

The logo for the Joint Advanced Materials and Structures Center of Excellence (JAMS) is displayed at the top center. It consists of the letters 'JAMS' in a bold, blue, sans-serif font with a slight 3D effect. Below the text are two thick, curved lines: a yellow one on top and a blue one on the bottom, both curving from left to right.

JAMS

# The Effect of Surface Treatment on The Degradation of Composite Adhesives

Prashanti Pothakamuri

Lloyd Smith



# The Effect of Surface Treatment on The Degradation of Composite Adhesives

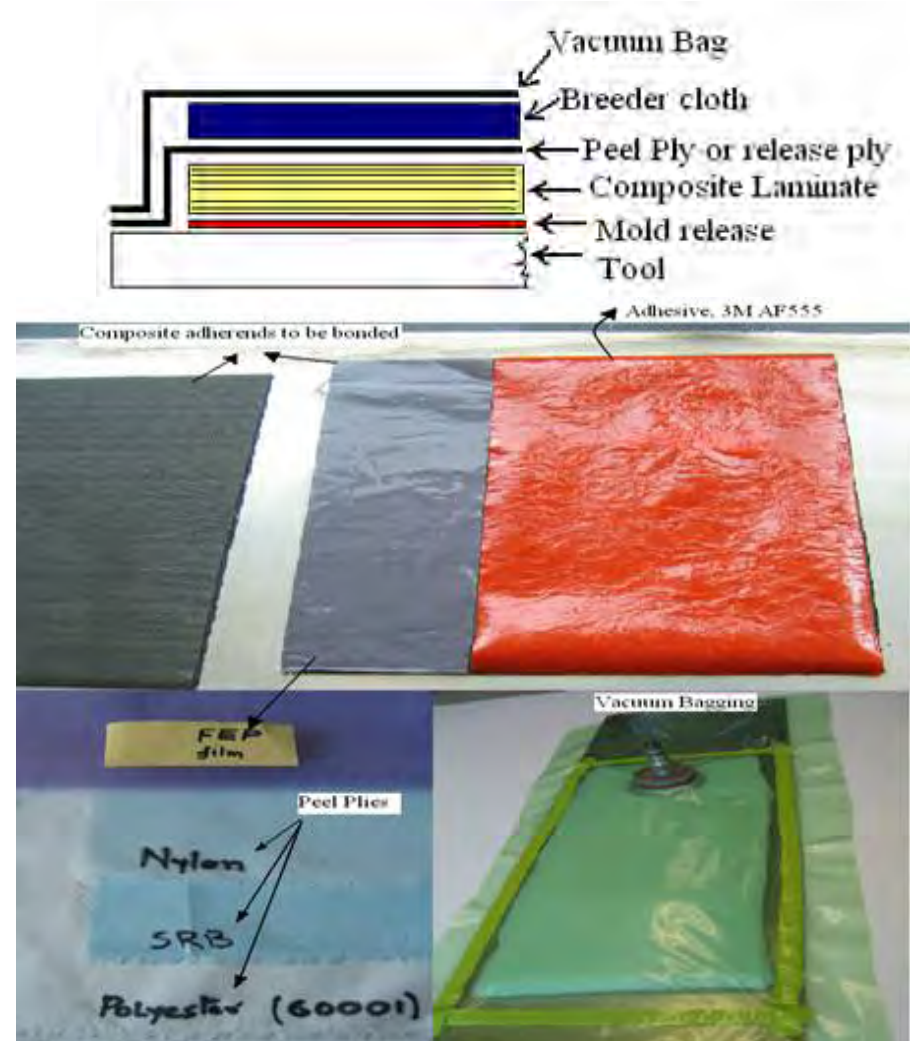
- Motivation and Key Issues
  - Commercial composite aircraft use surface preparations such as peel ply and abrasive techniques for bonding primary structure.
  - Critical parameters which dictate the durability of the adhesive bond are
    - Adherend surface quality
    - Pre-bond and post bond moisture effects
    - Service loads
- Objective
  - Quantify how surface preparation techniques affect the integrity of adhesive bonds.
  - Investigate test methods that may accelerate environmental degradation.

# JAMS The Effect of Surface Treatment on The Degradation of Composite Adhesives

- Approach
  - Compare relative degradation in 140°F water
    - Crack growth
    - Residual strength
    - Failure mode
  - Material
    - Boeing 8-276 form 3 laminates
  - Surface preparations
    - Peel ply: Polyester (precision fabrics 60001)- **fine**  
Nylon (precision fabrics 52006)- **medium**  
Siloxane coated polyester (super release blue, SRB) - **coarse**
    - Sanding: Grit-220
    - Grit blasting: Grit-220 and Grit-80
  - Adhesive Type
    - 3M AF555

- Principal Investigators & Researchers
  - Lloyd Smith
  - Prashanti Pothakamuri
- FAA Technical Monitor
  - Peter Shyprykevich
- Other FAA Personnel Involved
  - Curt Davies
- Industry Participation
  - Boeing: Peter VanVoast

- Material Specifications
  - BMS 8-276 Form 3 (Toray) uni-tape
  - Peel ply (surface prep)
  - 3M AF555 adhesive
- Laminates autoclave cured, 350°F and 85 psi, 45 min ramp, 2 hr soak
- Coupons bonded with AF555 (3M) at 350°F
- Coupons machined to form:
  - Double cantilever beam (DCB)
  - Wide area lap shear (WLS)
  - Wedge crack (WC)



# The Effect of Surface Treatment on The Degradation of Composite Adhesives

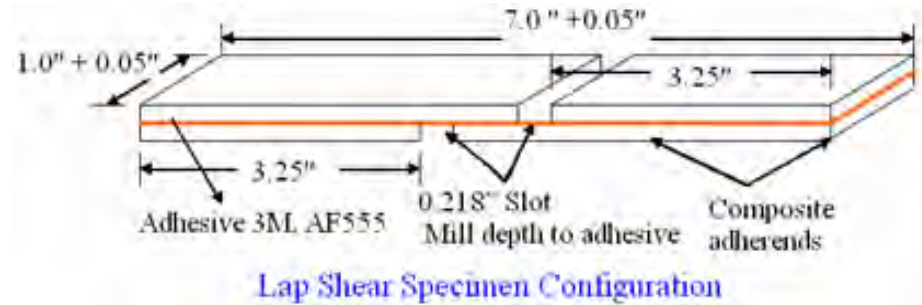
- Part I – Adherend moisture sensitivity
- Part II - Peel ply
- Part III – Abrasive techniques

- Material Specifications

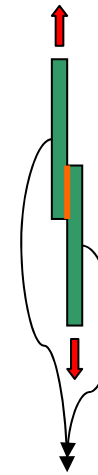
- 20 unidirectional plies
- Polyester peel ply (precision fabrics 60001)
- Adhesive, 3M AF555

- Specimen type

- Dry co-bonded (DCoB) adherends
- Wet co-bonded (WCoB) adherends
  - Pre-cured adherends soaked to 1% moisture content prior to bonding uncured skin

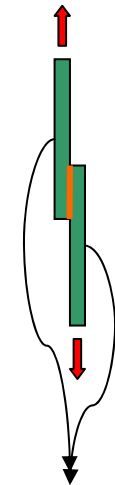


DCoB



Dry

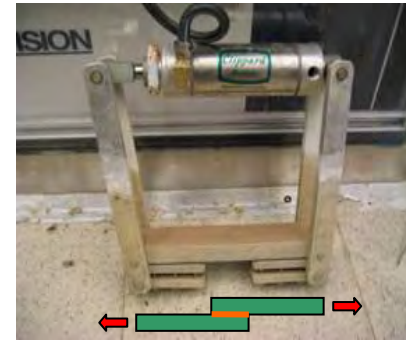
WCoB



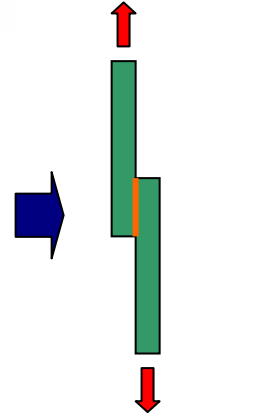
Wet, 1% MC



- Lap Shear Tests
  - Ultimate lap shear strength ( $L_{ST}$ )
  - Failure modes of the bonds
- Conditioning and loads
  - Water immersion, 140 ° F, 1k hrs
  - Load of 0, 2, 3 and 4 ksi



Creep test



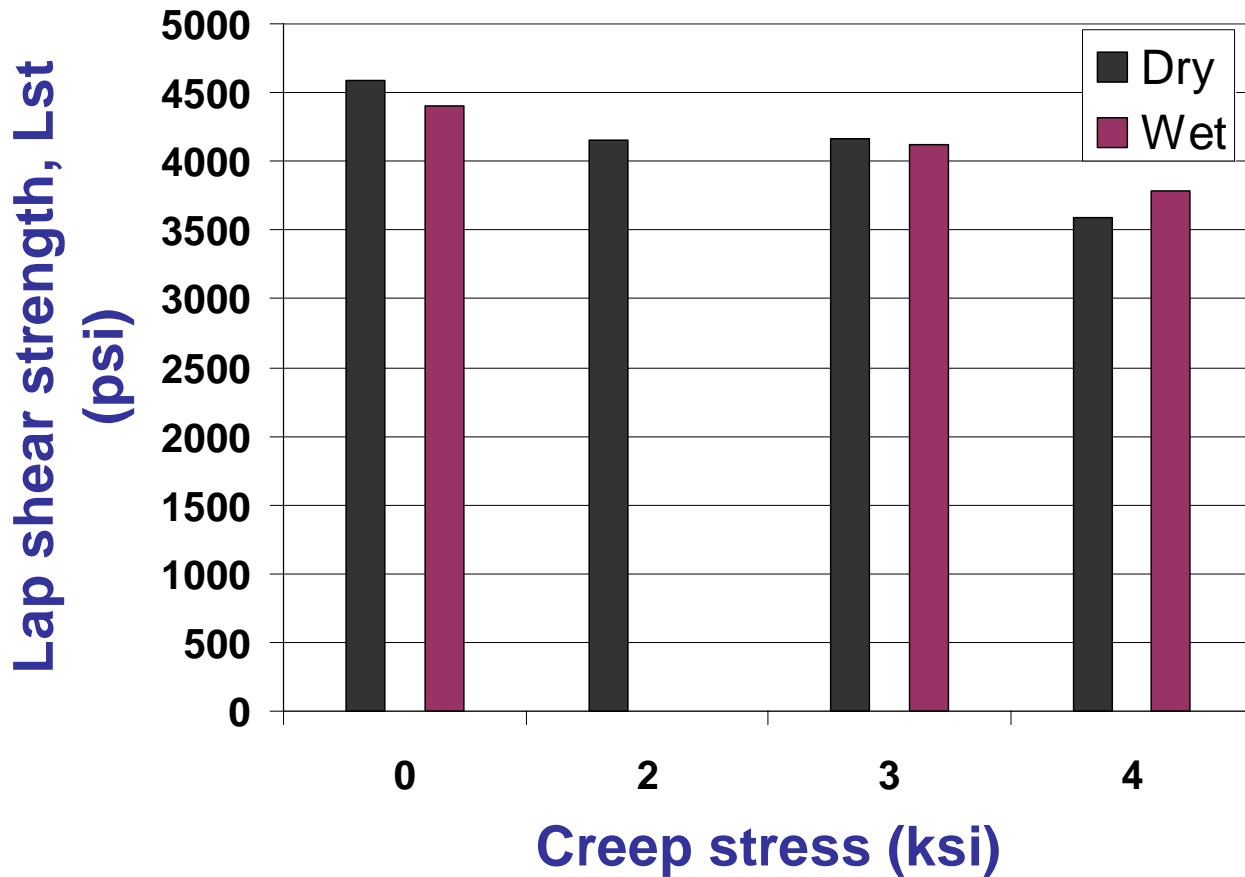
Lap Shear test

- Test matrix

Process	0 ksi	Creep Stress		
		2 ksi	3 ksi	4 ksi
Dry (DCoB)	3 (avg. 4.6 ksi)	3	3	3
Wet (WCoB)	3 (avg. 4.4 ksi)	-	3	3



# Part I-Moisture Sensitivity Results



- Failure mode:

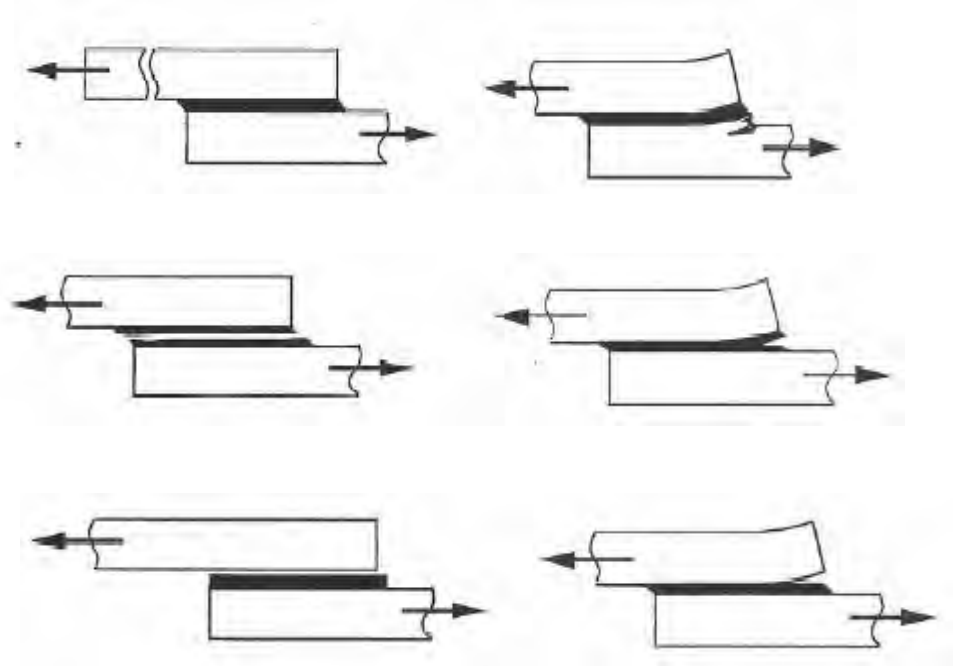
- Adherend

- Cohesive

- Adhesion

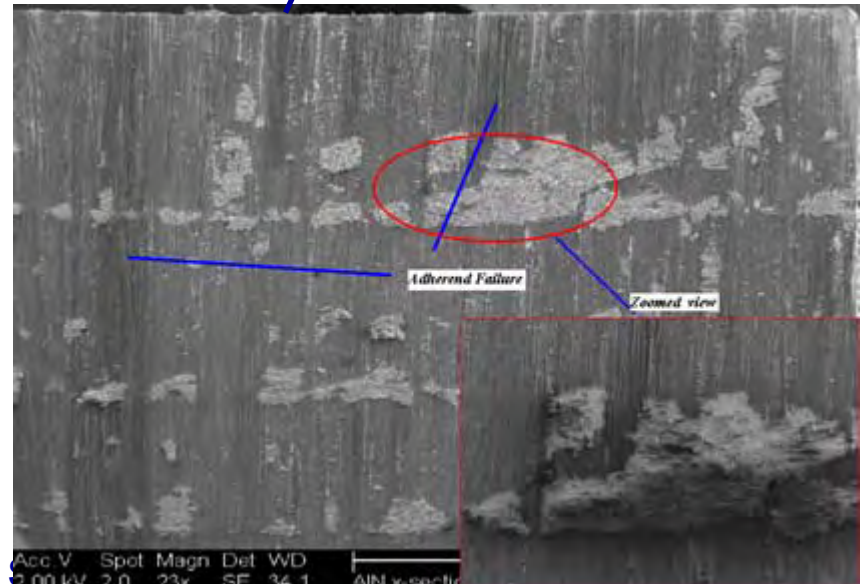
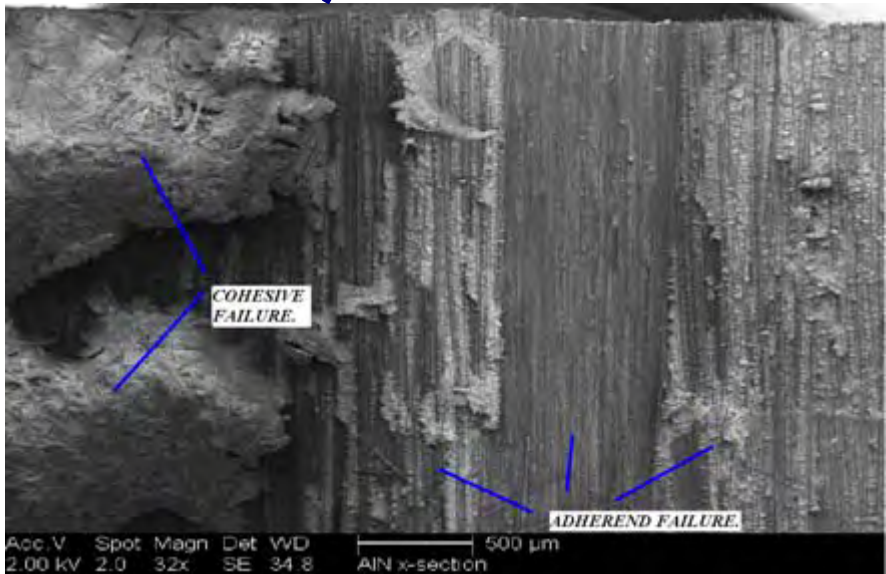
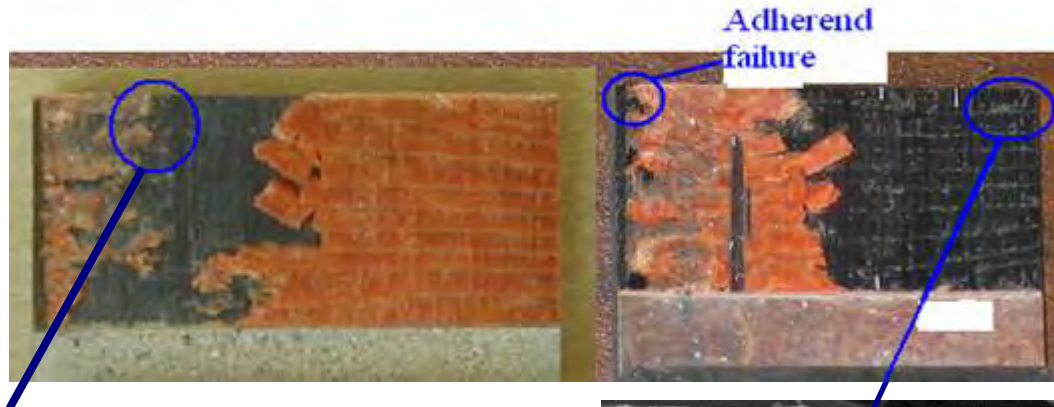
## Shear

## Peel



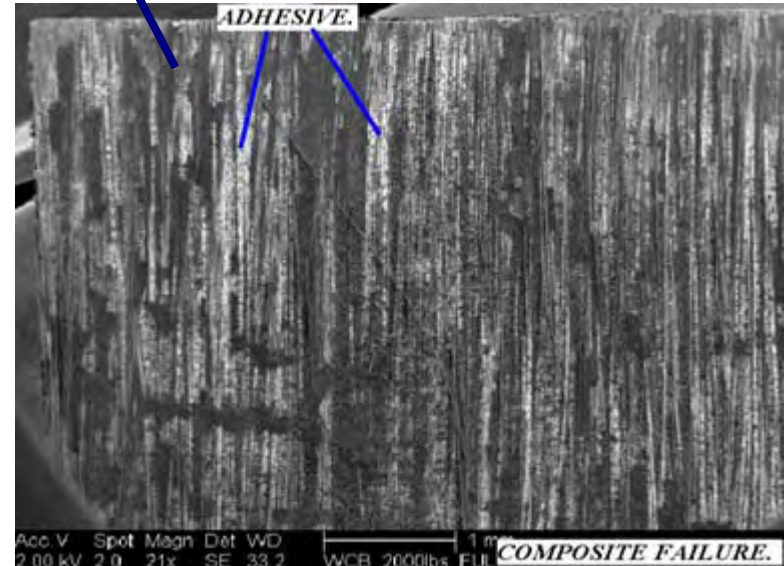
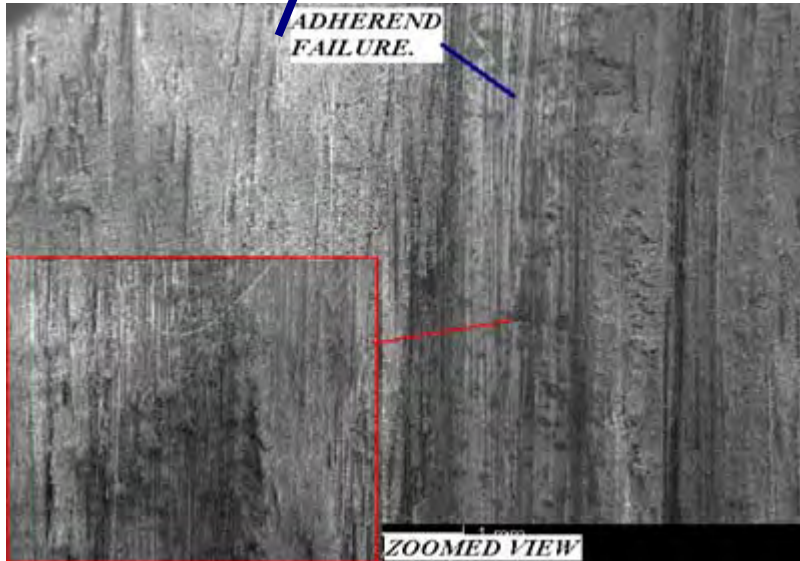
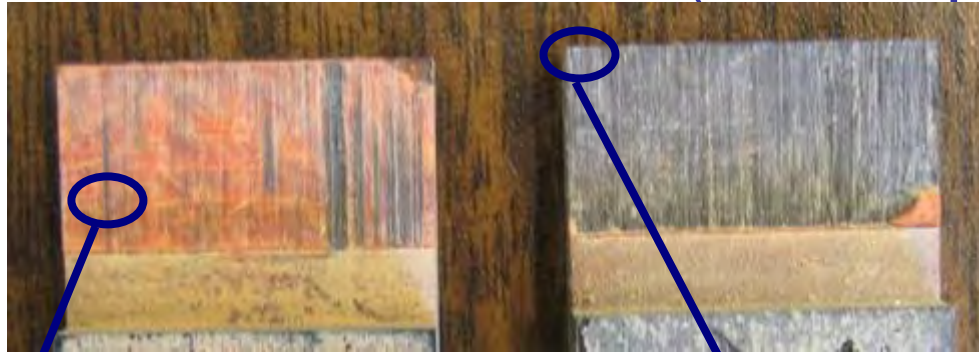
# Part I- Moisture Sensitivity Results – failure modes

- Adherend Moisture Effects (0 ksi, dry)



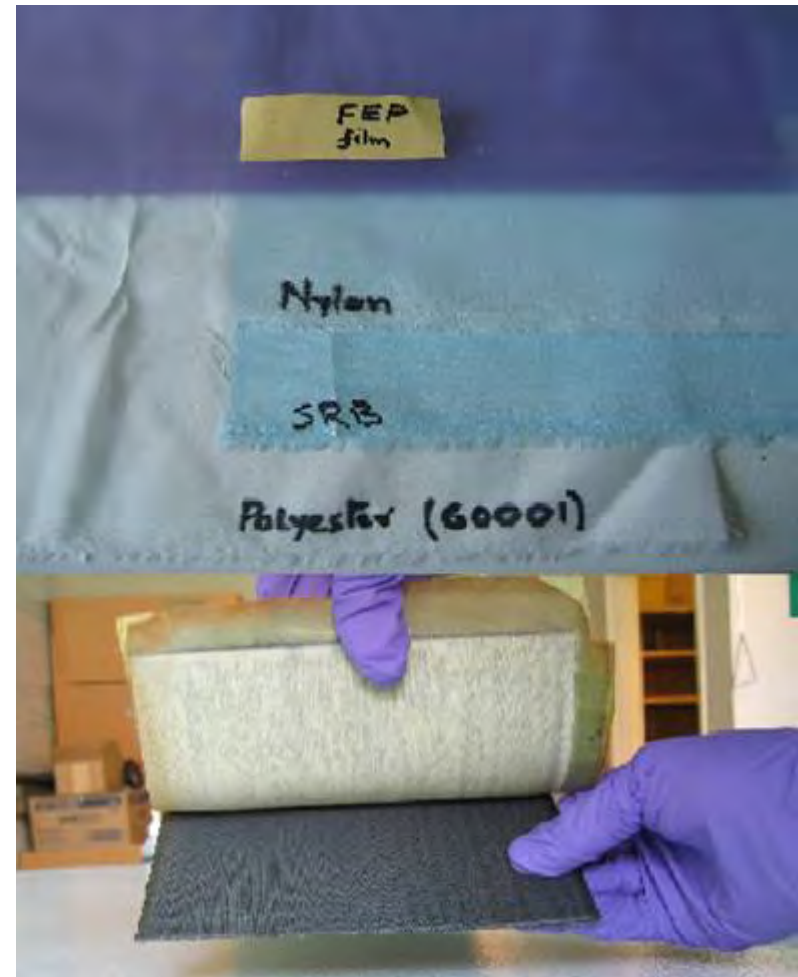
# Part I – Moisture Sensitivity Results - failure modes

- Adherend Moisture Effects (4 ksi rupture, wet)



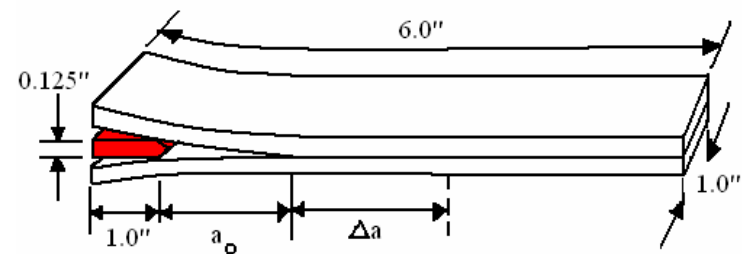
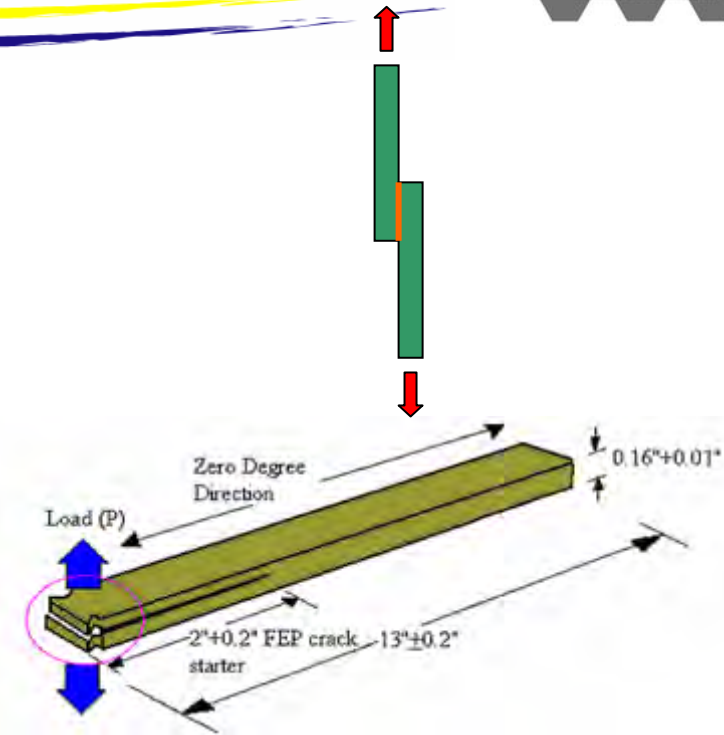


- Peel ply
  - Polyester (precision fabrics 60001)- **fine**
  - Nylon (precision fabrics 52006)- **medium**
  - Siloxane coated polyester (super release blue, SRB)- **coarse**





- Tests
  - Lap shear test (20 plies)
    - Saturate in 140°F water (6k hrs)
    - 80% UTS creep
    - Compare creep rupture duration
  - Double cantilever beam (DCB) test (10 plies)
    - Saturate in 140°F water (6k hrs)
    - Compare strain energy release rates
  - Wedge crack (WC) test (10 plies)
    - Insert wedge
    - Immerse in 140°F water
    - Compare crack growth



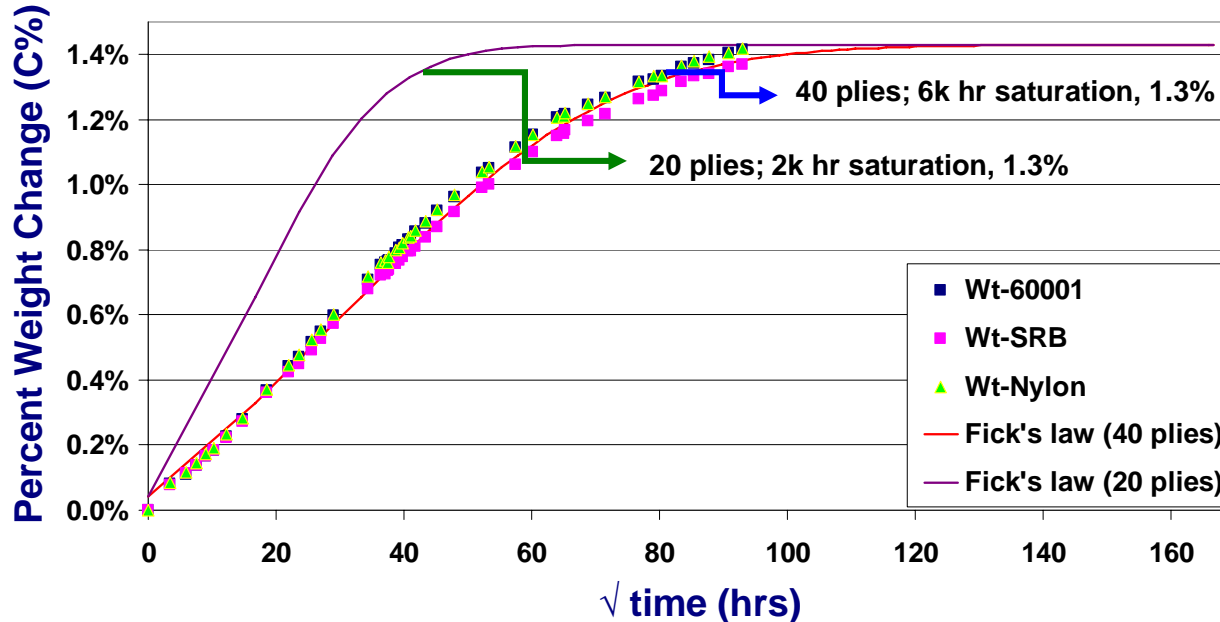
# Part II – Peel ply

## Test Matrix:

<u>Coupon/peel ply</u>	<u>6k hr</u>	<u>6k hr</u>	<u>10k hr</u>
WLS/60001	5	10 (80% UTS)	-
WLS/Nylon	5	10 (80% UTS)	-
WLS/SRB	5	10 (80% UTS)	-
DCB/60001	5	-	5
DCB/Nylon	5	-	5
DCB/SRB	5	-	5

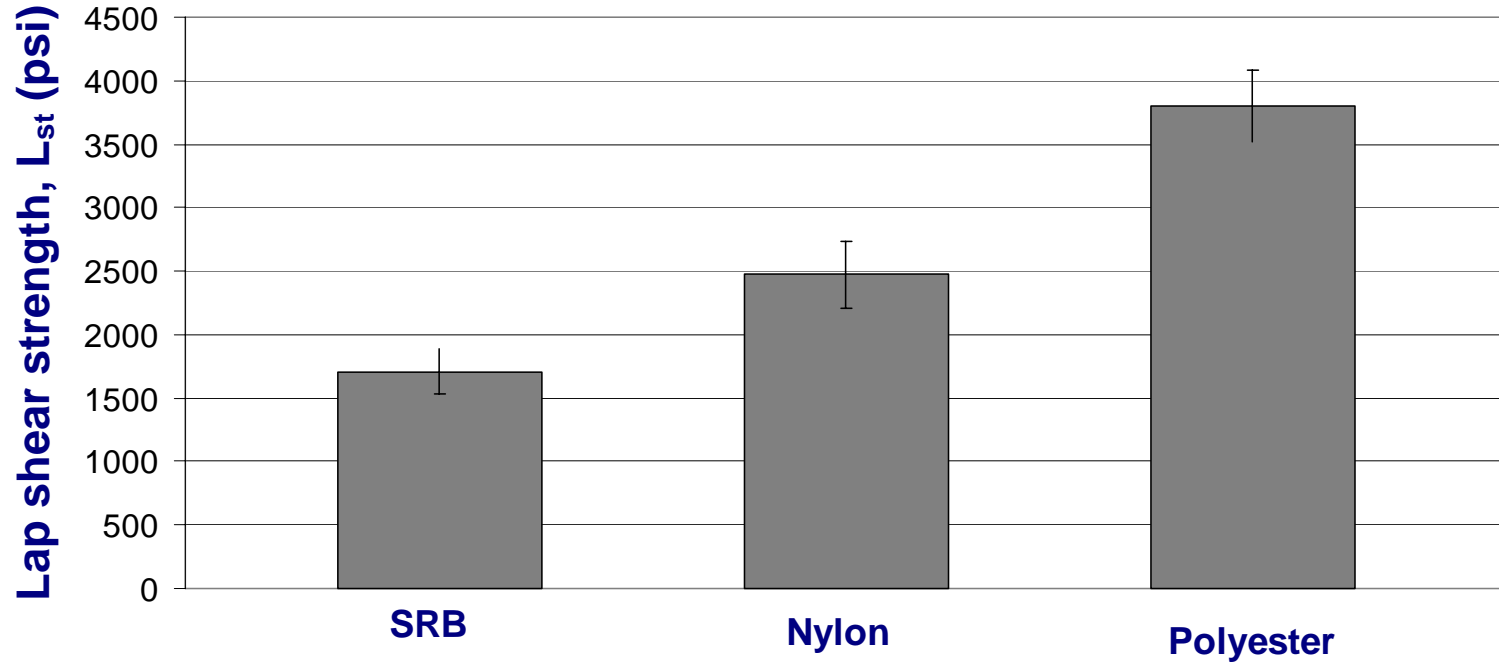
<u>Coupon/peel ply</u>	<u>0 hr</u>
WC/ SRB	5
WC/Nylon	5
WC/60001	5





- $D = 6 \times 10^{-7} \text{mm}^2/\text{s}$  , at  $140^\circ\text{F}$
- Saturation after 100 hr immersion is often assumed

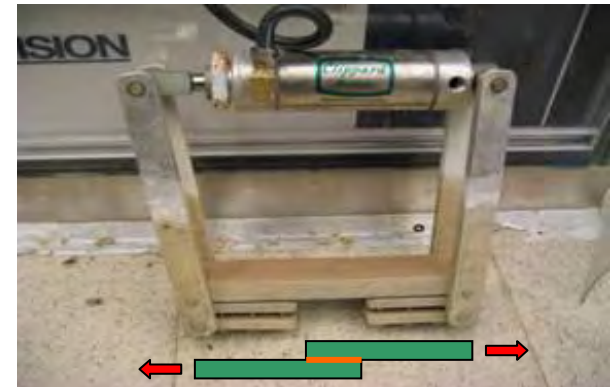
# a) Lap shear test – Baseline Results



# Part II – Peel ply

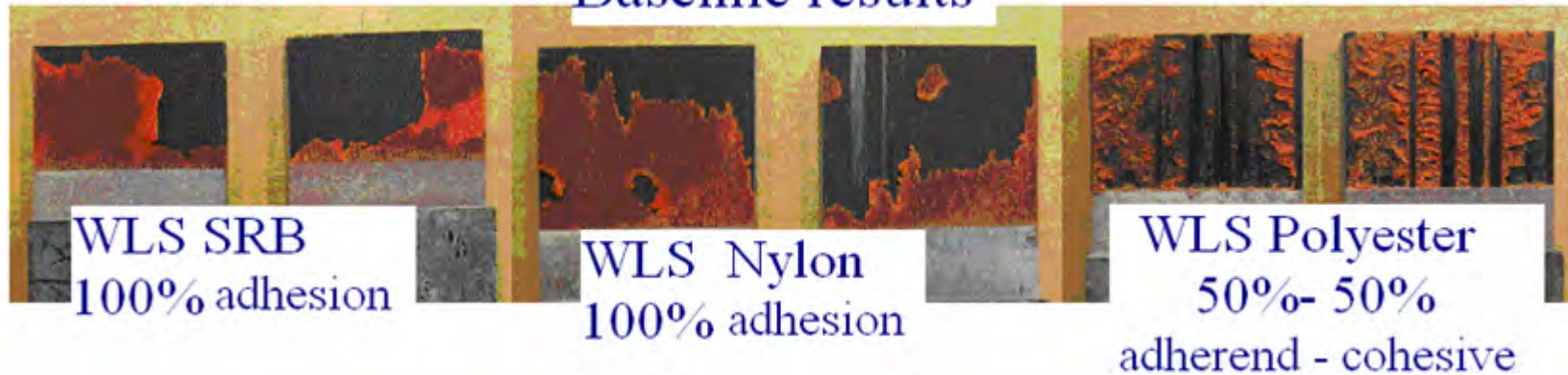
## a) Lap shear test – Creep test

- Creep load
  - 80% of  $P_{max}$  SRB = 700 lbs
  - Nylon = 1000 lbs
  - Polyester = 1700 lbs

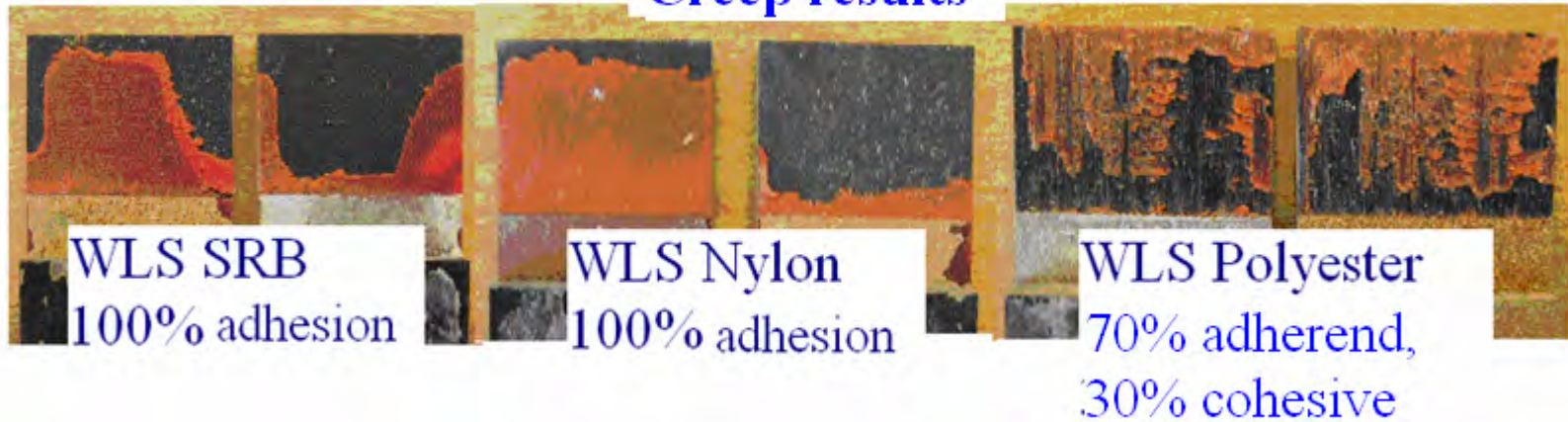


a) Lap shear test – Failure modes

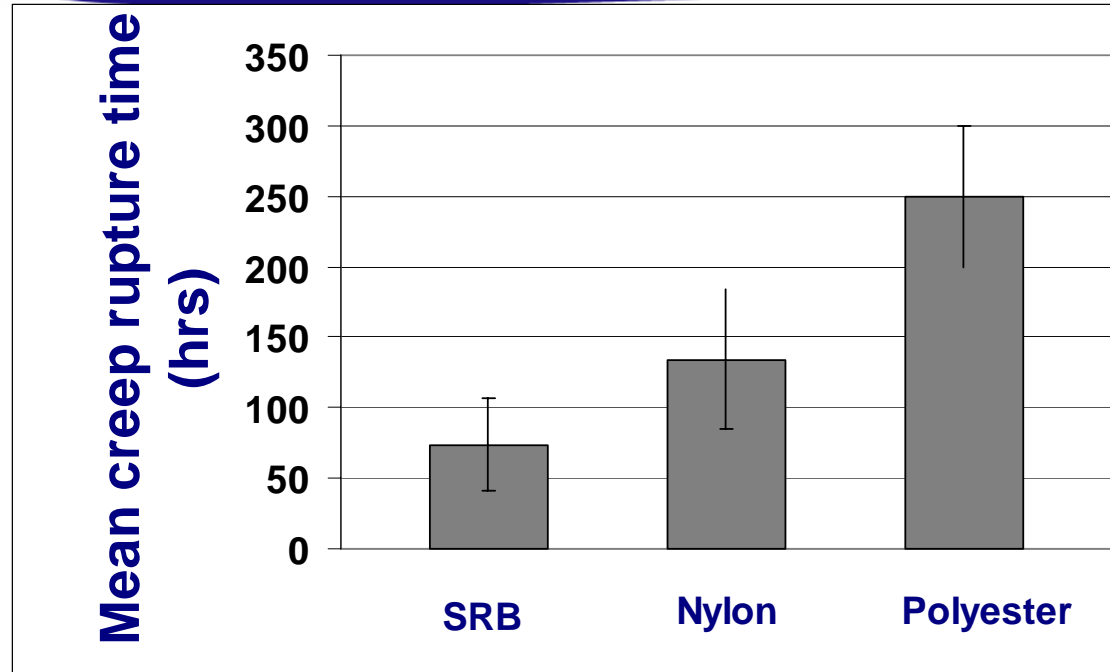
Baseline results



Creep results



## a) Lap shear test – Creep Results



- Creep rupture duration correlated with bond quality
- Large variation typical of creep rupture
- Moisture enhanced substrate failure



# Part II

## Work in progress

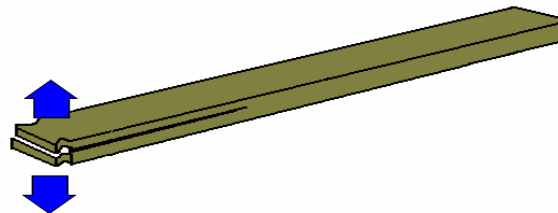
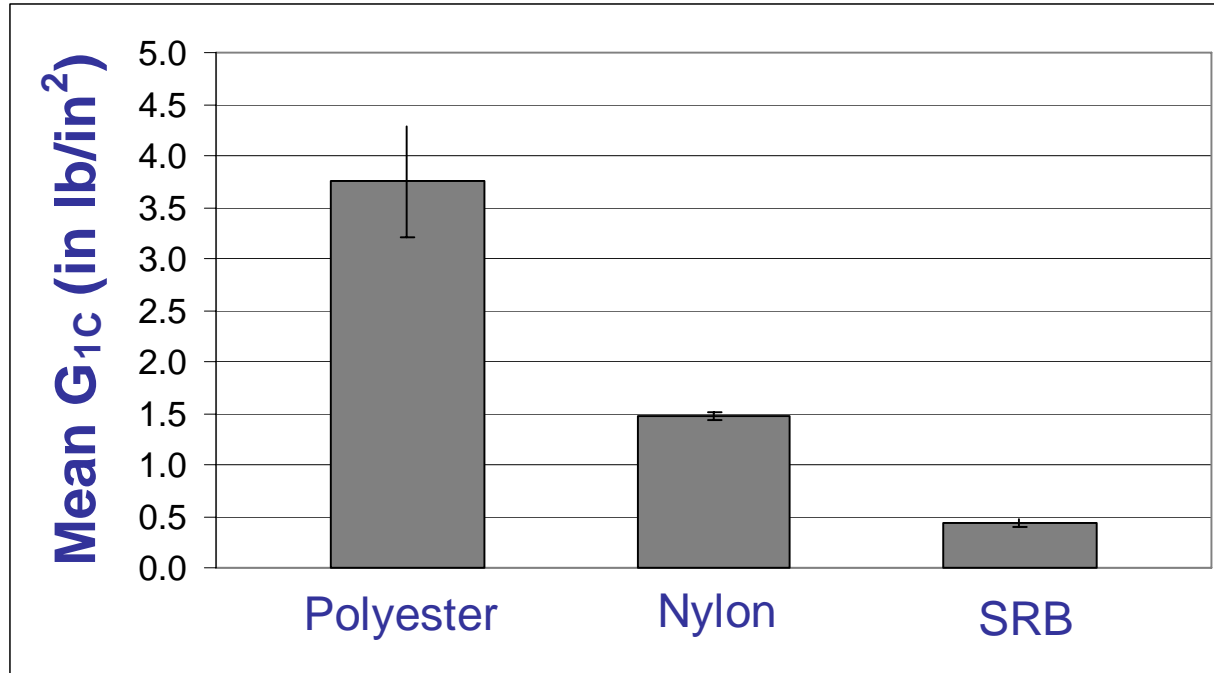
- Study the effect of moisture on the composite substrate integrity
- Immersion in water at 160°F from 2 to ~13 weeks (1.2%)
- Compression interlaminar shear (CILS) test
- Shear modulus test



Compression interlaminar shear coupon



## b) DCB baseline test results





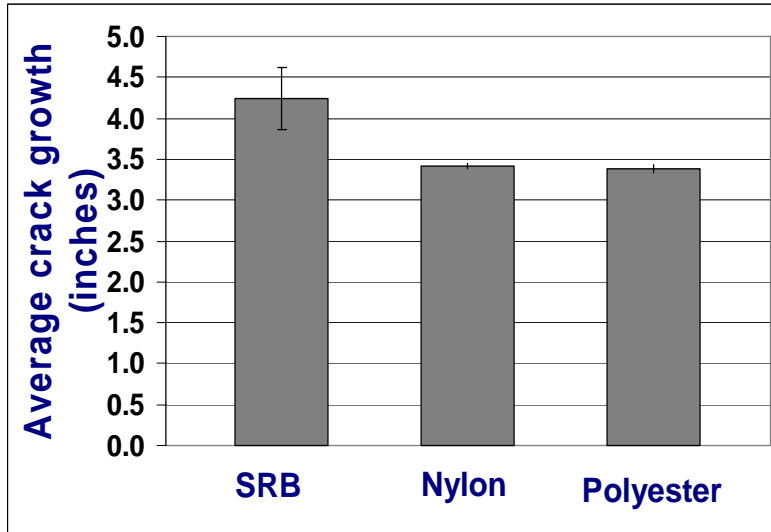
b) DCB baseline results- failure modes



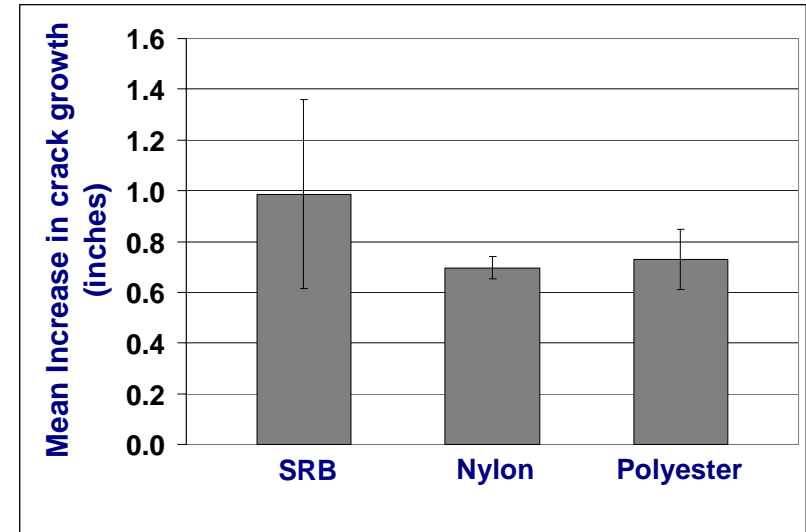
<p>DCB SRB                  100% adhesion</p>	<p>DCB Nylon                  90% adhesion, 10%                  adherend</p>	<p>DCB Polyester                  100% adherend</p>
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# Part II – Peel ply

## c) Wedge Crack Test- Results



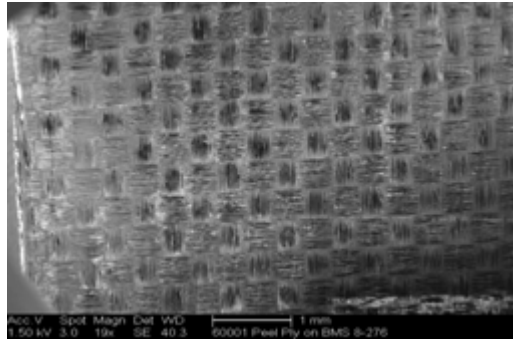
After inserting the wedge



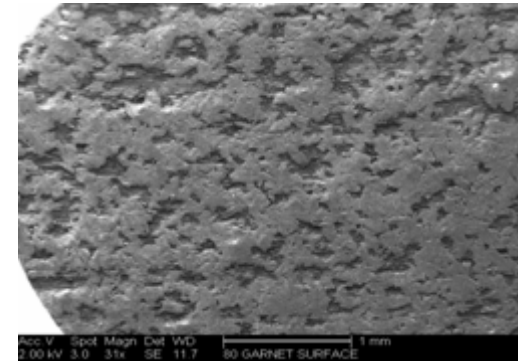
After 24 hrs water immersion

- 6 inch SRB coupons failed at 750 hrs
- 12 inch coupons showed small increase in crack growth

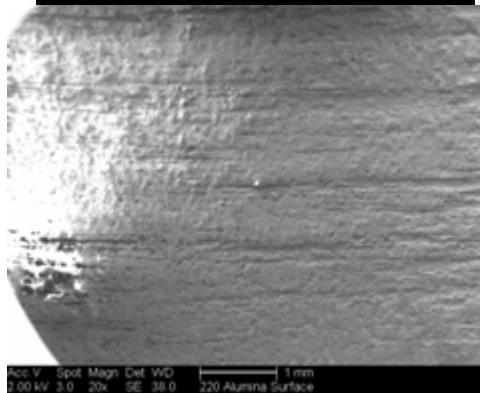
- Adherend surface preparations



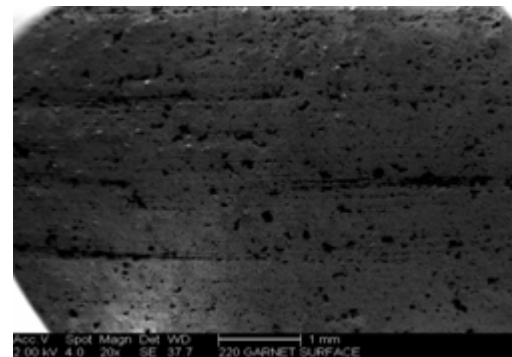
Polyester peel ply



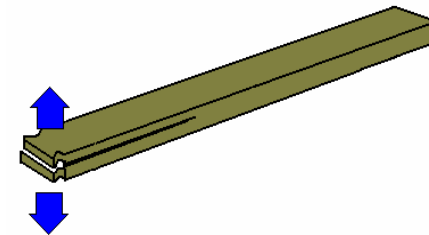
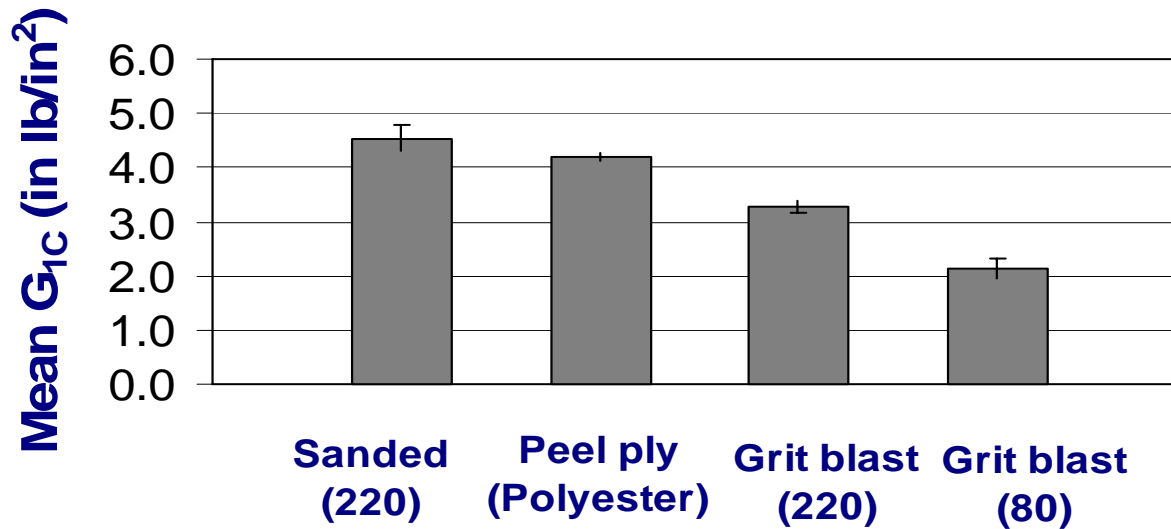
Grit blast 80



Sanding 220

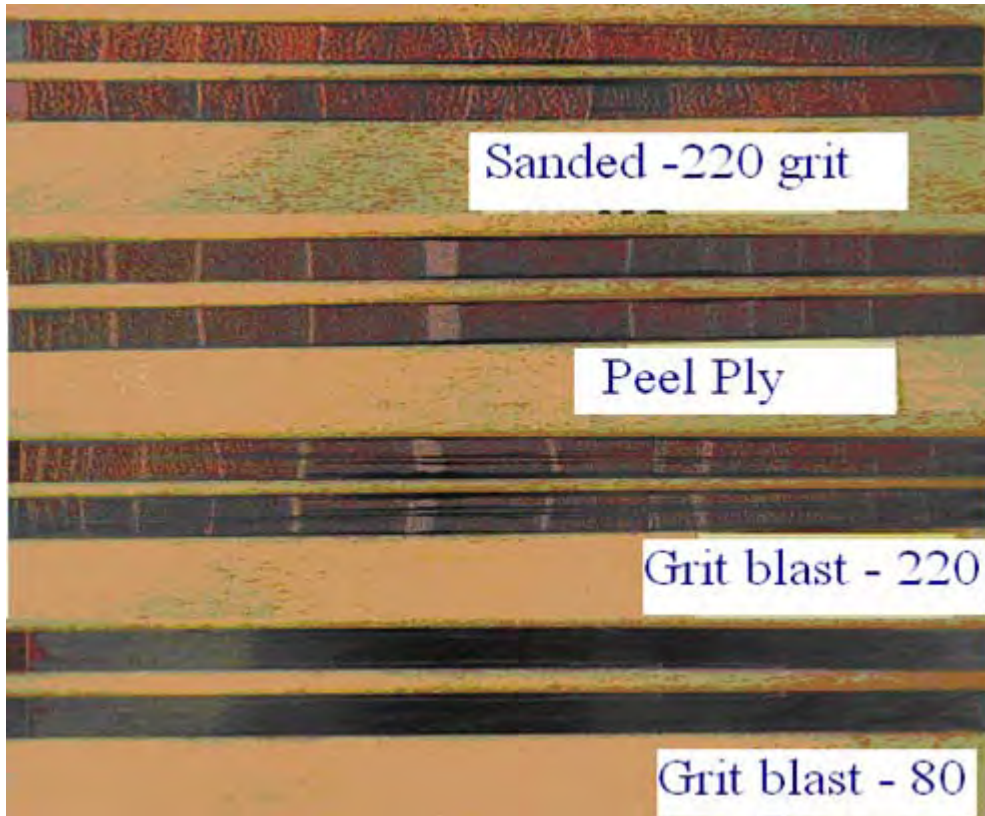


Grit blast 220



- Double cantilever beam test
- Lower  $G_{1c}$  of grit blasted surfaces, may be due to erosion (fiber damage)
- SEM results reveal harsher effects on grit blast surfaces

## Failure modes



Sanded → 99% cohesive,  
1 % adherend

Peel ply → 90% cohesive,  
10% adherend

Grit blast 220 → 70% cohesive,  
30% adherend

Grit blast 80 → 100% adherend



- Work in progress
  - Effect of creep and cyclic load on DCB coupons with sanded and grit blasted surfaces has begun



DCB creep and repeated load setup

Specimen Type	140 F water immersion			-65 F in air		No exposure	Total coupons
	Constant load	No load	Fluctuating load	Constant load	No load		
Peel ply (9.8lbs)	10	5	10	10	5	5	45
Sanded 220 (10.3lbs)	10	5	10	10	5	5	45
Grit blast 220 (8.4 lbs)	10	5	10	10	5	5	45
Grit blast 80 (6.2 lbs)	10	5	10	10	5	5	45

1. Coupons dried in oven at 160F
2. Applied load = 90%  $G_{1C}$
3. Crack growth measured daily for 100 hours, weekly for up to 4000 hours
4.  $G_{1C}$  may be measured at the conclusion of the test.



- Repeated load movie



- **Part I : Moisture sensitivity**
  - Residual shear strength decreased with increasing creep load.
  - 3M AF555 showed little sensitivity to adherend moisture content
  - Predominantly adherend failure
- **Part II : Peel ply**
  - **a) lap shear test**
    - Polyester
      - Highest strength
      - Adherend and cohesive failure
    - SRB and nylon
      - Lower strength
      - Adhesion failure (also observed by Flinn et al.)
    - Moisture increased substrate failure
      - Motivated further study involving CILS and shear modulus coupons

- b) DCB test
  - Polyester : Higher  $G_{1C}$   
: Adherend failure
  - SRB and nylon : Lower  $G_{1C}$   
: Adhesion failure
  
- c) Wedge crack
  - SRB : High initial crack growth  
: Comparable crack growth under exposure  
: Does not clearly describe observed lower durability
  
- Part III : Abrasive techniques
  - Grit blast : Lower  $G_{1C}$   
: Adherend failure
  - Sanded, peel ply : Higher  $G_{1C}$   
: Cohesive failure

- Benefit to Aviation
  - Better understanding of moisture, peel ply, abrasive technique effects on the bond integrity.
  - Greater confidence in adhesive bonds
  - Guide development of QA methods for surface prep.
- Future needs
  - Moisture effects on the composite substrate integrity
  - Application to other composite systems and adhesives
  - Durability of differing joint designs