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
  – Good bonds are produced but questions remain:
    • What are appropriate techniques to inspect surfaces?
    • What are key factors for making a good/poor bond?
    • How to predict material and surface preparation compatibility?

• Objective
  – Further understand the effect of peel ply surface preparation on the durability of primary structural composite bonds through surface analysis coupled with mechanical testing and fractography
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

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Samples were produced with standard composite processes and characterized.

- Autoclave Cure
- Peel ply removed before bonding
- Bonded with film adhesive
- Mode I testing

Characterization Via XPS, SEM, Contact Angle
The Rapid Adhesion Test (RAT) Method

– A quick, low cost test which assesses the adhesion between metal-composite bonds.
– A modification of metal-to-metal peel test developed by Boeing.
– The backing adherend clamped to while the peeling adherend is removed.
– Failure mode representative of bond
  • Adhesion Failure-Poor Bond
  • Cohesive Failure-Strong Bond
– Failure modes correlate with DCB test with ~90% less cost and flow time.

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RAT Method Assessment

Cohesive failure (left) vs. Adhesion failure (right)

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Peel Ply Material-250F Cure GFRP

**SUMMARY**

**Nylon - Strong**

**Polyester - Weak**

<table>
<thead>
<tr>
<th>RAT results</th>
<th>updated: 3/24/2006</th>
<th>key: strong bond</th>
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<tbody>
<tr>
<td>spec:</td>
<td>BMS 8-79</td>
<td>mixed strong / very strong bonds</td>
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<tr>
<td></td>
<td>mixed results</td>
<td>weak bond</td>
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<tr>
<td>Prepreg:</td>
<td>HexPly F155</td>
<td>other</td>
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<tr>
<th>peel ply:</th>
<th>3M AF500</th>
<th>3M AF 163-2</th>
<th>Cytec FM94</th>
<th>Henkel EA 9696</th>
<th>Cytec FMx 209</th>
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<table>
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<tr>
<th>Substrate Adhesive</th>
<th>PF60001 Polyester</th>
<th>PF51789 Nylon</th>
<th>Fiberglass-Epoxy</th>
<th>EA9895 PE-Epoxy</th>
<th>Nylon-Epoxy</th>
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<tr>
<td>Cytec 970 MB1515-3</td>
<td>MIXED</td>
<td>ADHESION</td>
<td>COHESIVE</td>
<td>COHESIVE</td>
<td>ADHESION</td>
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<tr>
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<td>MIXED</td>
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<td>COHESIVE</td>
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<tr>
<td>Toray 3631 MB1515-3</td>
<td>ADHESION</td>
<td>ADHESION</td>
<td>NA</td>
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<td>ADHESION</td>
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<tr>
<td>Toray 3631 AF555</td>
<td>ADHESION</td>
<td>ADHESION</td>
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<td>ADHESION</td>
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<td>COHESIVE</td>
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Peel Ply Surface Prep. - SEM Results

Composite surface after removal of:

- 260 F cure GFRP
- Cytec 970 (360F)
- Toray 3900 (360 F)

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Wettability envelopes showed the difference in the prepared surfaces.

- Fluids inside the envelope will wet spontaneously
  - Critical condition for bonding?
- Wettability envelopes a potential method to determine suitability of a surface for bonding
- Epoxy adhesives* on boundary for nylon prepared surfaces

* Literature values for aerospace epoxies
  - Curves generated using WET program (M. Tuttle)
XPS Survey Scan Results

Laminate surfaces before bonding, after peel ply removal

<table>
<thead>
<tr>
<th>Peel Ply</th>
<th>%C</th>
<th>%O</th>
<th>%N</th>
<th>%Si</th>
</tr>
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<tbody>
<tr>
<td>Nylon</td>
<td>77.5</td>
<td>12.6</td>
<td>9.8</td>
<td>Tr.</td>
</tr>
<tr>
<td>Polyester</td>
<td>75.5</td>
<td>21.6</td>
<td>1.9</td>
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<tr>
<td>SRB</td>
<td>68</td>
<td>24.2</td>
<td>0.9</td>
<td>6.9</td>
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</tbody>
</table>

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

• Bonding Depends on
  – Prepreg system (Resin and Fiber(?))
  – Peel Ply Material and Source
  – Adhesive

• Characterization Techniques (XPS, SEM and Surface Energy) provide useful information to help understand bonding requirements
A Look Forward

• Benefit to Aviation
  – Better understanding of peel ply surface prep.
  – Greater confidence in adhesive bonds

• Future needs
  – Contact angle (wetting) vs. bond quality
  – Does fiber type (glass, pitch, PAN) effect bonding?
  – Peel ply-resin interactions
  – Applicability to other composite and adhesive systems
  – Model to guide bonding based on characterization, surface prep. and material properties
A Closer Look at the Laminate Surface

Laminate surface after removal of nylon peel ply

Nylon from peel ply on surface before bonding?
Bond Quality Depends on:

- Peel Ply Material and Adhesive
  - Polyester peel ply: high toughness bonds, cohesive failure both adhesives
  - Nylon: low toughness, adhesion failure
  - One adhesive bonded well to all surfaces

- $H_2O$ Contact angle did not correlate well with $G_{IC}$
- Wettability envelopes more accurate
- XPS can provide important chemical information
250F Cure Systems

- 2 Peel Plies: Polyester 60001 and Nylon 52006
- 3 prepregs-260 °F cure
  - HexPly® F155
  - Yokohama G7781
  - Cytec MXB7701
- 6 adhesives-260 °F cure
  - 3M AF500; 3M AF163-2;
  - Henkel EA 9696; Henkel EA 9628
  - Cytec FM94; Cytec FMx 209
- Bond quality assessed by failure mode
  - Adhesion (poor) vs. Cohesive (good)
Bond Quality Depends on:

- **Peel Ply Material and Adhesive**
  - Nylon: high toughness bonds, cohesive failure all adhesives
  - Polyester peel ply: low toughness, adhesion failure
  - One adhesive bonded well to all surfaces

- **Opposite Trend than 350 F system**
  - Nylon bad, Polyester good