Evaluation of Peel-Ply Materials on Composite Bond Quality

Molly K.M. Phariss*, Brian D. Flinn*, Bjorn Ballien*, William Grace# and Peter J. VanVoast#

* University of Washington, Seattle WA
# The Boeing Company, Seattle, WA
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.

Bond quality is dependent on:

- Chemical Bonding
  - Surface preparation
    - Remove contamination
  - Create chemically active surfaces
- Mechanical Factors
  - Material Compatibility
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

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

Bonded with film adhesive A or B

Surface Characterization Via XPS and SEM

ASTM D-5528 To find $G_{IC}$

Autoclave Cure
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.

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Polyester Prepared</th>
<th>Nylon Prepared</th>
<th>SRB Prepared</th>
</tr>
</thead>
<tbody>
<tr>
<td>Failure Mode</td>
<td>909.6</td>
<td>910.7</td>
<td>93.9</td>
</tr>
<tr>
<td>$G_{IC}$ (J/m²)</td>
<td>812.3</td>
<td>122.1</td>
<td>86.0</td>
</tr>
</tbody>
</table>

- $G_{IC}$/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:

- **Nylon**
- **Polyester**
- **SRB**

- 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.

- Amount of N, especially in nylon prepared, unexpected
  - Further suggests material transfer

- Si explains SRB low bond quality
  - Siloxane coating transfers

- ESCA Survey Scans
  - Polyester
  - Nylon
  - SRB
  - Binding Energy (eV)
  - O (1s)
  - C (1s)
  - N (1s)
  - Si (2s) Si (2p)
XPS high-resolution confirmed nitrogen is from nylon peel ply.

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

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


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