Improving Adhesive Bonding of Composites Through Surface Characterization

(of Peel Ply Prepared Surfaces)

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The Joint Advanced Materials and Structures Center of Excellence

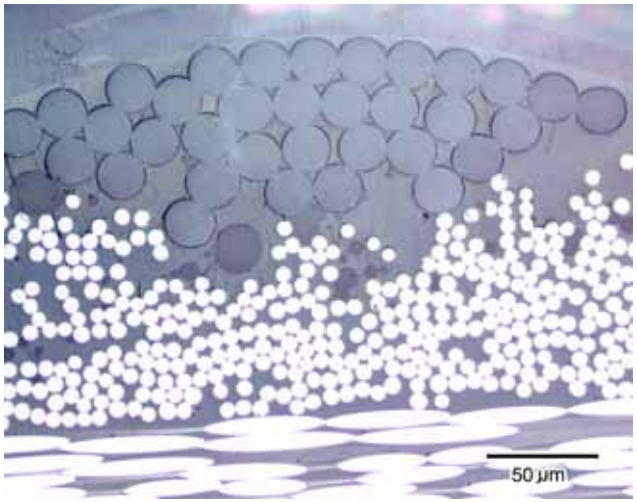
Improving Adhesive Bonding of Composites Through Surface Characterization

- Motivation and Key Issues
  - Peel ply surface preparation is being used for bonding primary structure on Boeing 777 and 787 and other commercial transport aircraft
  - Good bonds are produced but questions remain:
    - How can suitability of a surface for bonding be determined
    - Does contact angle (wettability) correlate with bonding
    - What is the effect of peel ply texture on surface and bonding
    - What is the effect of moisture in peel ply before cure
- Objective
  - Develop further understanding of the effect surface preparation has on the durability of primary structural composite bonds through surface analysis coupled with mechanical testing and fractography

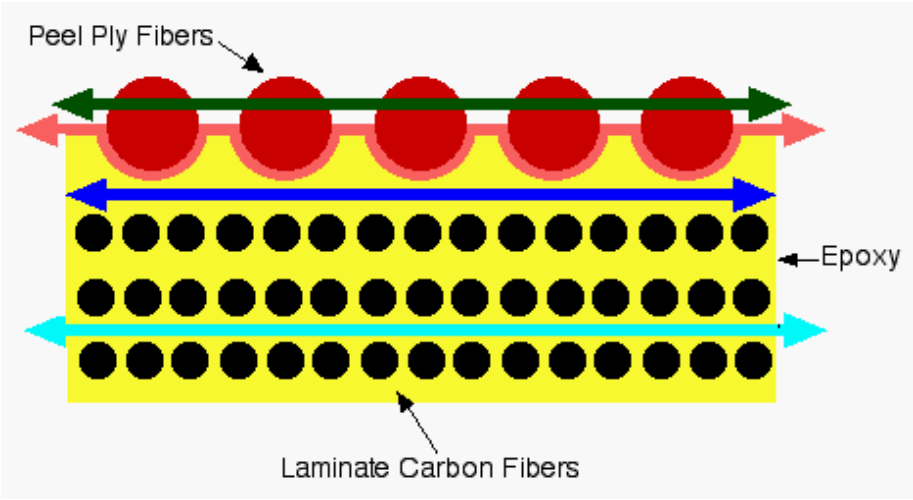
Improving Adhesive Bonding of Composites Through Surface Characterization

- Approach
  - Prepreg BMS8-276 form 3 (Toray)
  - Peel/Release Plies
    - Materials: polyester, nylon and SRB release (siloxane finish)
    - Texture: Fine, medium and coarse weaves
    - Moisture Content: dry to saturated
  - Adhesive Types
    - Cytec MB1515-3 and 3M AF555
  - Characterization
    - SEM
    - Surface Chemistry (ESCA/XPS, SIMS)
    - Profilometry
    - G_{IC} DCB fracture testing (ASTM D-5528)

Peel Ply Surface Preparation



Fracture Possibilities Upon Peel Ply Removal



- Fracture of the epoxy between peel ply and carbon fibers
  - Fresh, chemically active, epoxy surface is created
- Interfacial fracture between the peel ply fabric fibers and the epoxy matrix
- Peel ply fiber fracture
- Interlaminar failure

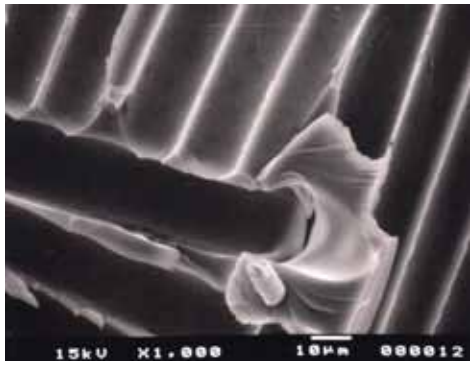
- **Fracture Mode controls surface characteristics and bond quality**

Task 1: Peel Ply Material Type

- Laminates produced with 3 peel/release plies
  - Polyester BMS 8-308 (Precision Fabrics 60001)
    - Currently used for primary structural bond prep.
  - Nylon scoured and heat set (Precision Fabrics 52006)
  - Super Release Blue (60001 with siloxane coating)
- Samples removed for surface characterization
  - SEM, XPS, Contact Angle (wettability), SIMS
- Laminates bonded and machined in to DCB specimens

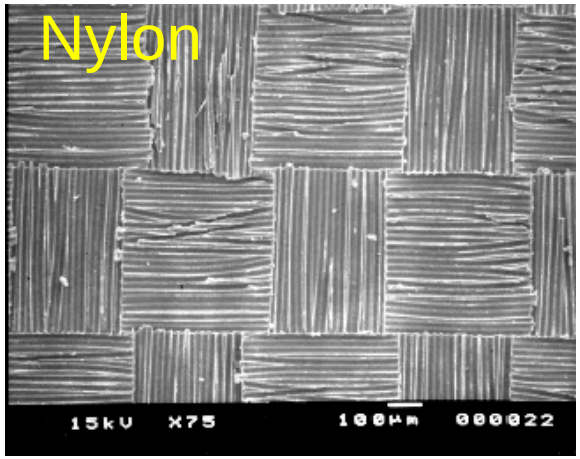
Peel Ply Surface Prep. - SEM Results

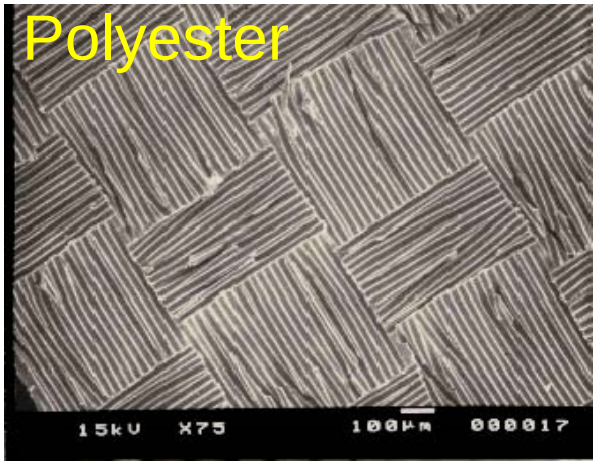
- All samples show acceptable surface on macro scale

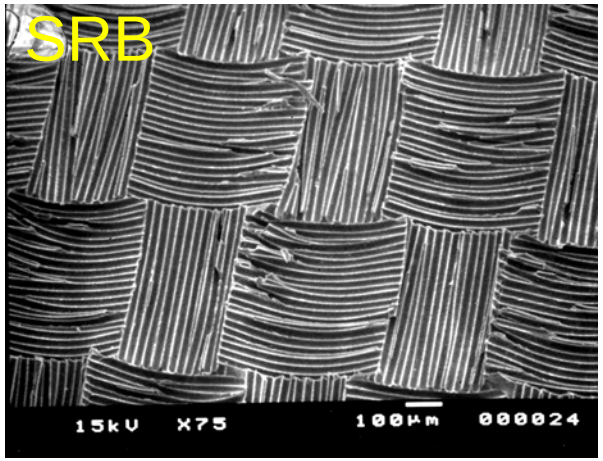


  - Interfacial fracture between the peel ply fabric fibers and the epoxy matrix
  - Limited epoxy fracture between peel ply fibers

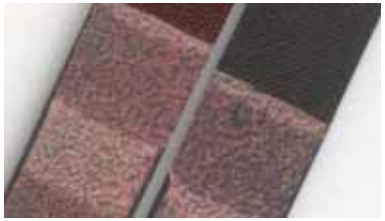
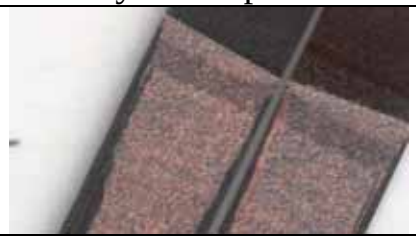
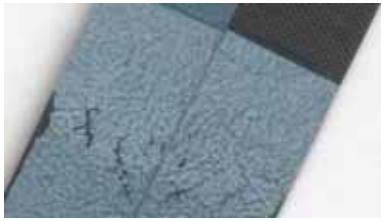
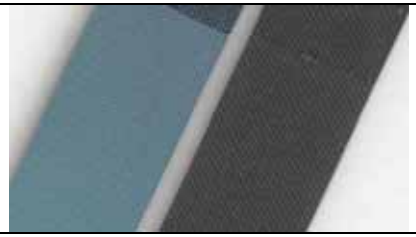
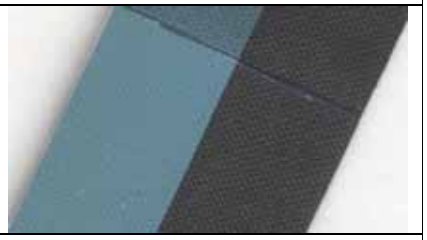
Composite surface after removal of:





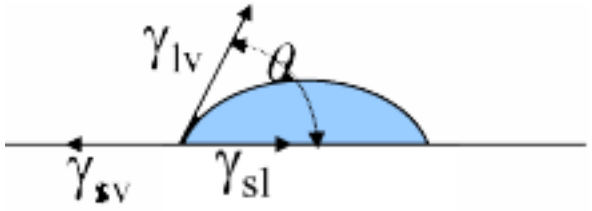


Task 1: Peel Ply Material Type

	Polyester Prepared	Nylon Prepared	SRB Prepared
Adhesive A			
Failure Mode	Cohesive	Cohesive & Interlaminar	Adhesion
G_{IC} (J/m^2)	909.6	910.7	93.9
Adhesive B			
Failure Mode	Cohesive	Adhesion	Adhesion
G_{IC} (J/m^2)	812.3	122.1	86.0

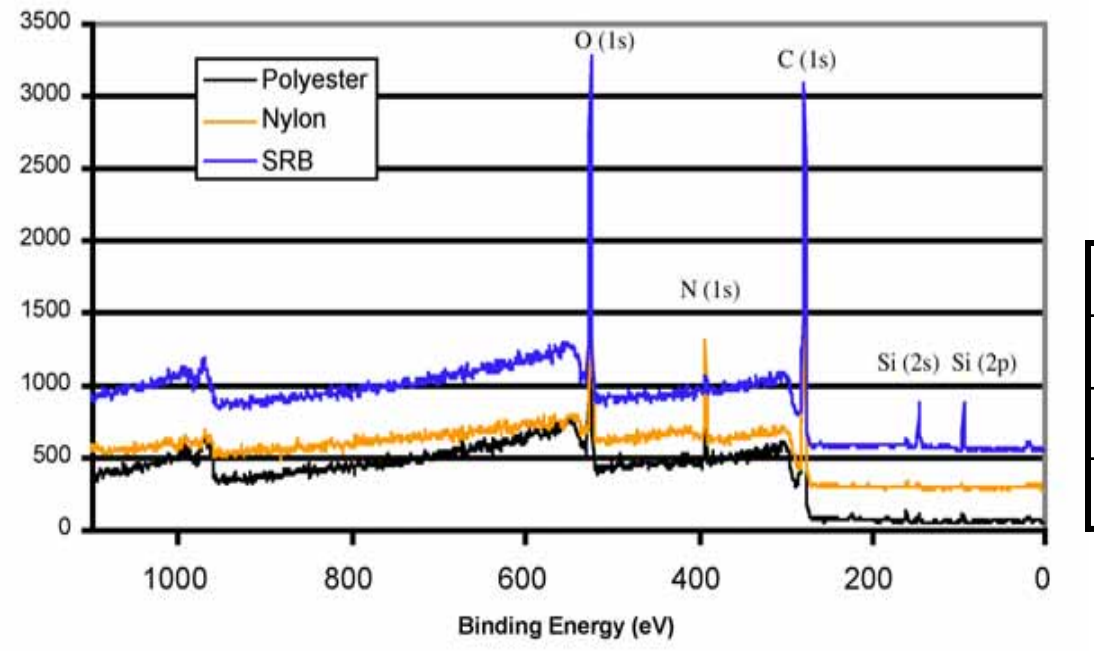
G_{IC} and Contact Angle do not always correlate

- G_{IC}: Polyester >>Nylon> SRB
- Contact Angle: Nylon < Polyester<< SRB



XPS Survey Scan Results

Laminate surfaces before bonding, after peel ply removal



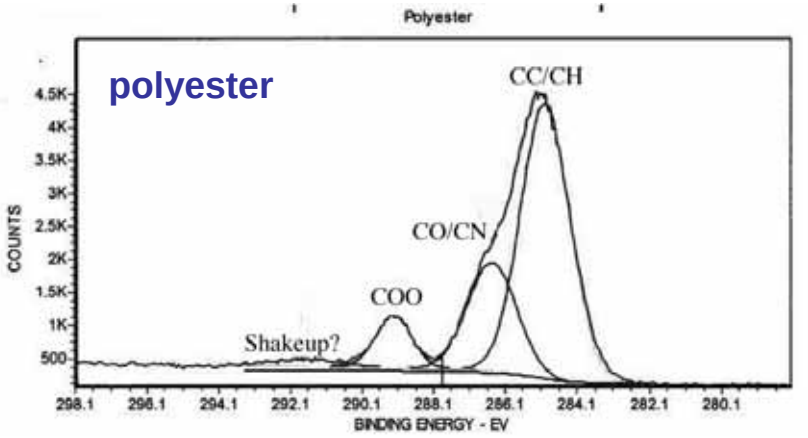
Laminate Surface Composition

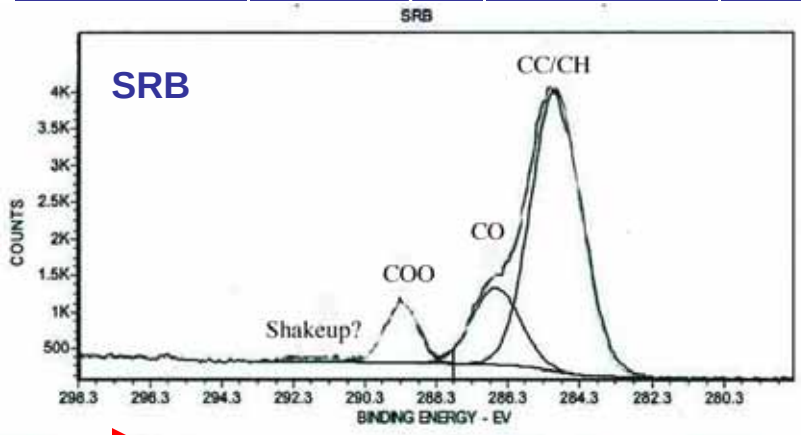
Peel Ply	%C	%O	%N	%Si
Nylon	77.5	12.6	9.8	Tr.
Polyester	75.5	21.6	1.9	1.0
SRB	68	24.2	0.9	6.9

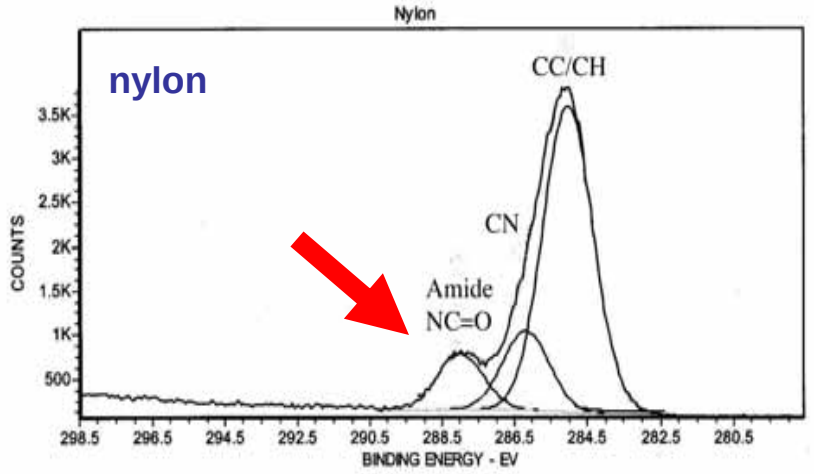
- Si explains SRB low bond quality….Siloxane coating transfers
- Amount of N on nylon peel ply prepared sample surprising

XPS High-Res Results

Peel Ply	Species	BE (eV)	%
Nylon	CC/CH	285	71
	CN	286.2	17.1
	Amide (NC=0)	288	11.9
Polyester	CC/CH	285	63.8
	CO/(CN)	286.5	24.9
	COO	289.2	8.8
	Shakeup?	291.8	2.4 (broad)
SRB	CC/CH	285	70
	CO	286.7	19.1
	COO	289.3	9.8
	Shakeup?	291.8	1.1(broad)



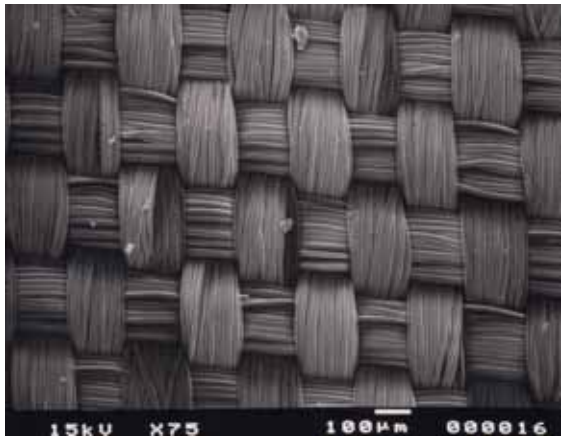




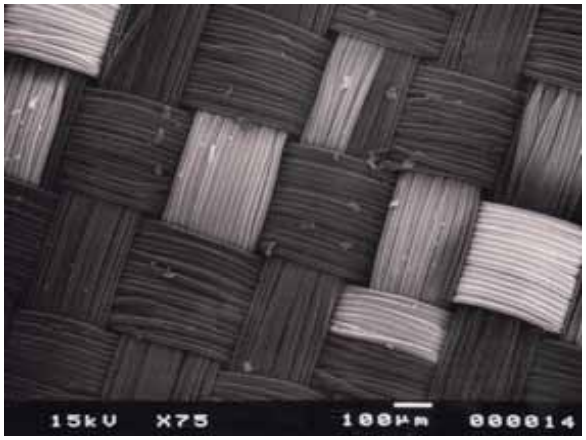
Amide detected on nylon prepared surface- nylon transfer to surface?

Task 2: Peel Ply Texture

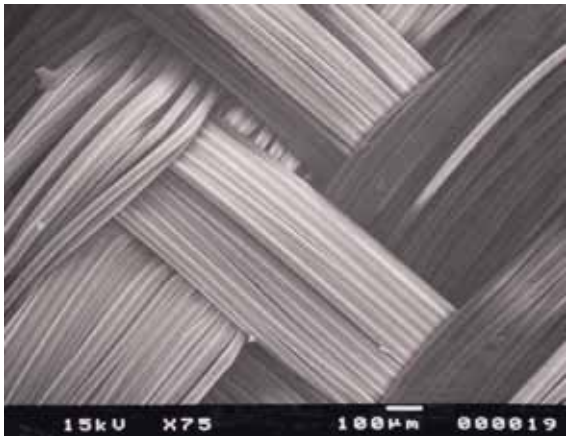
SEM's of As-Received Peel Plies



Fine 160 x103
(PF 52006)



Medium 101 x 82
(PF 52008)



Coarse 60 x 50
(PF 52000)

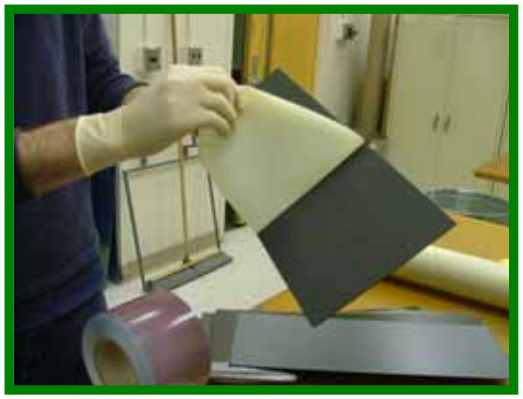
- Different weaves, deniers, filament diameters will produce different surfaces on laminate

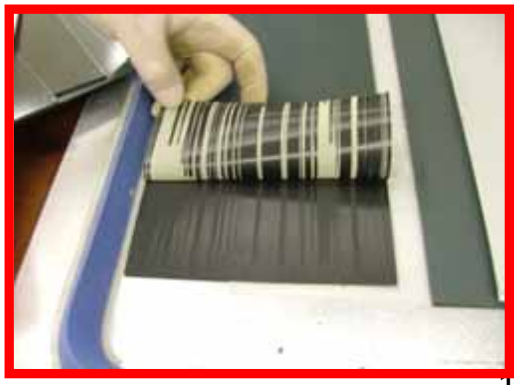
Task 2: Peel Ply Texture

- All polyester peel plies successfully removed
- Nylon peel plies were more difficult to remove
  - Fine weaves were removed without damage
  - Coarse weaves have not been removed without damage to laminate

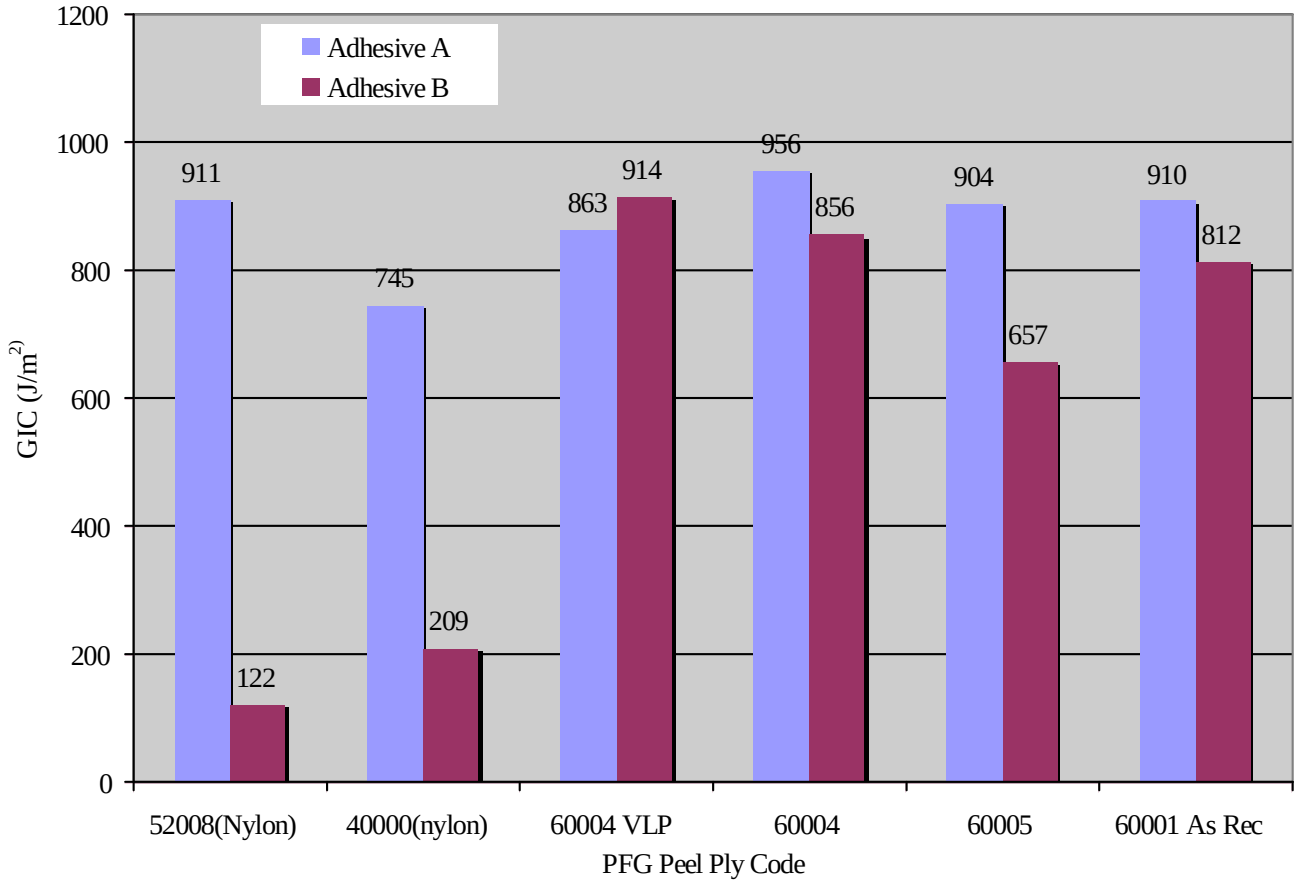
(3 attempts, different technicians)

Material	Precision Code	Warp (ends/in.)	Fill (picks/in.)
Polyester	60001	70	50
Polyester	60001 VLP	70	50
Polyester	60004	120	59
Polyester	60005	90	58
Nylon 6,6	52006	160	103
Nylon 6,6	52008	101	82
Nylon 6,6	50000	60	50
Nylon 6,6	40000	76	51
Nylon 6,6	41661	60	50





Task 2: Peel Ply Texture



- Peel ply texture does not seem to affect bond quality

Task 3: Peel Ply Moisture Content

- No specifications on moisture content of peel ply
- Saturation of polyester peel ply 60001
  - Dried peel ply
  - Soaked at 80°F/90% RH and 140°F/95% RH
  - Measured mass change at 0.5,1, 2, 4, 18 hrs
  - No measurable weight change at 80°F/90% RH
  - 25% weight gain at 140°F/95% RH after 0.5 hours
  - no change at longer times
  - Bonded with AF555
- Cohesive failure in all samples

No significant difference in surface chemistry or mechanical properties detected

Conclusions

- Polyester: No Material Transfer; Strong Bonds
- SRB: Siloxane Coating Transfers; Weak Bonds
- Nylon: Fiber May Transfer; bond depends on adhesive
  - Significant nitrogen, amide groups, detected
    - May have contributed to the poor bond quality
    - Further investigation needed
      - » Chemical or mechanical transfer?
- Contact angle did not correlate well with G_{IC}
  - Wetting is necessary….

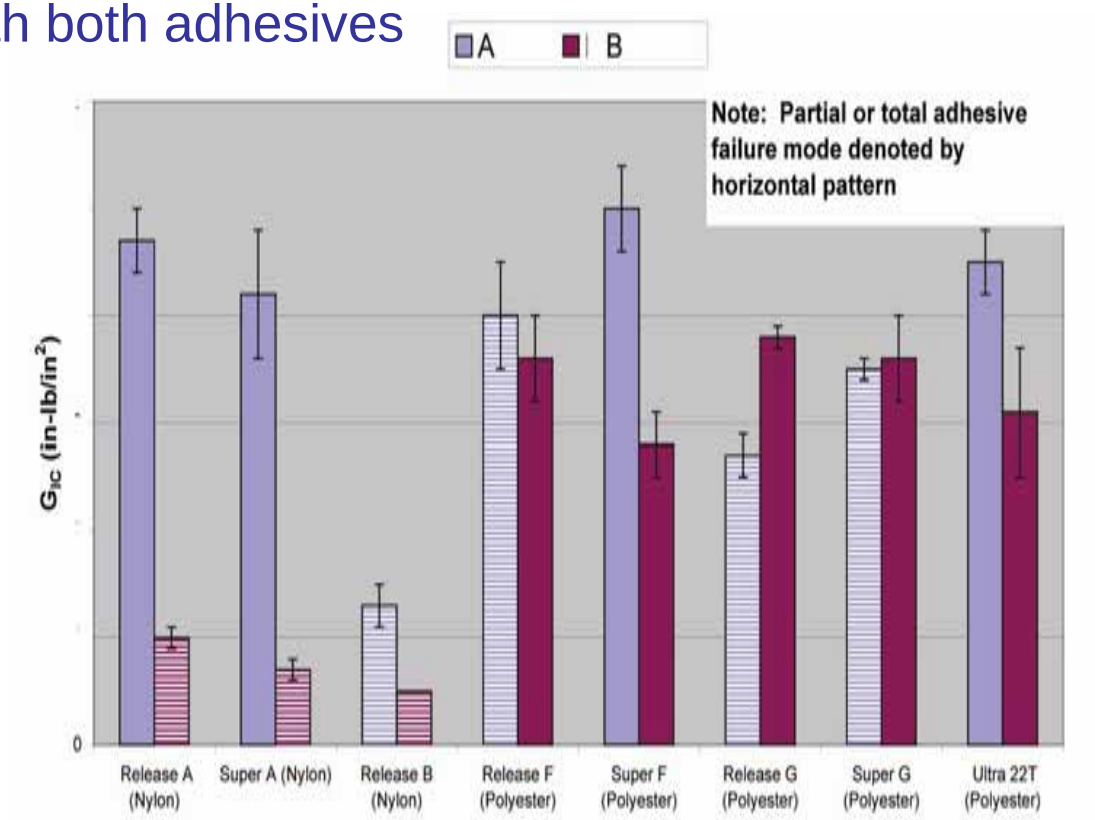
    ….but not always sufficient for good bond
- Peel Ply Texture – no detectable effect
- Peel Ply Moisture- no detectable effect

Year 2 Research Topics

- **Does the source of peel ply (different manufacturers) influence bond quality?**
- **Does bonding of laminate surfaces prepared with dry peel plies vs. wet peel plies differ?**
- **How does the degree of cure of laminates affect bond behavior?**

Peel Ply Material Source

- Previous work with Precision Fabrics polyester peel plies showed good bonding with both adhesives



- **Adhesion failure on some surfaces with polyester peel plies**

Peel Ply Material Source

- What is difference between polyester peel plies
  - Airtech vs. Precision Fabrics
- MB1515-3 vs. AF555 on polyester
  - AF555 did not bond well to 3 polyesters
  - MB1515-3 bonded well to all
- Surface Characterization
  - ESCA
  - Contact angle/wettability envelopes

"Wet" vs. Dry Peel Ply

- Investigate bonding of Wet vs. Dry Peel Plies
  - Henkel EA9895
    - Polyester fabric preimpregated w/ 350° F resin
    - Reported to be compatible with:
      - All prepregs tested to date
      - All adhesives tested to date
  - Boeing developing own wet peel ply
  - Use year 1 testing and characterization for wet peel ply
    - Compare with year 1 data on dry peel ply

Degree of Cure

Does the degree of cure of laminates affect bonding behavior

- Perhaps a partially cured laminate is a more active surface (cross linking not complete) to bond to…
- Prepare coupons at different cures (“green”)
- Characterize peel ply and composite surfaces
- Measure bond performance
- Fractography
- Do green laminates absorb more moisture?

Benefit to Aviation

- Better understanding of peel ply surface prep.
  - Composite system compatibility
    - Prepreg
    - Peel Ply
    - Adhesive
- Greater confidence in adhesive bonds
- Guide development of QA methods for surface prep.
  - Go/no-go testing

FAA Sponsored Project Information

- Principal Investigators & Researchers
  - Brian D. Flinn (PI)
  - Fumio Ohuchi (Co-PI)
  - Molly Phariss (Ph.D. Candidate, U. of Wa.)
  - Bjorn Ballien (Senior, U. of Wa.)
- FAA Technical Monitor
  - Peter Shyprykevich
- Other FAA Personnel Involved
  - Curt Davies, Larry Ilcewicz
- Industry Participation
  - Boeing: Peter Van Voast, William Grace, Paul Shelley
- JAMS Participation
  - Lloyd Smith (WaSU): Parallel study on durability
  - Bill Stevenson (WiSU) and Xiangyang (Joe) Zhou (FIU): samples