Improving Adhesive Bonding of Composites through Surface Characterization

Role of Surface Preparation on Durability of Bonded Composite Joints

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Outline

- Background
  - AGATE materials
  - Bonding/Surface Preparation
- Materials and Methods
- Surface Characterization
  - Contact Angle Results
  - Surface Energy
  - SEM
- Bond Quality Tests
- Summary

FAA Sponsored Project Information

- Principal Investigators & Researchers
  - Brian D. Flinn (PI)
  - Curtis Hickmott (BS 2009, new PhD student, UW-MSE)
  - John Aubin (MS 2009 UW-MSE)
- University Participation- Prof. Mark Tuttle
- 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

AGATE Materials

- AGATE – Advanced General Aviation Transport Experiment
- A database for general aviation composite materials is currently being built, it is hoped that this research will help add to that data base
- Benefits of AGATE Program- database reduces testing, easier design with standardized materials
- No guidance yet for bonding /surface prep.

➢ **Objective: determine the best surface preparation for bonding AGATE materials**
  - Fracture Mode
  - Fracture Energy
  - Durability

Bonding

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

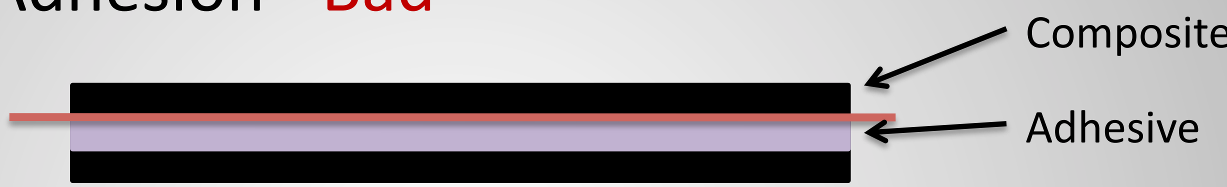
- Evaluation of bond quality is very difficult
  – Producing strong bonds is still very dependant on compatibility between adhesives, substrates and surface preparation
  – Most effective way to evaluate a bond is to test it

Bond Quality Evaluation

- The only sure way to measure bond quality is to break it
  - Standard Specimens:
    - Lap Shear
    - DCB Interlaminar Mode I Fracture Energy
  - Fractography- Failure Mode
    - Cohesive/Interlaminar
    - Adhesion- at Bond Line-
  - Durability- Accelerated testing by hot wet exposure

Types of Bond Failures

- Failure of Adhesion - Bad

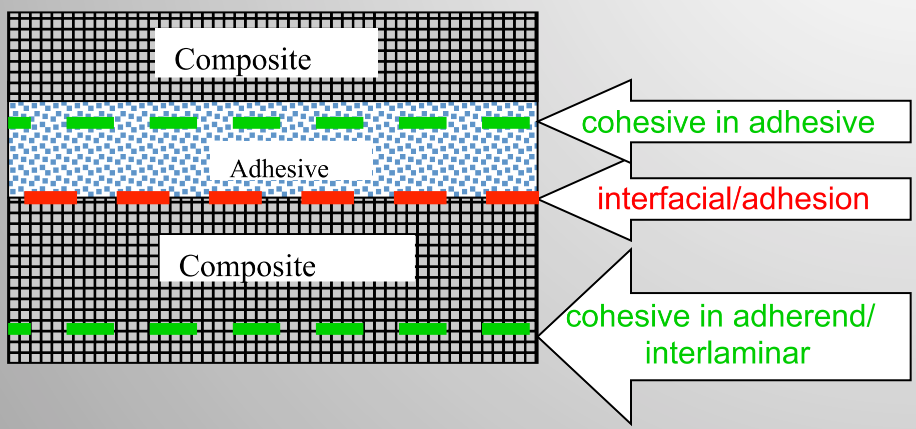


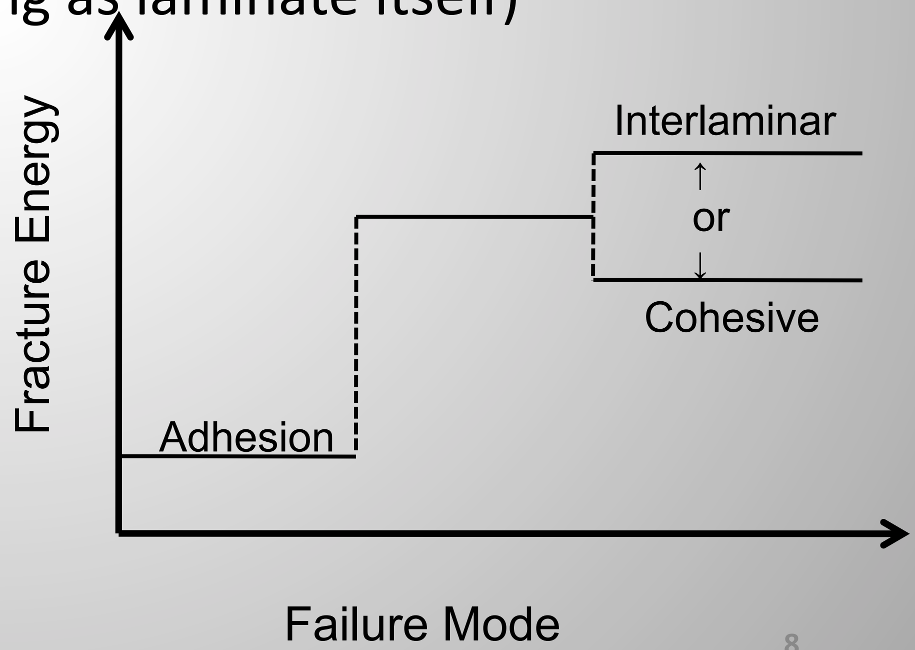
- Cohesive (matrix or adhesive) - Good

- Interlaminar - Good

Fracture Evaluation

- Failure mode and relation to bond strength:
  - **Adhesion** (weak bond)
    - x Considered unacceptable in aerospace
  - **Cohesion** (bond as strong as adhesive itself)
    - ✓ Acceptable
  - **Interlaminar** (bond as strong as laminate itself)
    - ✓ Acceptable





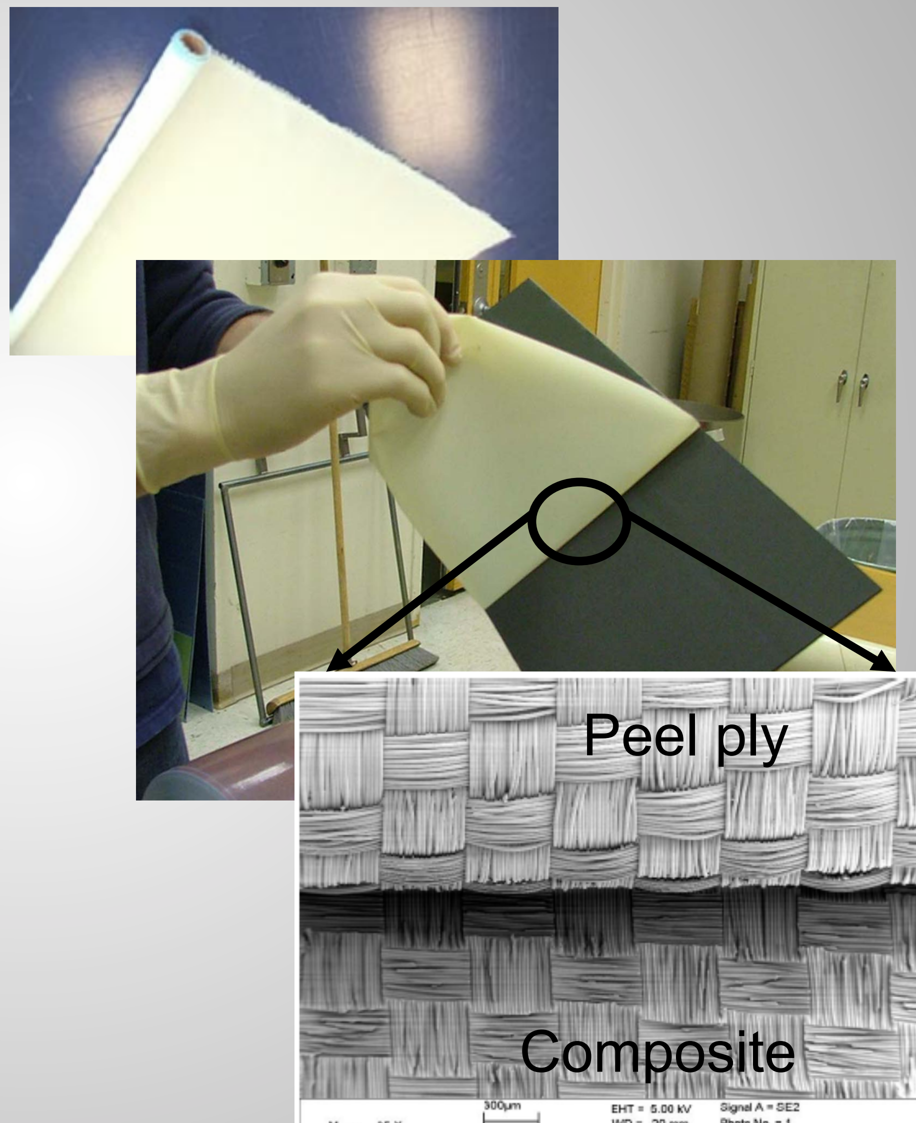
Surface Preparation

- Crucial for proper adhesion in composites
- Several methods
  - Peel ply (as tooled)
  - Abrasion (Sanding or grit blasting)
  - Plasma and other chemical treatments
- Surface preparation influences surface energy and the wettability of a surface, also can prevent and remove contamination
- A high energy surface promotes intimate contact between the surface and the adhesive –requirement for strong bond

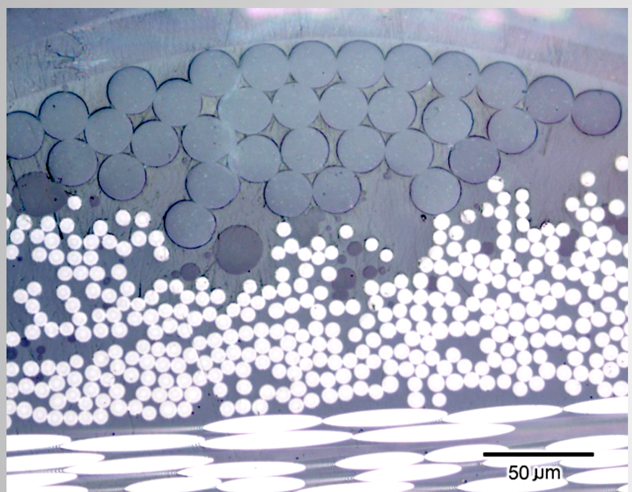
Peel Ply Surface Preparation

- Peel Ply-Woven fabric
  - Typically thermoplastic polymer
  - Placed on surface during layup
- Cured with the part – matrix resin infiltrates peel ply weave
- Removed just before bonding
- Ideally Leaves rough, clean, chemically active surface
- Benefits:
  - straightforward
  - consistent
- If only they always worked…

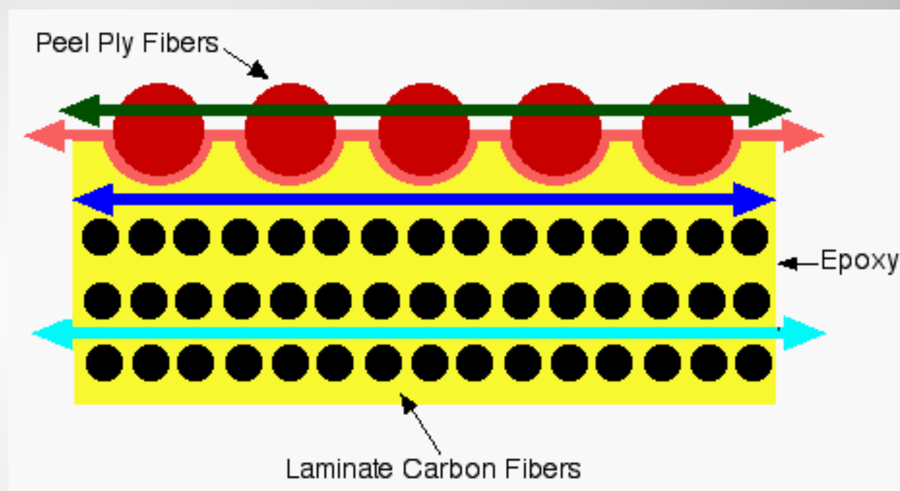
system dependent (peel ply, matrix & adhesive)



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

Materials & Methods

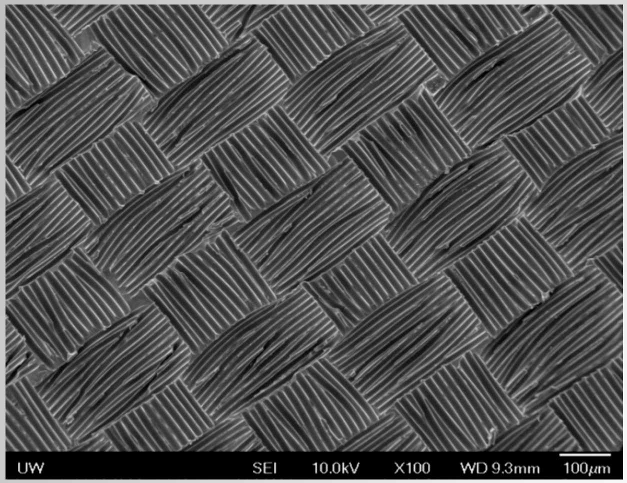
- Three materials tested – Toray AGATE materials
  - Carbon Tape, Carbon Fabric, and Glass Fabric (Toray 2510 resin)
  - 132 C (270 F) cure for 2 hours with vacuum bag cure (no autoclave)
- Surface preparation
  - Peel ply – 52006 nylon or 60001 polyester
  - Sanded – Hand sanded with 60 grit Al_2O_3
- Contact angle measurements taken with six standard fluids
- Surface energy determined using Owens-Wendt two parameter model

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

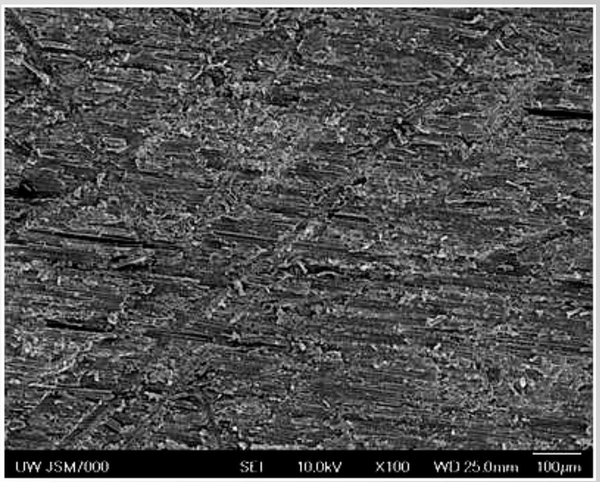
$$\sqrt{(\gamma_S{}^d\,\gamma_L{}^d)} + \sqrt{(\gamma_S{}^P\,\gamma_L{}^P)} = \gamma_L$$

- Bonding procedure - film adhesive (Henkel EA 9696), vacuum bag cure, 132 C (270 F).
- Bond Quality : Rapid Adhesion Test (RAT) and DCB (ASTM D-5258)
- SEM images were taken of the surfaces prior to bonding and the DCB sample fracture surfaces

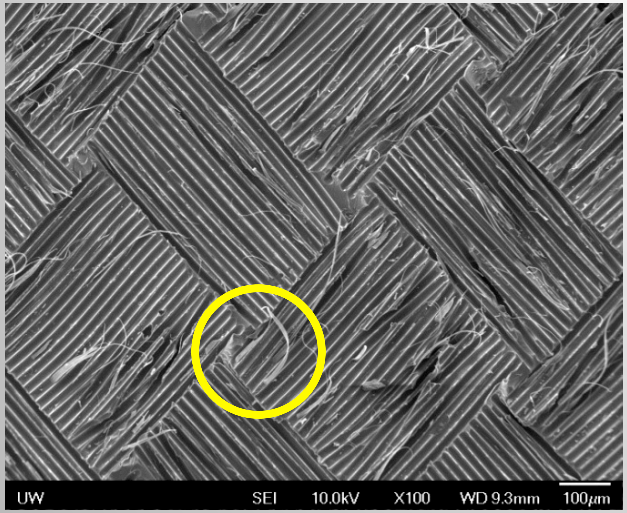
SEM Images of AGATE Substrates after Surface Preparation



Nylon



Sanded

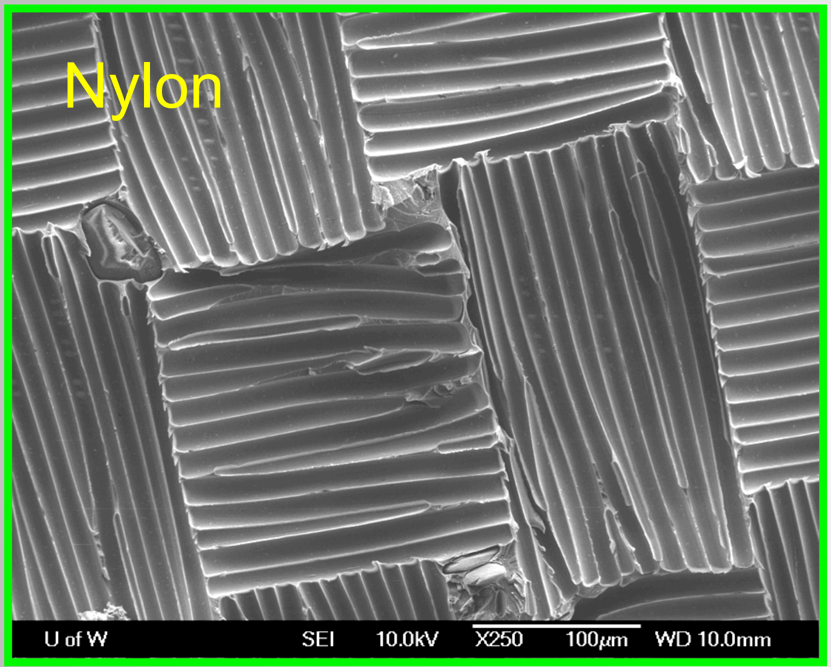


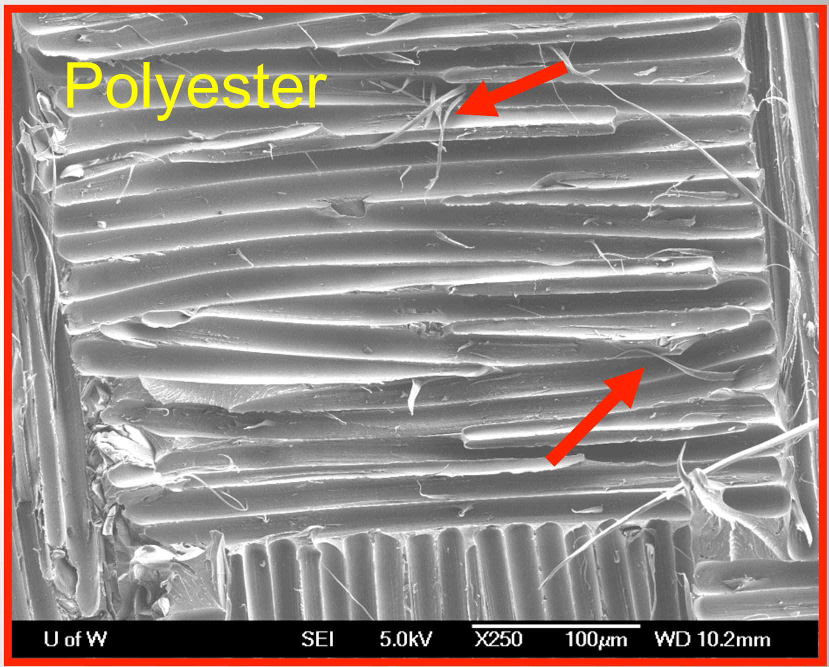
Polyester

- 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

Peel Ply Surface Prep. - SEM Results

Composite surfaces after removal of peel ply:



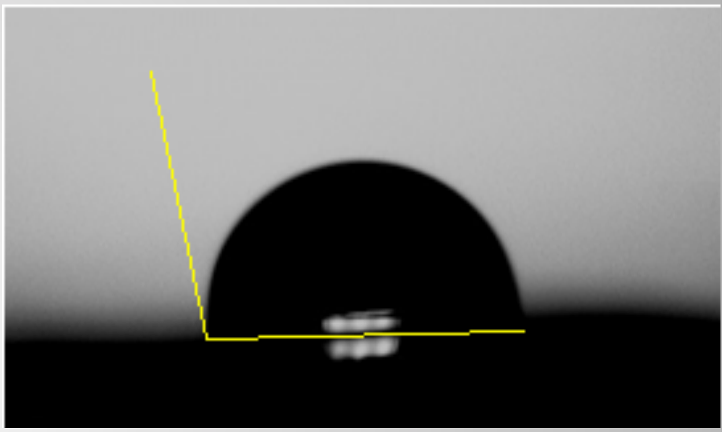


"Clean" surface

Remnants of polyester peel ply fibers left on surface

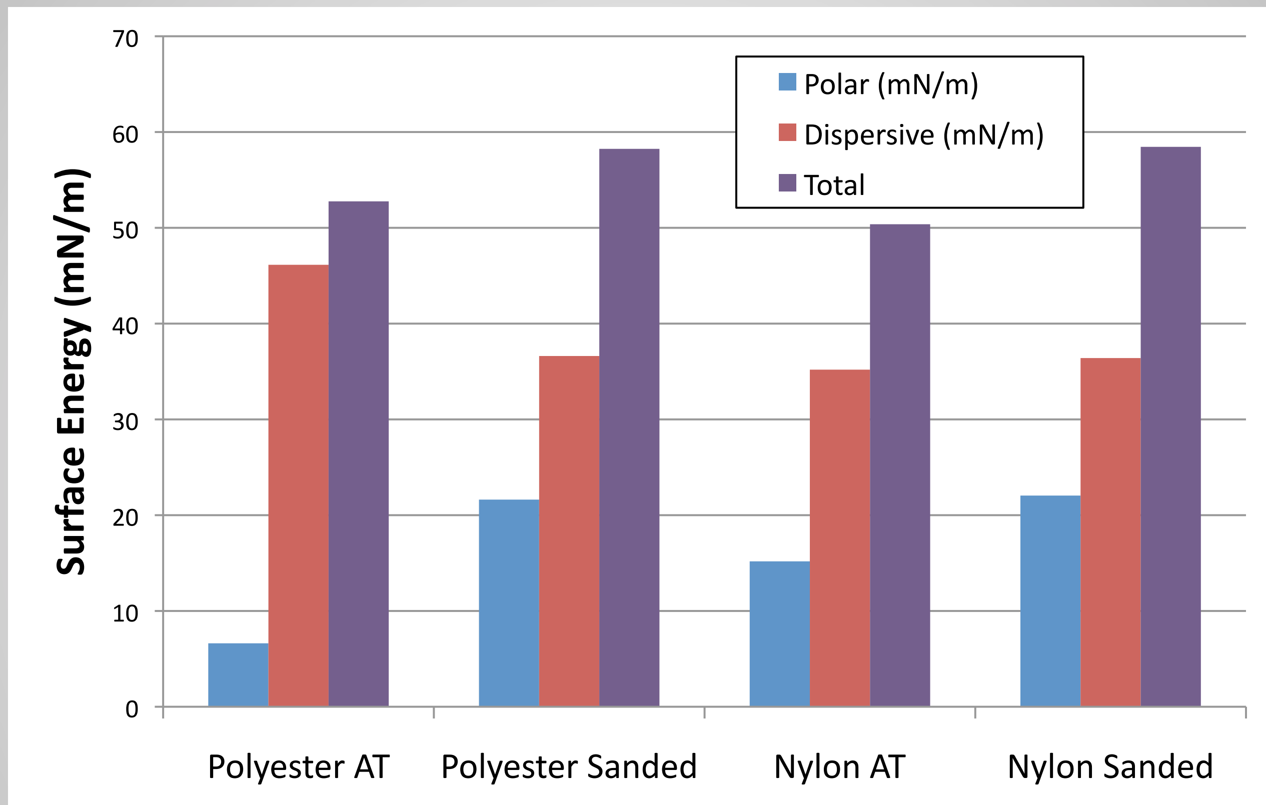
Contact Angle Measurements

- 10 values were taken on each side of the drop and averaged
- Fiber did not influence contact angle
- "Wet out" defined as $\Theta = 0^o$
- Sanding increased the number of fluids that "wet out" the surface



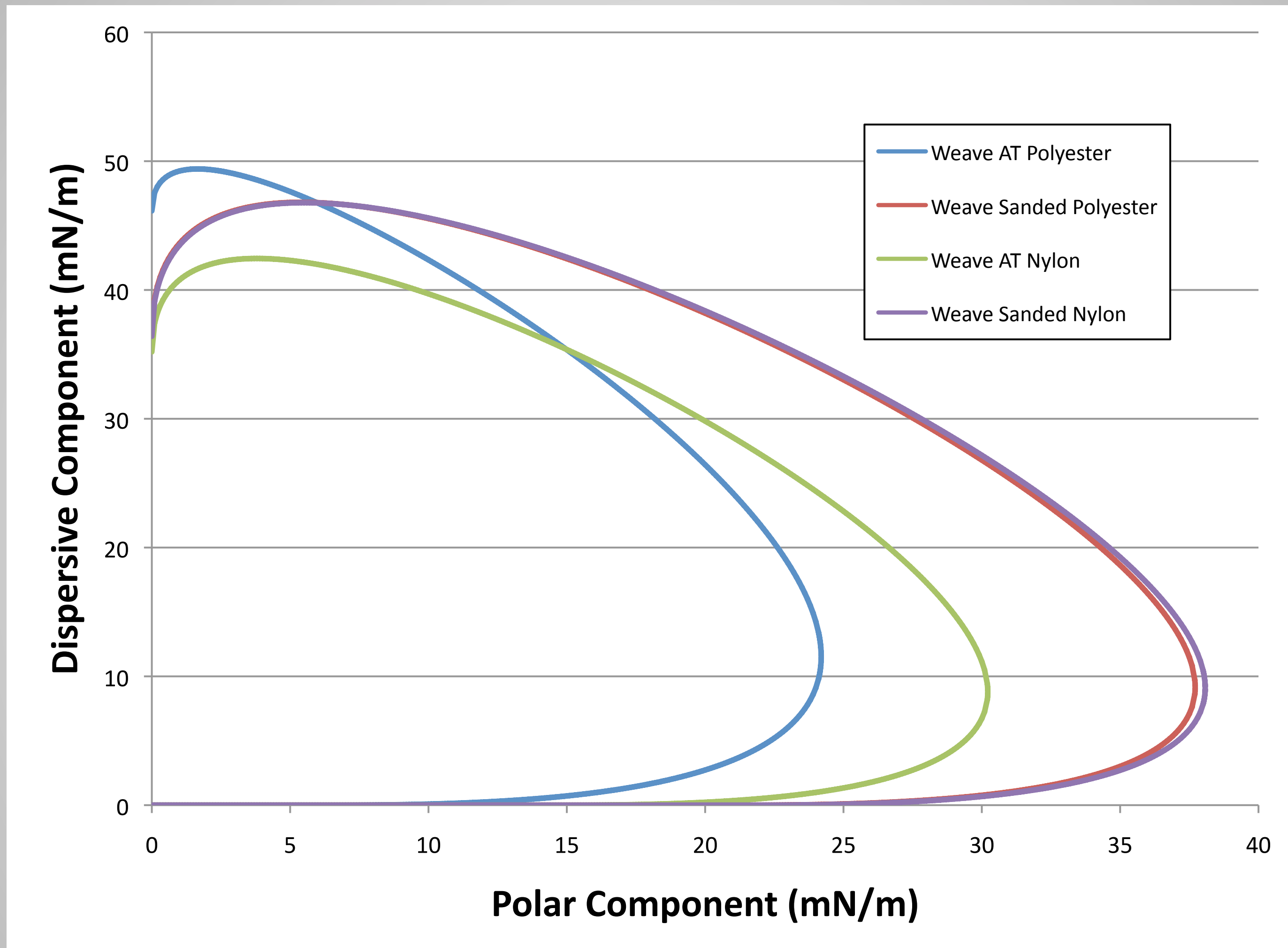
Substrate Preparation	H2O	Ethylene Glycol	DMSO	Tetrabro-moethane	Diiodo-methane	Formamide
Weave Polyester Sanded	50.7	Wet out	Wet out	Wet out	Wet out	Wet out
Weave Nylon Sanded	49.8	Wet out	Wet out	Wet out	Wet out	Wet out
Weave Polyester As Tooled	81.4	14.6	Wet out	Wet out	Wet out	29.1
Weave Nylon As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

Surface Energy Toray 2510



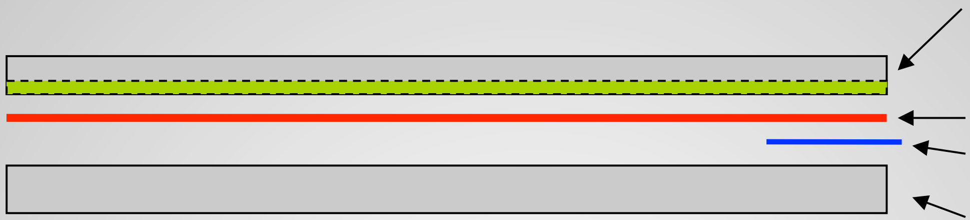
- The polar component and total surface energy were found to increase after sanding
- Sanding decreased dispersive component of polyester surface

Wettability Envelopes – Carbon Fabric



- Wettability envelopes converged after sanding

Bond Evaluation-Rapid Adhesion Test (RAT)

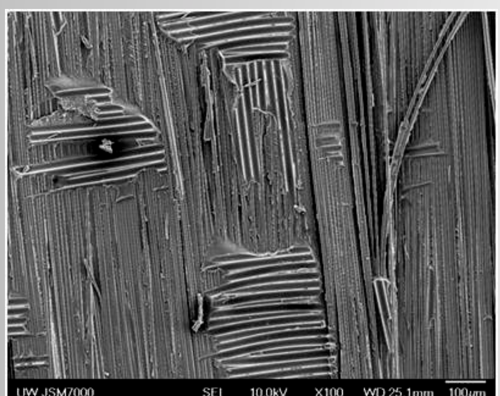


- The RAT sample is a simple and accurate bond test used to determine mode of failure
- Typically 1 ply of composite is used
- By examining the surface and determining the mode of failure the quality of the bond can be assessed

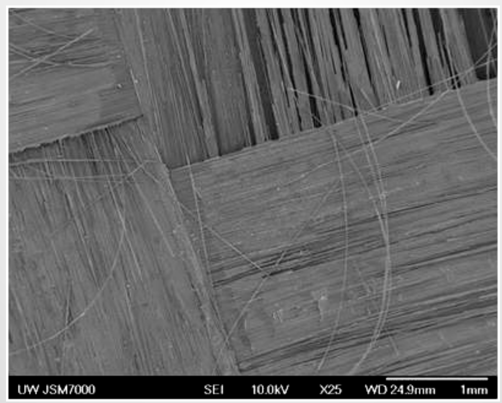
Peel Test

Rapid Adhesion Test Results

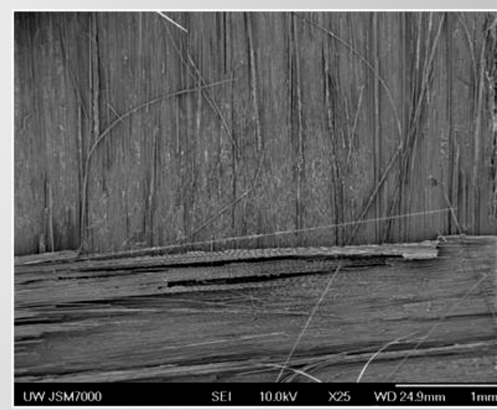
	Surface Preparation		
Substrate	**As- Tooled polyester**	**As- Tooled nylon**	**Sanded**
Uni Carbon	MIXED	COHESIVE/INTERLAMINAR	COHESIVE/INTERLAMINAR
Carbon Fabric	MIXED	COHESIVE/INTERLAMINAR	COHESIVE/INTERLAMINAR
Glass Fabric	MIXED	COHESIVE/INTERLAMINAR	COHESIVE/INTERLAMINAR



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
- Substrate reinforcement did not influence failure mode

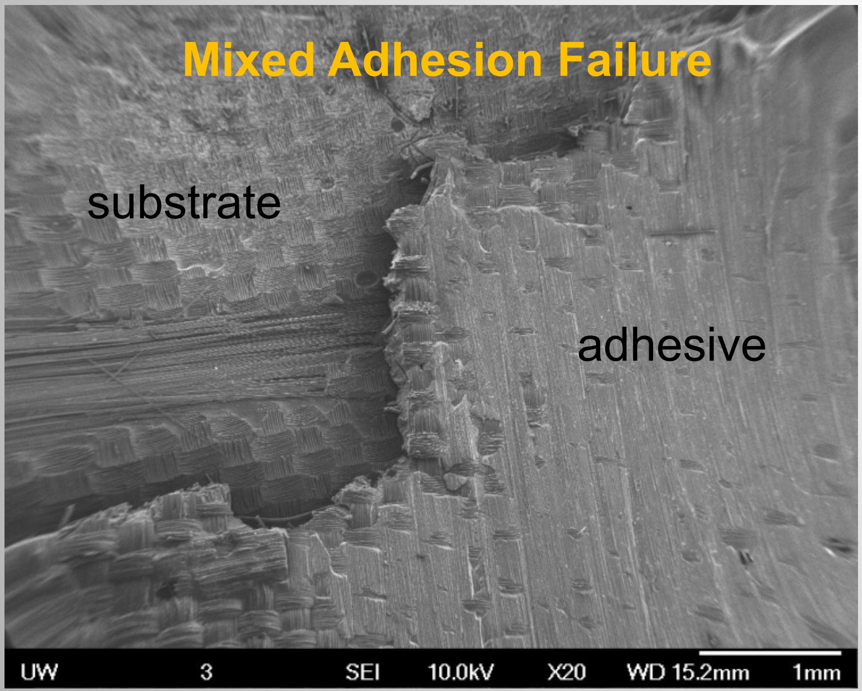
Fracture Energy and Durability

- Based on RAT results, DCB tests conducted only on Carbon Fabric Prepreg
- Dry DCB specimens exhibited same fracture modes as RAT specimens
- Durability evaluated by Hot Wet conditioning (14 days, 160F)
- Hot Wet conditioning changed fracture mode significantly for as tooled nylon, but not fracture energy

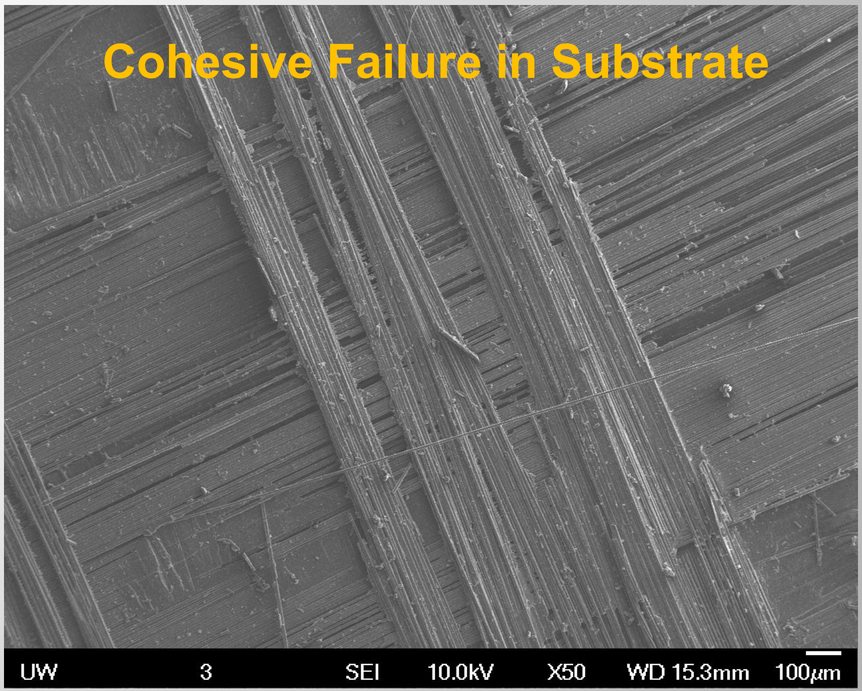
	Surface Preparation		
Condition	**As- Tooled Polyester**	**As- Tooled Nylon**	**Sanded**
Dry	**1.3 in-lbs** MIXED	**4.8 in-lbs** COHESIVE/INTERLAMINAR	**5.7 in-lbs** COHESIVE/INTERLAMINAR
Hot Wet	**1.4 in-lbs** MIXED	**4.7 in-lbs** MIXED	**5.2 in-lbs** COHESIVE/INTERLAMINAR

Durability- Hot Wet

- Nylon peel ply substrates changed fracture mode-but not G1C!
- Sanded and polyester peel ply samples fracture energy and mode not affected by hot wet conditioning



As Tooled Nylon



Sanded

Discussion of Findings

- Polyester peel ply remnants left on substrate surfaces
- No nylon peel ply remnants seen on substrates surfaces
- Polyester surfaces had a greater dispersive character
- Sanding removed peel ply imprint and remnants
- Sanding created equivalent surface energies
- Sanded surfaces bonded well
- As tooled polyester peel ply substrates did not bond well
- As tooled nylon peel ply surfaces bonded well
- Sanded surfaces had greatest durability (after hot wet)
- **As tooled nylon peel ply samples changed fracture mode after hot wet conditioning- durability needs further investigation**

Conclusions

- Fiber type had little effect on surface chemistry, surface preparation and bond quality.
- Wettability envelopes illustrated the different surface characteristics produced by the surface preparations.
- Wettability envelopes may have a role in assessing surface preparation.
- Good bonds were produced with the Toray AGATE materials and the Henkel EA 9696 adhesive with the proper surface preparation.
- Hot Wet conditioning revealed differences in bonding between sanded and nylon peel ply surfaces.
- Sanding appears to be the most reliable surface preparation for bonding Toray 2510 composites.

Acknowledgements

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- FAA JAMS Center of Excellence

Feedback and Discussion

- **Change in failure mode (cohesive to adhesion) after hot wet conditioning-**
  - **New to me—anyone else seen this?**

- Questions?
- Comments?

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