	**release fabric - release fabric**	**release fabric – release fabric**	**vac bag – vac bag**	**vac bag – vac bag**
Blast	**no**	**Yes**	**no**	**yes**
Failure	**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**

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DCB G_{Ic} Calculations

- **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**

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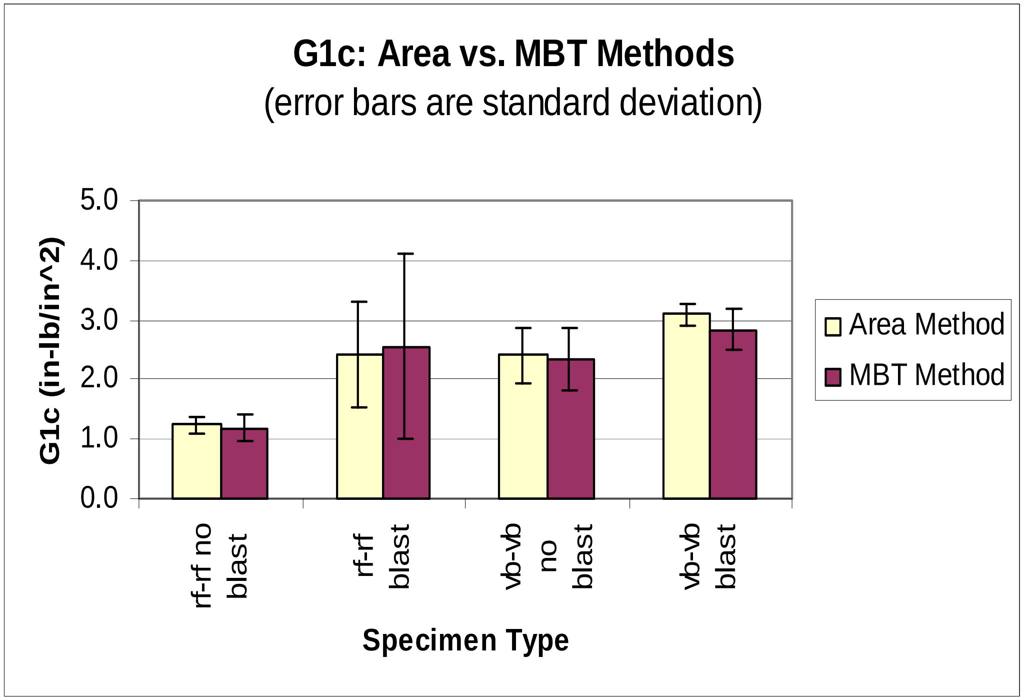
DCB Test Results (Paste Adhesive)

	rf - rf, no blast	rf - rf, blast	vb - vb, no blast	vb - vb, blast
AREA G_{Ic}: in-lb/in^2	1.244 ± 0.144	2.412 ± 0.896	2.410 ± 0.464	3.086 ± 0.198
MBT G_{Ic}: in-lb/in^2	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**



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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 (4th 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.)

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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^2	G_{Ic} standard deviation in-lb/in^2
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)

- **Adherend:**
  - **$[0]_{11}$ 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_2O	
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_2O_3 media, 180 grit/100-400 mesh size
Blow clean with 100psi dry N_2 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

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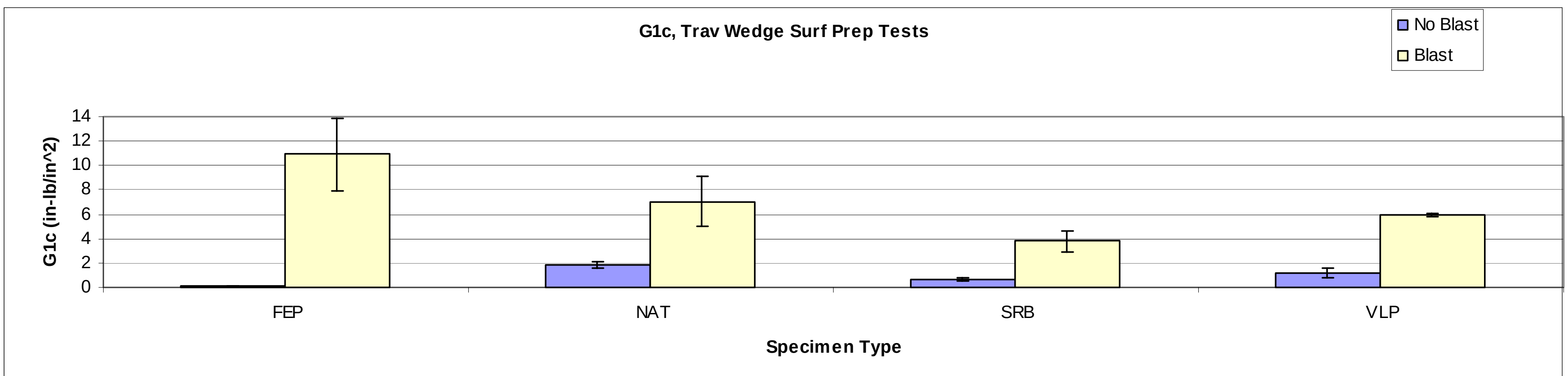
Travelling Wedge Tests: Results (Film Adhesive)

crack travel ↓

↓ un-spoilt frac. surf.

Bond	FEP, no blast	FEP, blast	NAT, no blast	NAT, blast	SRB, no blast	SRB, blast	VLP, no blast	VLP, blast
Failure	interfacial	cohesive/ interlaminar	interfacial/ interlaminar	cohesive/ interlaminar	interfacial	interfacial/ interlaminar	interfacial	cohesive/ interlaminar



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

Surf Prep	G_{Ic} [in-lb/in^2]	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)

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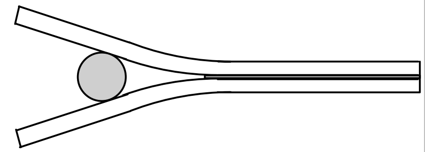
Travelling Wedge Test Discussion (Film Adhesive)

- **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_2O (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

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Future Work (Film Adhesive)

- 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)

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Summary (Paste Adhesive)

- 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)

- **Travelling wedge tests:**
  - **Grit blasting increased G_{Ic} test values greatly (and usually σ)**
  - **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 σ 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**