Improving Adhesive Bonding of Composites Through Surface Characterization

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Improving Adhesive Bonding of Composites

- Motivation and Key Issues
  - Adhesive bonding is being used for primary composite structure in commercial transport aircraft manufacture and repair- surface preparation is a critical step
  - 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 requirements for surface preparation to produce strong primary structural composite bonds with different substrates and adhesives

Effect of Surface Preparation Technique on Bond Quality of AGATE Composite Laminates

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Effect of Surface Preparation Technique on Bond Quality of AGATE Composite Laminates

- Motivation and Key Issues
  - AGATE database for general aviation composite materials exists
  - No guidance found for:
    - bonding or surface preparation procedures
    - QA for surface for bonding
- Objective
  - Investigate surface preparation procedures for bonding AGATE materials and characterize surfaces
- Approach
  - Apply research protocols established in prior research on surface preparation and bonding for commercial aircraft structures

FAA Sponsored Project Information

- Principal Investigators & Researchers
  - Brian D. Flinn (PI)
  - Curtis Hickmott (BS 2009, new PhD student, UW-MSE)
  - Jeffery Saterwhite (MS 2009 UW-MSE)
- FAA Technical Monitor
  - David Westlund (2009) & Curt Davies (2008)
- Other FAA Personnel Involved
  - Larry Ilcewicz
- Industry Participation
  - Toray Composites
  - Henkel International
  - Precision Fabrics & Richmond Aerospace & Airtech International
  - The Boeing Company

Outline

- Background
  - AGATE materials
  - Bonding/Surface Preparation
- Processing and Test Procedures
- Surface Characterization
  - Contact Angle Results
  - Surface Energy
  - SEM
- Bond Quality Tests
- Summary

AGATE Materials

Three prepreg materials tested:

Toray AGATE materials(Toray 2510 resin)

- Carbon Tape,
- Carbon Fabric
- Fiberglass Fabric
- 132 C (270 F) cure for 2 hours with vacuum bag cure (no autoclave)

Surface Preparation

- Crucial for proper adhesion in composites
- Several methods
  - Peel ply (as tooled)
  - Abrasion (Sanding or grit blasting)
- Surface preparation influences surface energy and the wettability of a surface, also prevents/removes contamination
- A high energy surface promotes intimate contact between the surface and the adhesive

Low Energy Surface

High Energy Surface

Bonding

- Secondary Bonding
  - Most sensitive to surface preparation
- Bonding Mechanisms
  - Chemical
  - Mechanical

- Evaluation of bond quality is very difficult
  - Producing strong bonds is still very dependant on compatibility between adhesives and substrate surface preparation
  - Most effective way to evaluate a bond is to test it

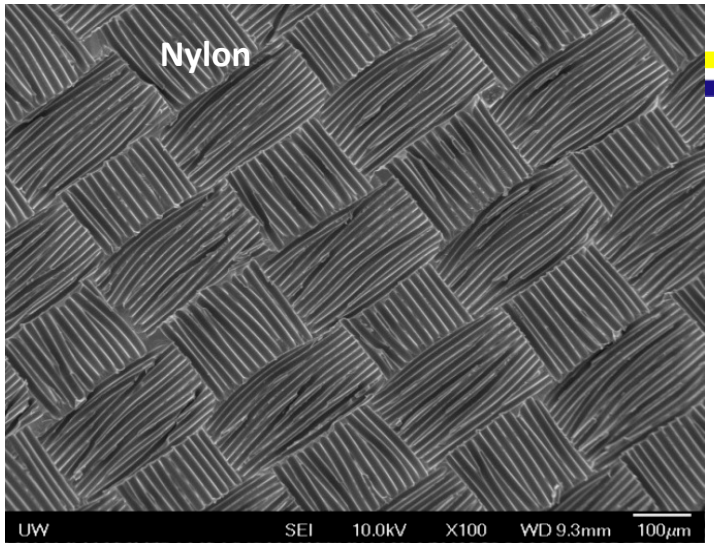
Procedures

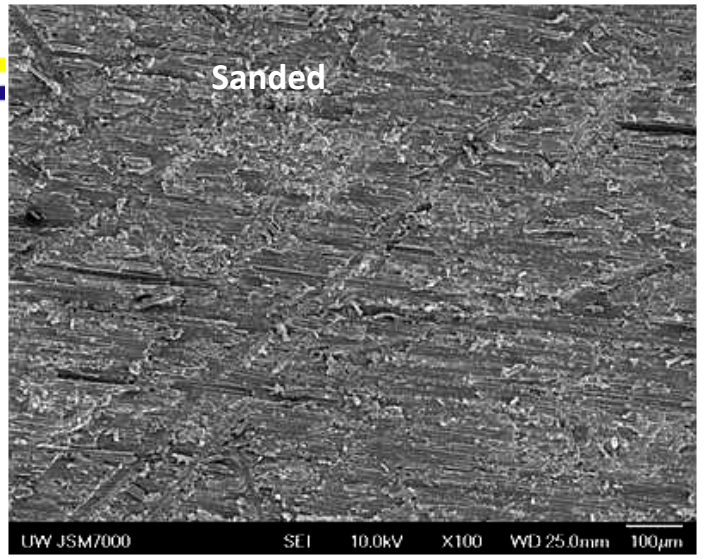
- Surface Preparation
  - Peel ply – Precision Fabrics Group 52006 nylon or 60001 polyester
  - Sanded – Hand sanded with 60 grit Al_2O_3
- Contact angle measurements taken with six standard fluids
- Surface energy determined using Owens-Wendt two parameter model

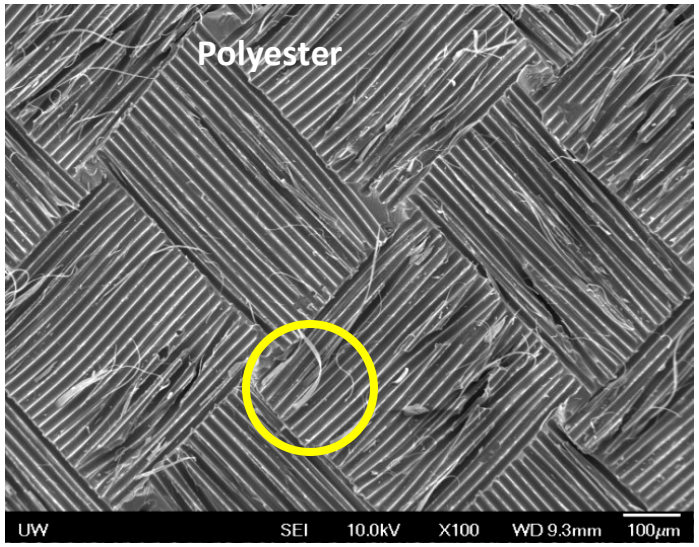
$$\frac{\gamma_L\,[(\cos(\theta)+1]}{2\sqrt{(\gamma_L{}^d)}} = \sqrt{(\gamma_S{}^p)}\,\frac{\sqrt{(\gamma_L{}^p)}}{\sqrt{(\gamma_L{}^d)}} + \sqrt{(\gamma_S{}^d)} \qquad \sqrt{(\gamma_S{}^d\,\gamma_L{}^d)} + \sqrt{(\gamma_S{}^p\,\gamma_L{}^p)} = \gamma_L$$

- Bonding procedure –
  - film adhesive (Henkel EA 9696), vacuum bag cure, 132C (270F)
- Rapid Adhesion Tests (RAT)
  - for bond quality assessment (Mode I peel test)
- SEM images of the substrate and the RAT bond fracture surfaces

Substrates after Surface Preparation





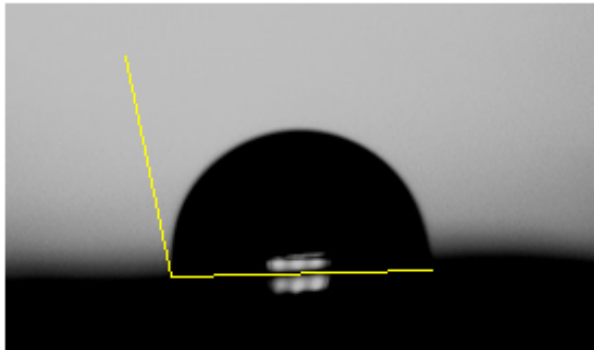


- Fiber reinforcement did not have significant effect on surface characteristics
- Representative carbon fabric surfaces shown
- Sanding removed any peel ply imprint
- Remnants of polyester peel ply visible

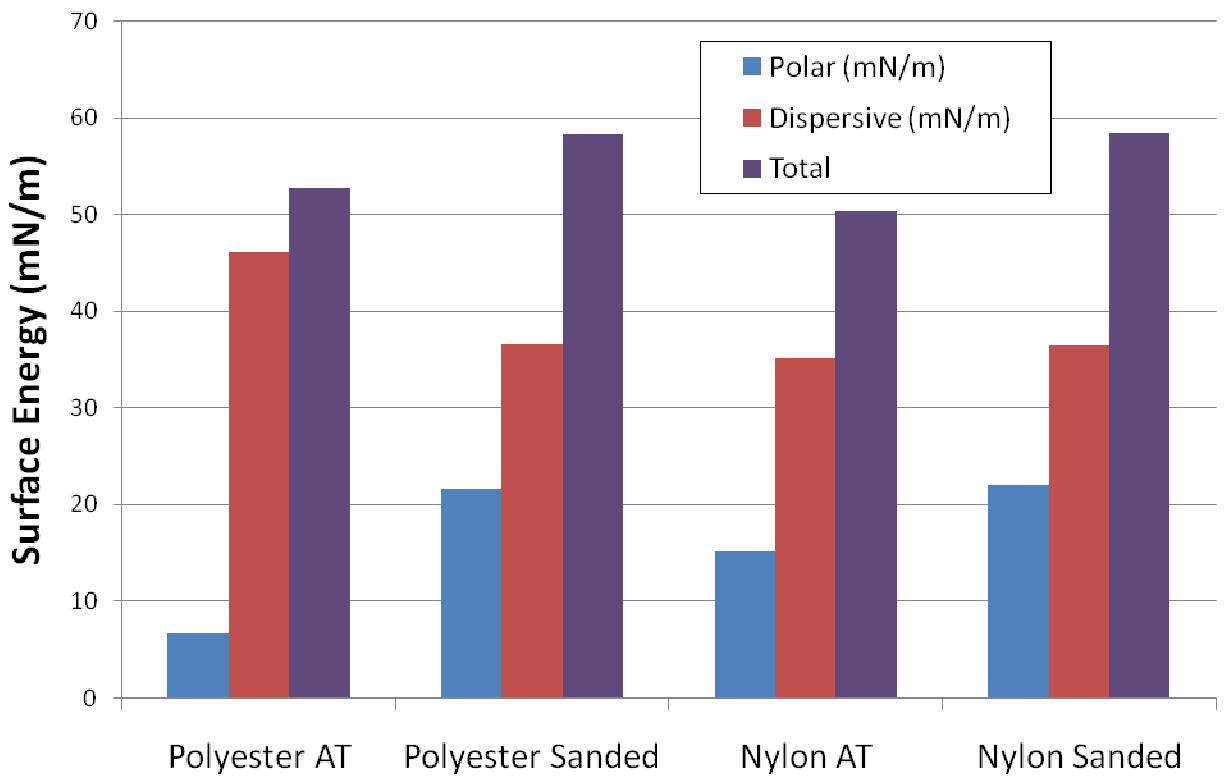
Contact Angle Measurements

Substrate Preparation	H2O	Ethylene Glycol	DMSO	Tetrabro-moethane	Diiodo-methane	Formamide
Weave Polyester Sanded	50.7	Wet out	Wet out	Wet out	Wet out	Wet out
Weave Nylon Sanded	49.8	Wet out	Wet out	Wet out	Wet out	Wet out
Weave Polyester As Tooled	81.4	14.6	Wet out	Wet out	Wet out	29.1
Weave Nylon As Tooled	57.9	34.6	28.1	11.2	22	41.6

- 10 values were taken on each side of the drop and averaged
- "Wet out" defined as $\Theta = 0^{o}$
- Sanding appears to increase the number of fluids that have "wet out" the surface

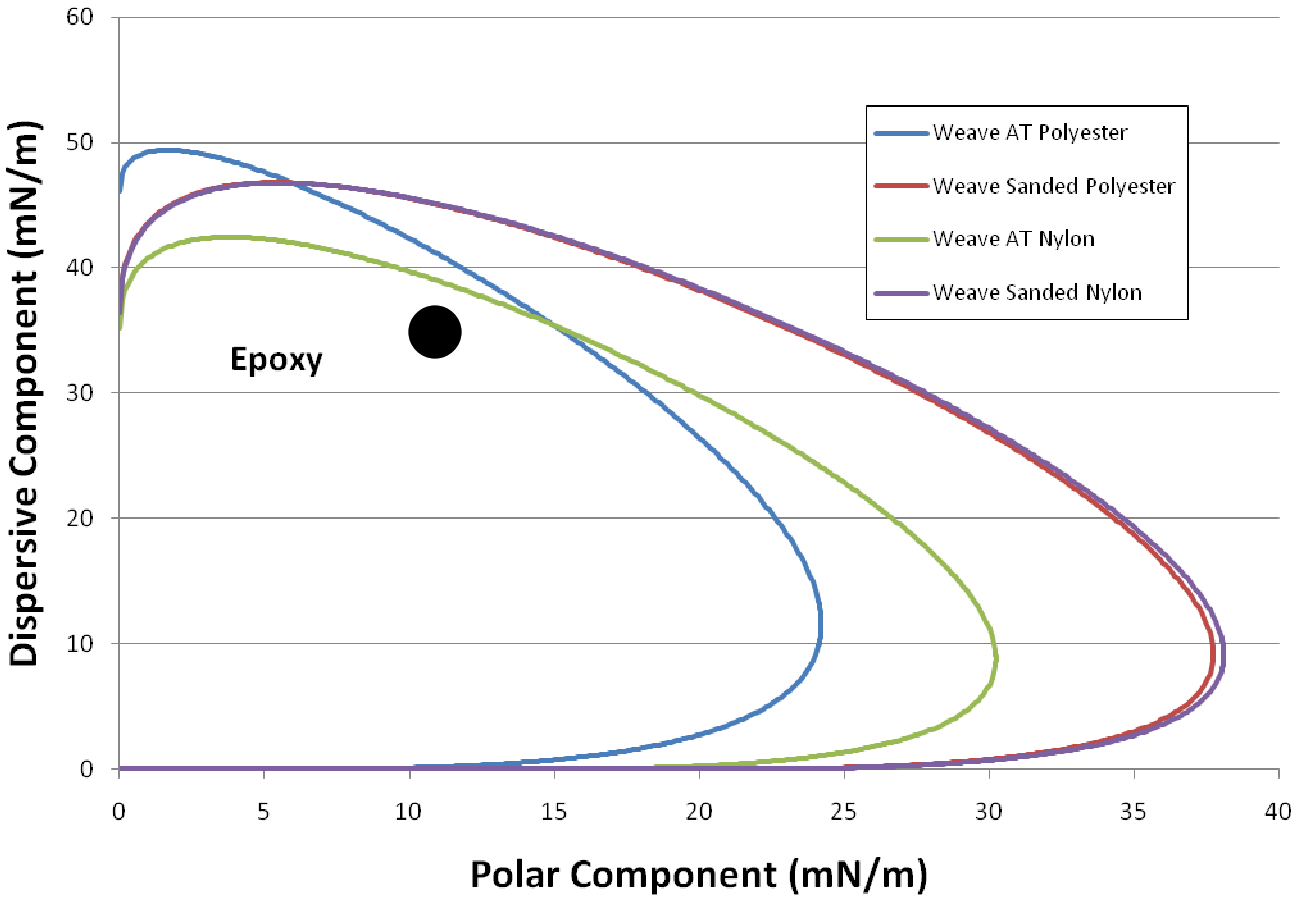


Surface Energy



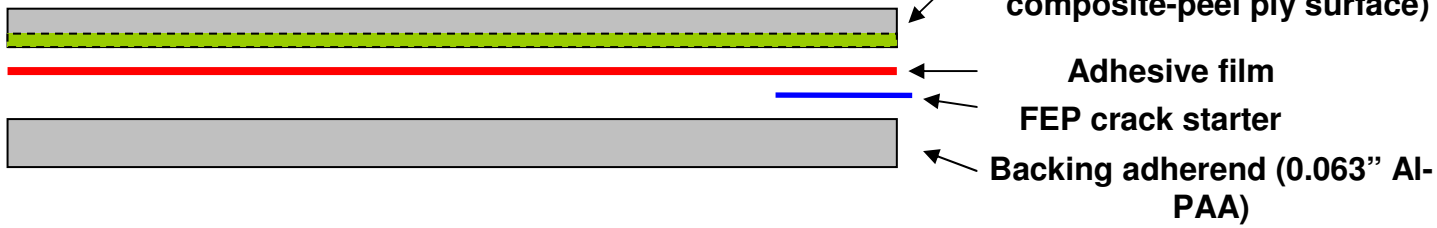
- The polar component and total surface energy were found to increase after sanding
- Sanding decreased dispersive component of polyester surface

Wettability Envelopes – Carbon Fabric



- **Wettability envelopes converged after sanding**
- **Fiber form did not influence wettability envelop significantly**

Rapid Adhesion Test (RAT)



- The RAT sample is a simple and accurate bond test used to determine mode of failure
- By examining the surface and determining the mode of failure the quality of the bond can be assessed

RAT Peel Test

Types of Bond Failures

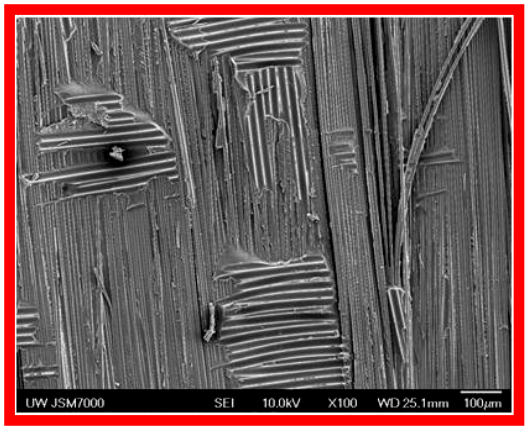
- Failure of Adhesion - Bad

- Cohesive (epoxy or adhesive) - Good

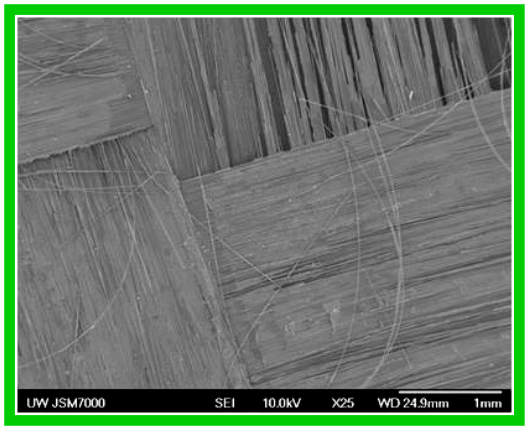
- Inter/intralaminar - Good

Rapid Adhesion Test Samples

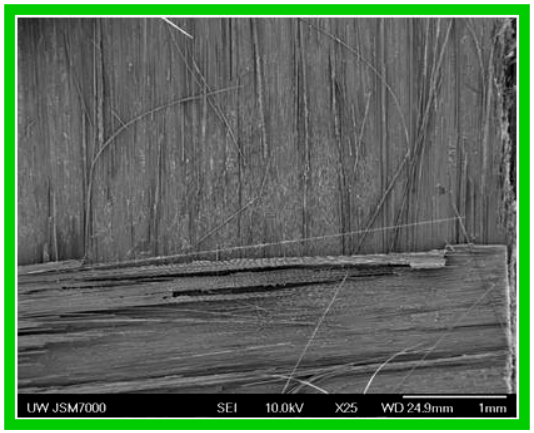
	Surface Preparation		
Substrate	As- Tooled polyester	As- Tooled nylon	Sanded
Carbon Tape	MIXED	COHESIVE/INTERLAMINAR	COHESIVE/INTERLAMINAR
Carbon Fabric	MIXED	COHESIVE/INTERLAMINAR	COHESIVE/INTERLAMINAR
Glass Fabric	MIXED	COHESIVE/INTERLAMINAR	COHESIVE/INTERLAMINAR



Polyester As Tooled



Nylon As tooled



Sanded

- The polyester as tooled samples were the only samples that did not produce a good bond
- Polyester fibrils may have contaminated the surface of the substrate

Discussion of Findings

- Polyester peel ply remnants left on substrate surface
- No nylon remnants detected in SEM
- Polyester had a greater dispersive character
- Sanding created equivalent surface energies
- As tooled polyester substrates did not bond well
- As tooled nylon substrates bonded well
- Sanded surfaces bonded well
- Sanding removed peel ply imprint and remnants
- Findings similar to commercial aircraft 250F autoclave cure systems (BMS 8-79 & BMS 8-168)

Work In Progress…

- More Adhesives
  - Paste (Henkel 9394, etc)
  - Other films
- Other Substrates? (input requested)
- Quantitative Fracture Testing
  - DCB Testing
  - Hot Wet Testing
    - Does surface preparation affect durability?

Conclusions

- Fiber type had little effect on surface chemistry, surface preparation and bond quality.
- Good bonds were produced with the Toray AGATE materials and the Henkel EA 9696 adhesive with the proper surface preparation.
- Wettability envelopes illustrated the different surface characteristics produced by the surface preparations.
- Wettability envelopes may have a role in assessing surface preparation.

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A Look Forward

- Benefit to Aviation
  - Better understanding of peel ply surface prep.
  - Guide development of QA methods for surface prep.
  - Greater confidence in adhesive bonds
- Future needs
  - Surface energy (wetting) vs. bond quality
  - Surface energy at cure temperature
  - QA method to ensure proper surface for bonding
  - Applicability to other composite and adhesive systems
  - Model to guide bonding based on characterization, surface prep. and material properties

QUESTIONS ?
COMMENTS?
SUGGESTIONS?