Evaluation of Peel-Ply Materials on Composite Bond Quality

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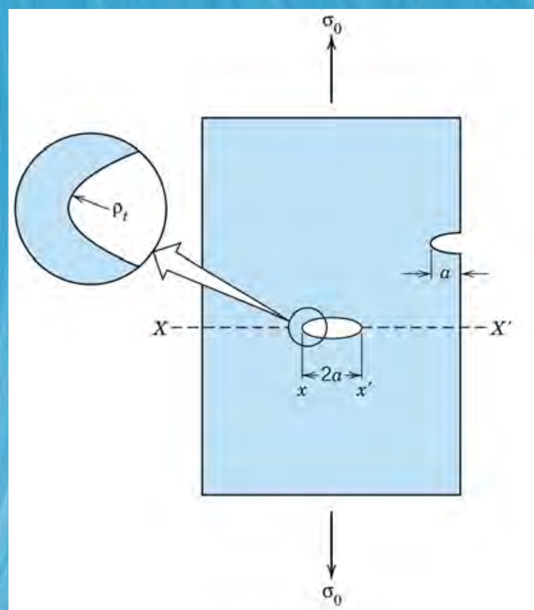
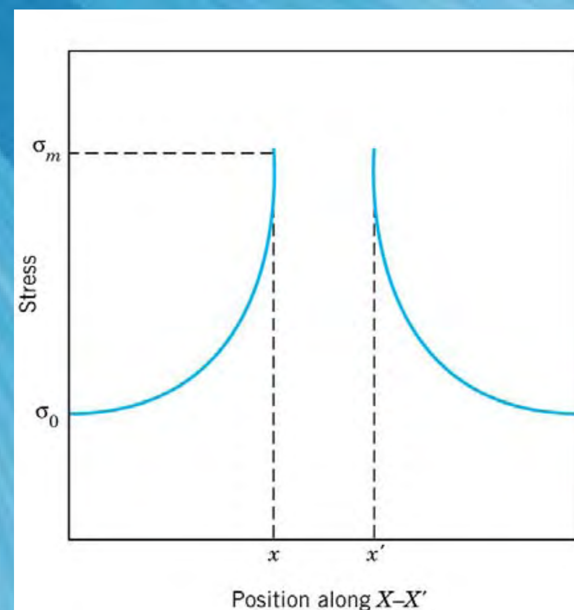
Joints are a great challenge in structure design.

- Mechanically fastened joints are interruptions part geometry



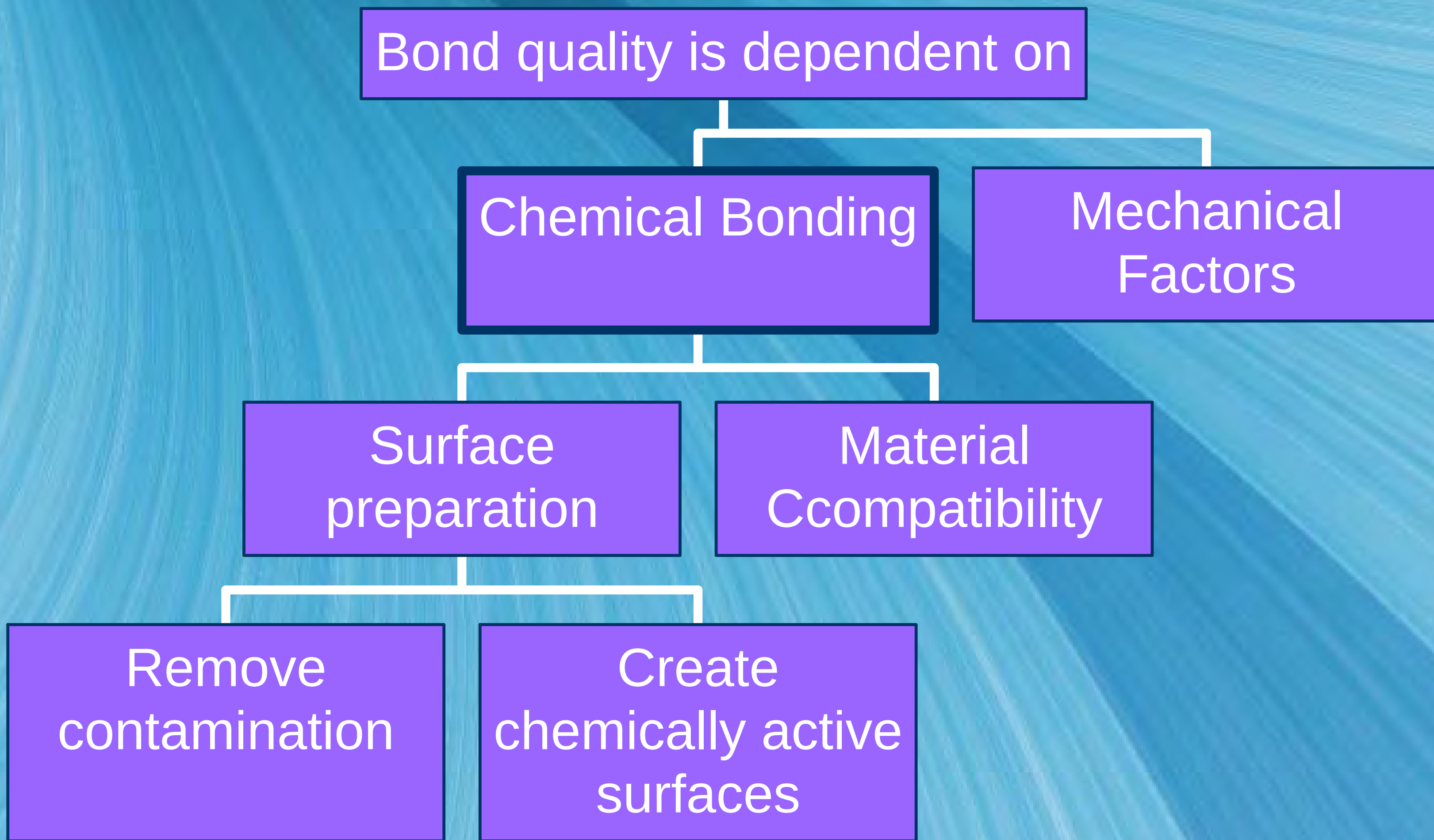


- Create material discontinuities
- Produce local highly stressed areas

Adhesive joints are more efficient.

- Minimize stress concentrations
- Structural weight saving
- Needed to take full advantage of composite properties

There are many concerns with bonded joints.



Bonded primary structure gives engineers ulcers.

- Lacks structural redundancy
- Lacks reliable inspection methods
  - X-Ray or ultrasonic reveal gaps
  - Can’t detect contamination, guarantee adequate load transfer

Techniques for producing strong composite bonds exist.

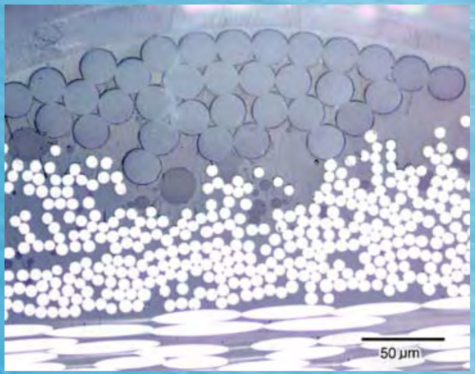
- Developed by trial-and-error
- Lack understanding of the role of surface preparation at atomic level

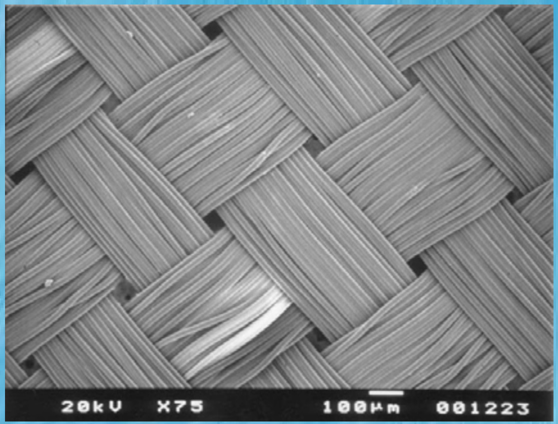
Objective

To further understand the effect of surface preparation on the durability of composite bonds through surface analysis coupled with mechanical testing and fractography

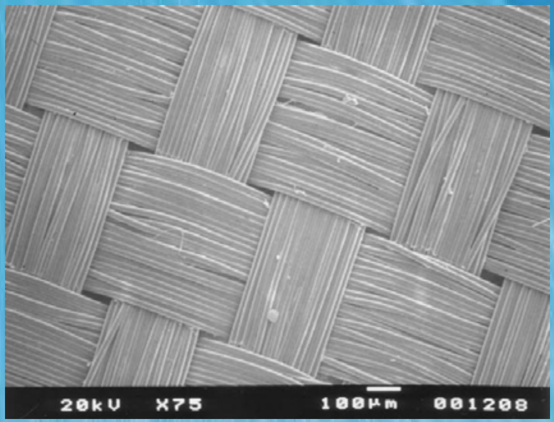
Peel ply preparation is used for primary structural composite bonds.

- Fabric is last layer on part before curing
- Removed before bonding
- Leaves clean, chemically active surface

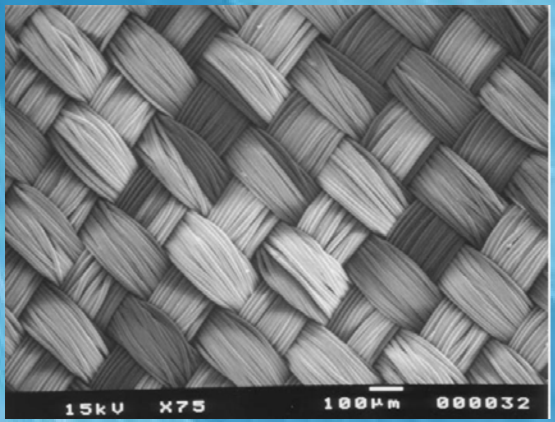




Precision Fabrics 60001
Polyester

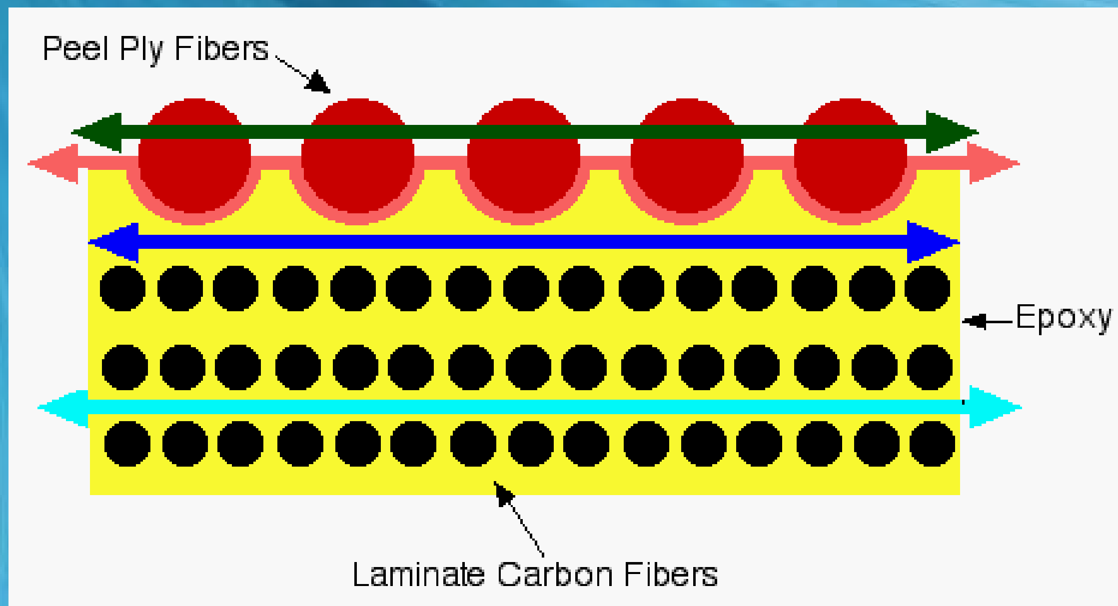


PF 52006
Nylon

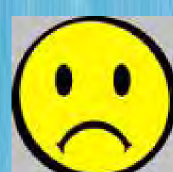


PF 60001 SRB
Siloxane Coated Polyester

Peel ply release mode controls surface and therefore bond quality.



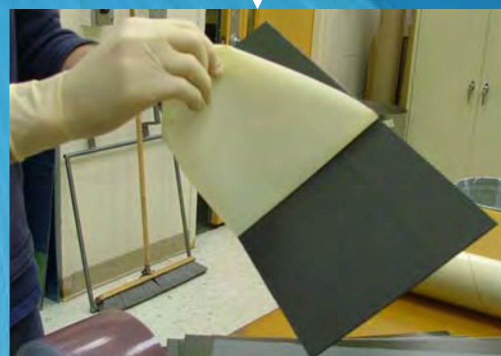
- Fracture of the epoxy between peel ply and carbon fibers
  - Fresh, chemically active, epoxy surface is created
- Interfacial fracture between the peel ply fabric fibers and the epoxy matrix
- Peel ply fiber fracture
- Interlaminar failure



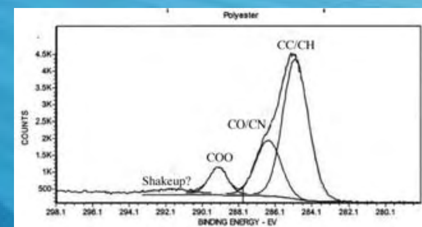
Samples were produced with standard composite processes and characterized.

Unidirectional 10 Ply Toray Carbon Fiber Prepreg Laminates

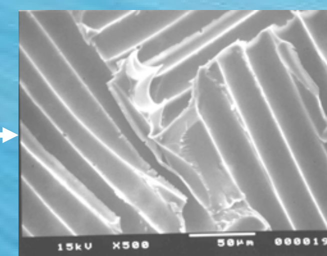
Autoclave Cure



Polyester (PF 60001)
Nylon (PF 52006)
SRB (PF 60001 SRB)



Surface Characterization Via XPS and SEM



Bonded with film adhesive A or B

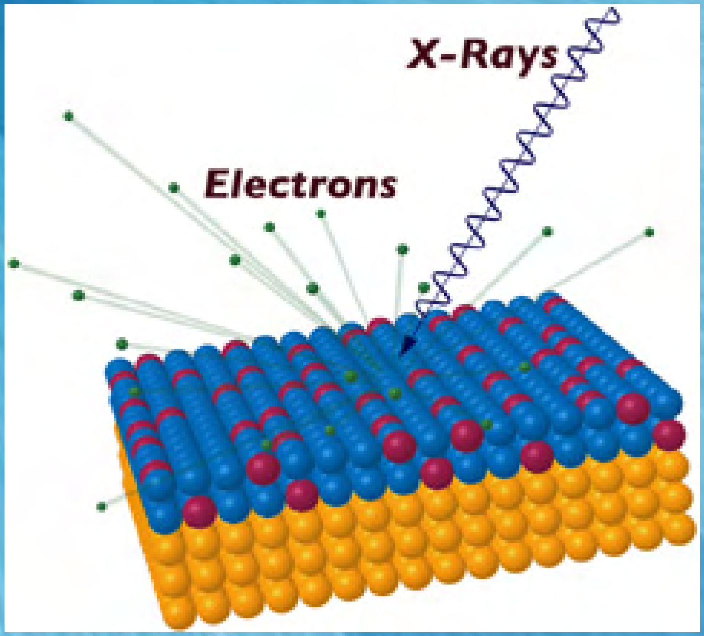
Autoclave Cure

ASTM D-5528
To find G_{IC}

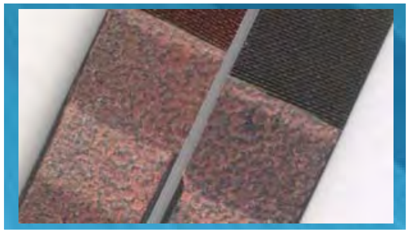
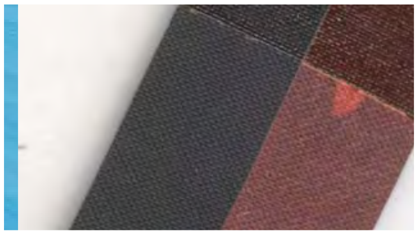
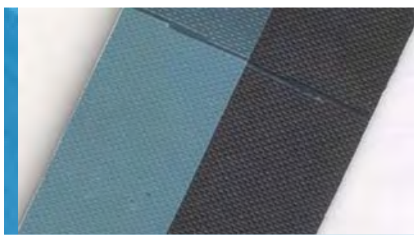
Surface analyzed by XPS/ESCA.

ESCA/XPS: X-Ray Photoelectron Spectroscopy

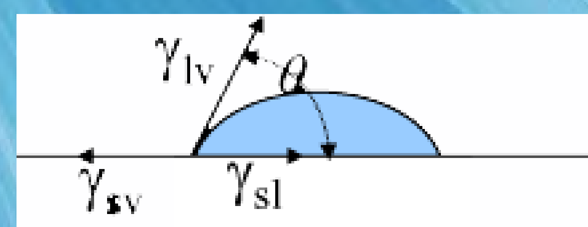
- X-Ray probes energy distribution of valence and nonbonding core electrons
- Gives chemical composition of surface (first few atomic layers)
- Peel ply removed just prior
- Survey scans and high-res scans over C (1s) peak



DCB showed surfaces from polyester bonded well, SRB didn't, and nylon varied.

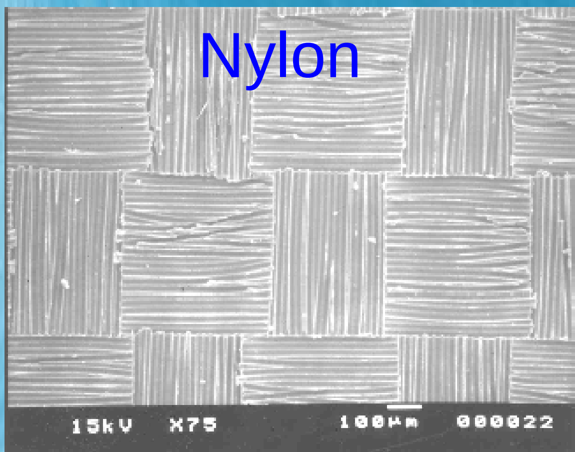
	Polyester Prepared	Nylon Prepared	SRB Prepared
Adhesive A			
Failure Mode	Cohesive	Cohesive & Interlaminar	Adhesion
G_{IC} (J/m^2)	909.6	910.7	93.9
Adhesive B			
Failure Mode	Cohesive	Adhesion	Adhesion
G_{IC} (J/m^2)	812.3	122.1	86.0

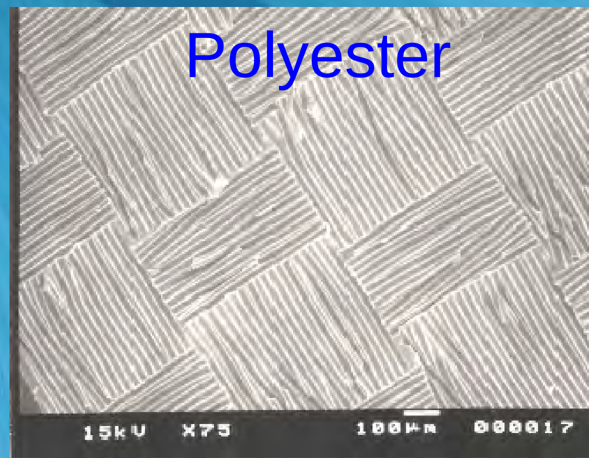
- G_{IC}/Contact angle did not correlate
  - G_{IC}: Polyester >> Nylon > SRB
  - Contact angle: Nylon < Polyester << SRB

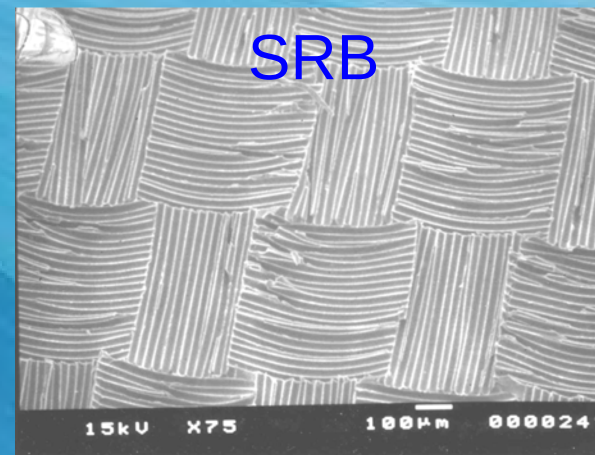


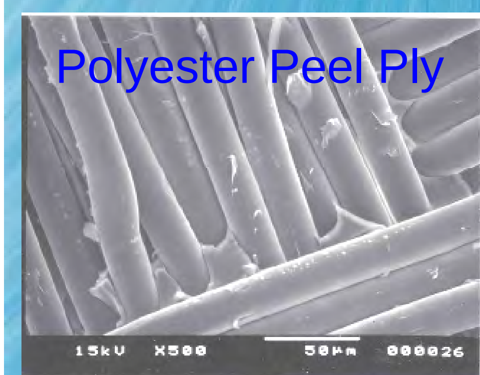
SEM showed that on the macro scale, the samples are acceptably prepared.

Composite surface after removal of:

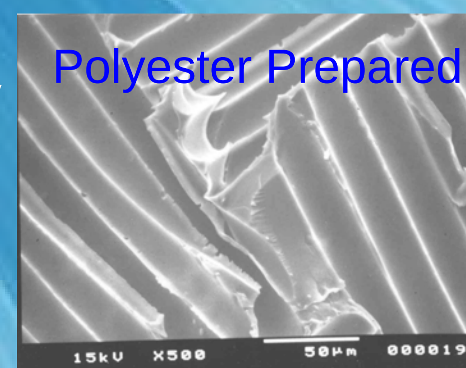




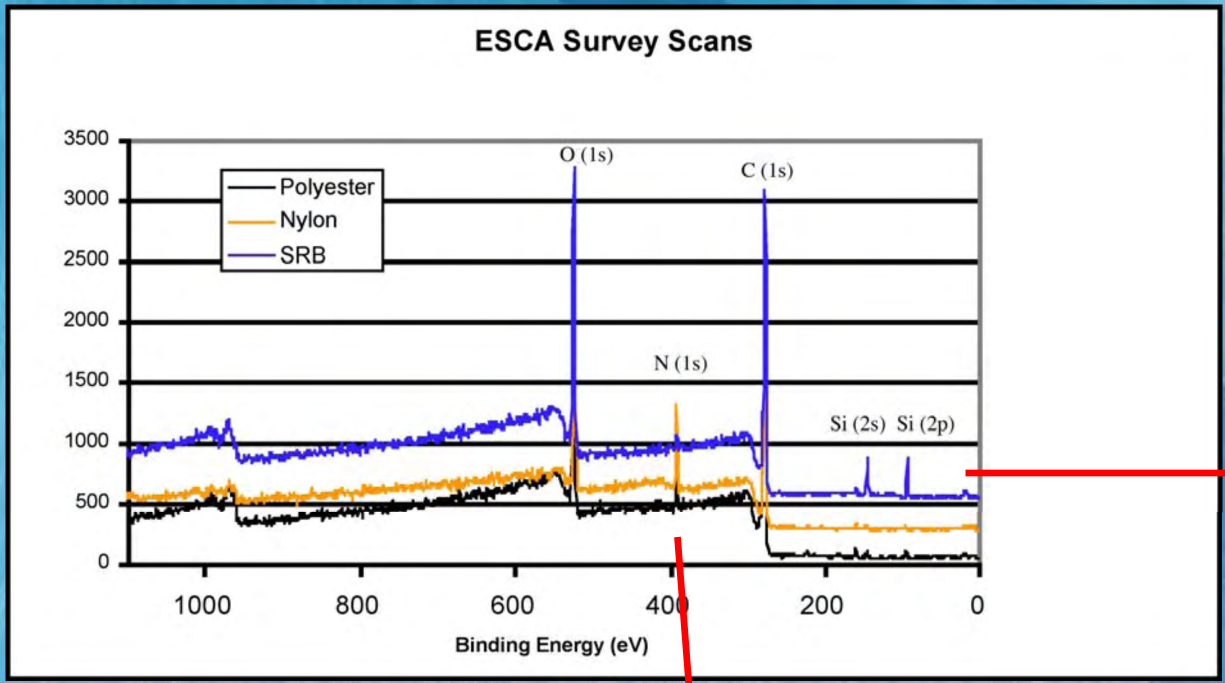




- Texture of peel ply transfers
- Interfacial fracture between the peel ply fabric fibers and the epoxy matrix
- Epoxy is impregnating peel ply
- Limited epoxy fracture between peel ply fibers



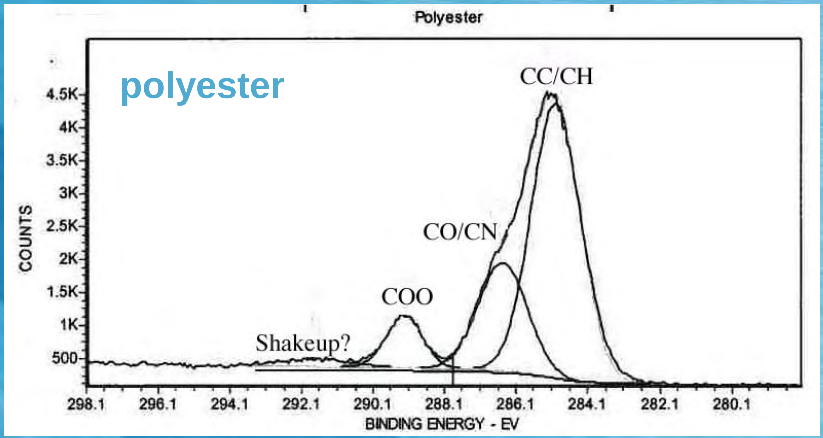
XPS survey scans found contamination on SRB and 52006 prepared surfaces.

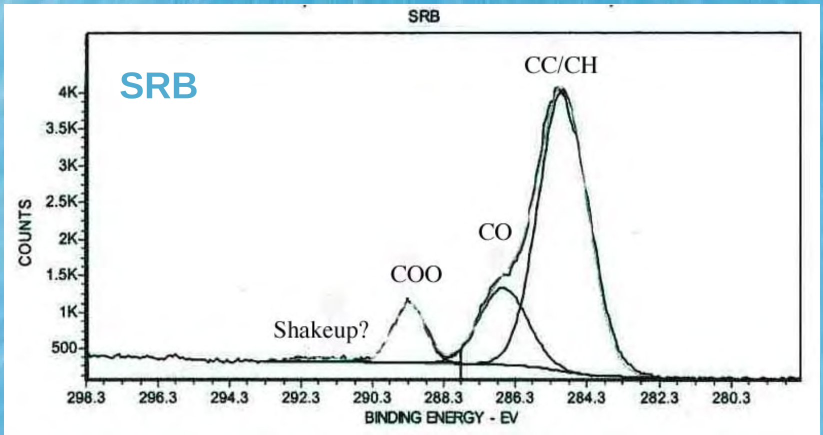


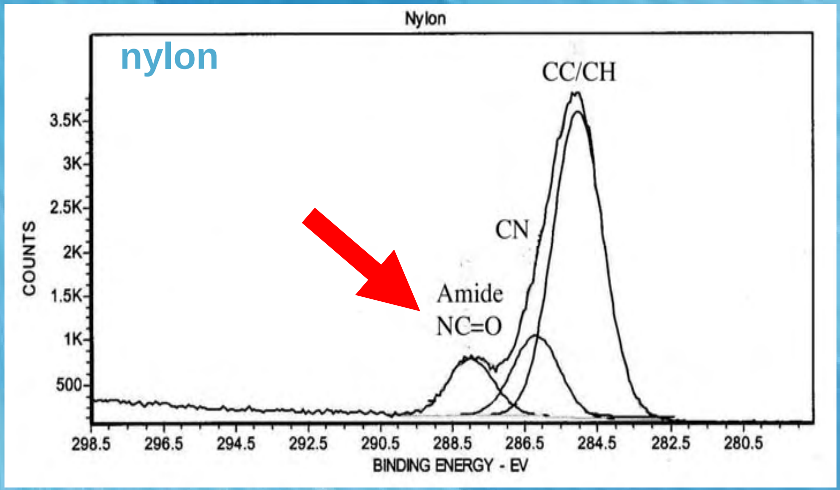
- Si explains SRB low bond quality
  - Siloxane coating transfers
- Amount of N, especially in nylon prepared, unexpected
  - Further suggests material transfer

XPS high-resolution confirmed nitrogen is from nylon peel ply.

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)



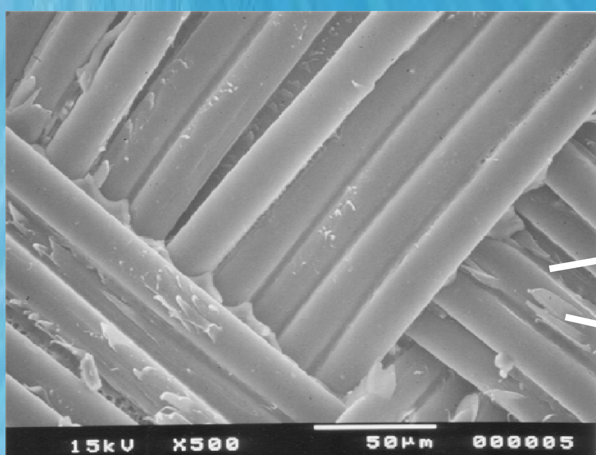


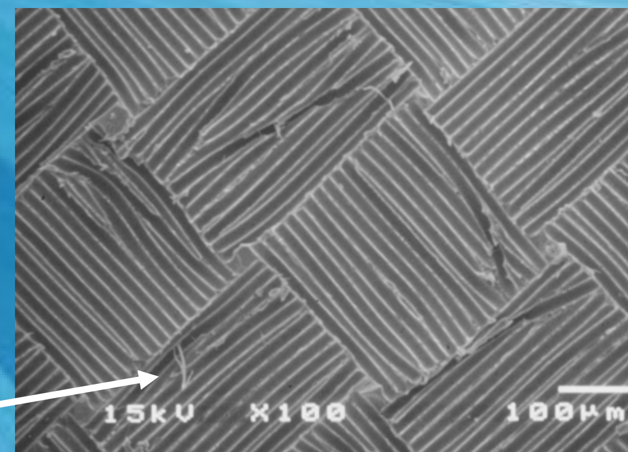


Nitrogen on nylon prepared surface is in amide groups - nylon transfers

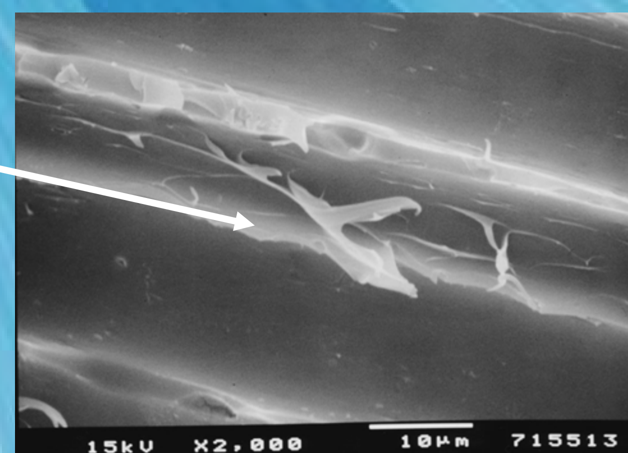
Return to SEM revealed damage to peel ply and remnants on surfaces.

Nicks and strips exfoliated from peel ply surfaces





Blemishes and fibers on laminate surfaces



SRB transfers coating, nylon perhaps transfers fibers?

Conclusions

- Polyester: No Material Transfer; Strong Bonds
- SRB: Siloxane Coating Transfers; Weak Bonds
- Nylon: Fiber May Transfer; bond depends on adhesive
  - Significant amide groups detected
    - Chemical or just mechanical transfer?
- Contact angle did not correlate well with G_{IC}
  - Wetting is necessary….

    ….but not always sufficient for good bond

Questions remain for future work.

- **Is wetting indeed an insufficient criteria for good adhesive/adherend combinations?**
  - **Cure temperature wetting angles**

- **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**?

Acknowledgements

- Precision Fabrics
- The Boeing Company
- FAA/AMTAS
- The University of Washington

Select References

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- 2. Hart-Smith, L, Ochsner, W., and Radecky, R, *Douglas Service Magazine*, First quarter 1984, 12-22.
- 3. Hart-Smith, L, Ochsner, W., and Radecky, R, *Canadair Service News*, Summer 1985, 2-8.
- 4. Persson, P., Lunell, S., Szöke, A., Ziaja, B. and Hajdu, J. *Protein Science* (2001), 10, 2480-2484.

ESCA Details

- Surface Science Instruments M-Probe
- High vacuum environment (<1x10^-8 torr)
- Nominal pass energies of 150 (survey) and 50 eV (detailed)
- Elliptical spots :
  - Major axes ~ 1.7 mm
  - Minor axes ~ 0.4 mm to
  - Depths 10-20 atom layers of the sample surface
  - Low energy electron load-gun set at ~4.0 eV was used for charge neutralization of the samples
    - spectra corrected by setting hydrocarbon C (1s) peak to 285.0 eV