Improving Adhesive Bonding Through Surface Characterization

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University of Washington

Improving Adhesive Bonding Through Surface Characterization

Motivation and Key Issues

- Most important step for bonding is SURFACE PREPARATION!
- Inspect the surface prior to bonding to ensure proper surface prep

Objective

- Develop quality assurance (QA) techniques for surface prep

Approach

- Investigate surface preps, process variables

FAA Sponsored Project Information

Principal Investigators & Researchers
- Brian D. Flinn (PI)
- Ashley C. Tracey (PhD student, UW-MSE)
- David Pate (MS student, UW-MSE)
- Jonathan T. Morasch (undergraduate, UW-MSE)

FAA Technical Monitor
- Curt Davies

Other FAA Personnel Involved
- Larry Ilcewicz

Industry Participation
- Toray Composites
- Precision Fabrics, Richmond Aerospace & Airtech International
- The Boeing Company (Marc Piehl, Kay Blohowiak, Will Grace, Tony Belcher, Pete VanVoast, Liz Castro, John Osborne)

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2012-2013 Statement of Work

	Surface Characterization/QA Technique			
	Contact Angle		**FTIR**	
	Goniometer	**Surface Analyst**	**DATR**	**Diffuse Reflectance**
Cure Temp and Dwell Time	✔	✔	---	In progress
Peel Ply Prep	✔	✔	✔	✔
Si Contaminants	✔	✔	✔ (Boeing)	
Peel Ply Orientation	✔	✔ No effect	N/A	In progress
Peel Ply + Abrasion	✔		---	✔
Scarfed/Sanded Surfaces	In Progress	In progress	---	In progress
Effect of Measurement on Bonding Surface	✔	TBD	TBD	N/A
Sandpaper Type	✔		---	In progress

✔ = work completed --- = not of focus, diffuse reflectance for rough surfaces

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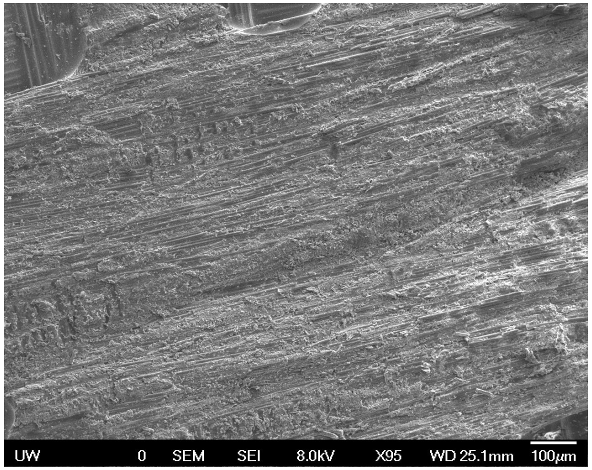
Scarfed/Sanded Surfaces

Motivation: examine surfaces prior to bonding to ensure surface was properly abraded

Need to understand variables that could affect QA measurements to develop robust process

- Reinforcement fiber orientation
- Fiber type
- Resin type
- Fiber arrangement (tape vs. fabric)
- Type of sandpaper
- Amount of sanding



Electron micrograph of sanded composite surface

Experimental Overview: Scarfed/Sanded Surfaces

Investigate variables that could affect contact angle measurements on scarfed or sanded surfaces

Abrade various composite surfaces and measure contact angle (CA) of multiple fluids

CA variables

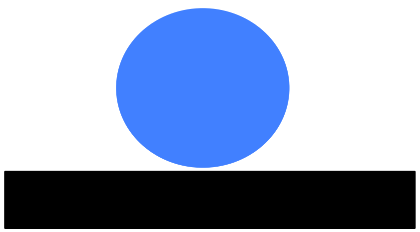
- Reinforcement fiber orientation
- Fiber arrangement (tape vs. fabric)

Surface Energy to Examine Surfaces

Adhesive must wet substrate – controlled by surface energy

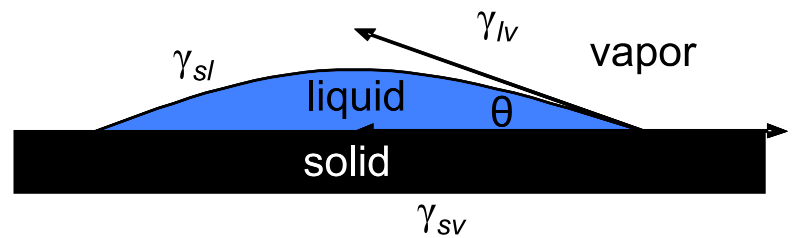
Surface energy = measure of energy associated with unsatisfied bonds at the surface [free energy/unit area]

CAs used to measure surface energy



Non-wetting, θ = 180°

Wetting, θ = 0°



$$\gamma_{sl} = \gamma_{sv} - \gamma_{lv} \cos\theta$$

Historically: water break test for metal bond QA, not sufficient for composites – esp. peel ply material

- Need multiple fluids to determine surface energy, wettability envelopes

Materials and Process

4-ply composite laminates (autoclave cure)

- Toray T800/3900 unidirectional (350 °F)
- Toray T800/3900 fabric (350 °F)
- Cycom 97714A/T300 fabric (250 °F)
- Cycom 970/T300 fabric (350 °F)
- Cytec MXB7701-GF fabric (250 °F)

Sanding surface preparation

- Orbital sander, 120 grit Al_2O_3 sanding pads
- Acetone wipe, double wipe method

Contact angle analysis

- Sessile drop method
- Fluids: deionized water (DI H_2O), ethylene glycol (EG), diiodomethane (DIM), glycerol (GLY)

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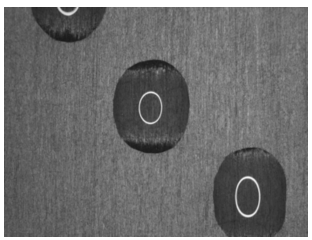
Reinforcement Fiber Orientation

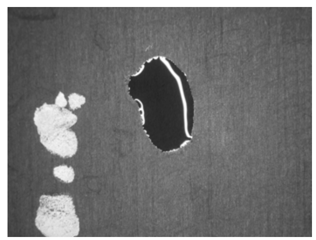
Tape laminates (Toray T800/3900)

- Drops elongated along fiber direction

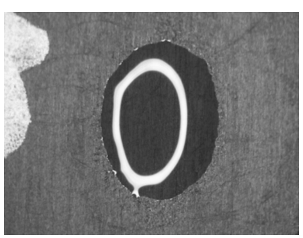
Fiber Direction



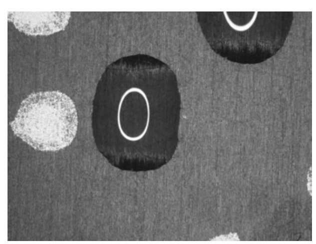
DI H2O



DIM



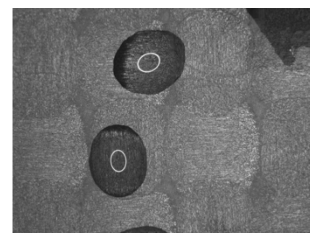
EG



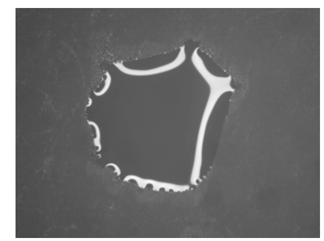
GLY

Fabric laminates

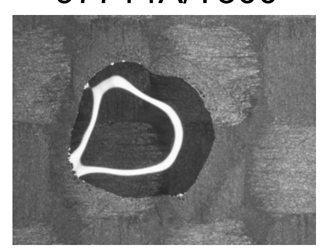
- Drop shape is circular with amorphous edges



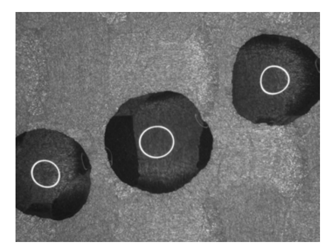
DI H_2O on Cycom 97714A/T300



DIM on Cytec MXB7701-GF



EG on Toray T800/3900



GLY on Cycom 970/T300

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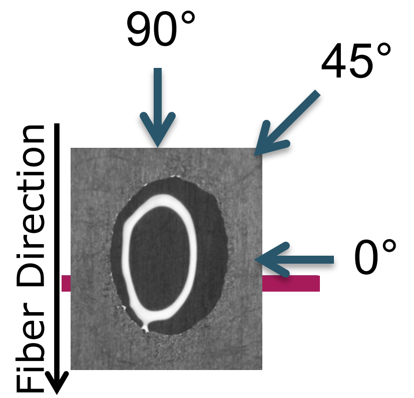
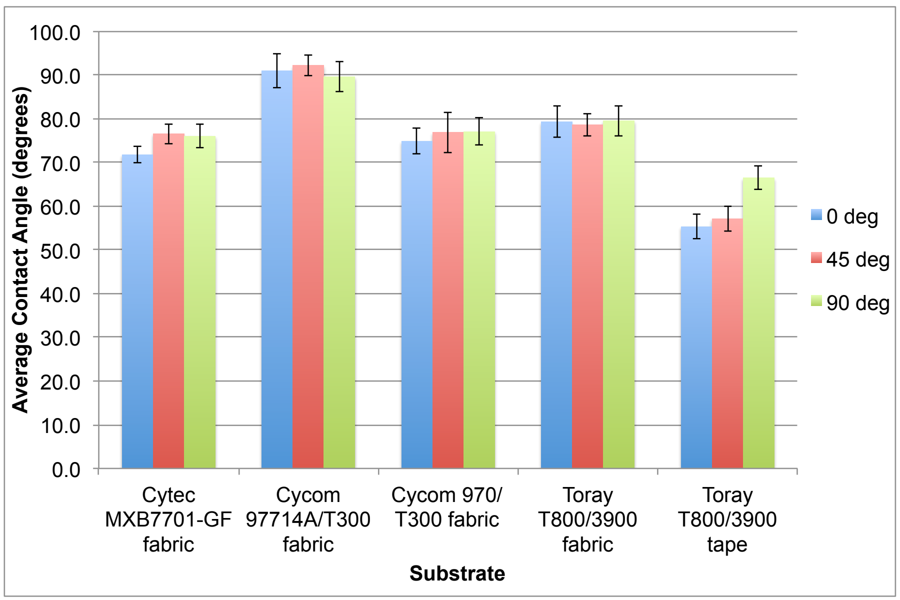
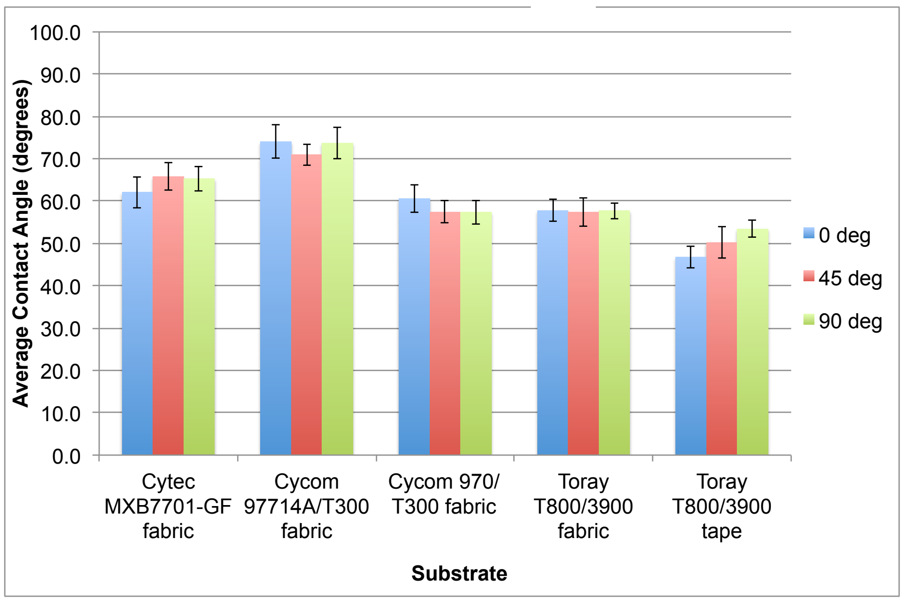
Reinforcement Fiber Orientation



DI H_2O



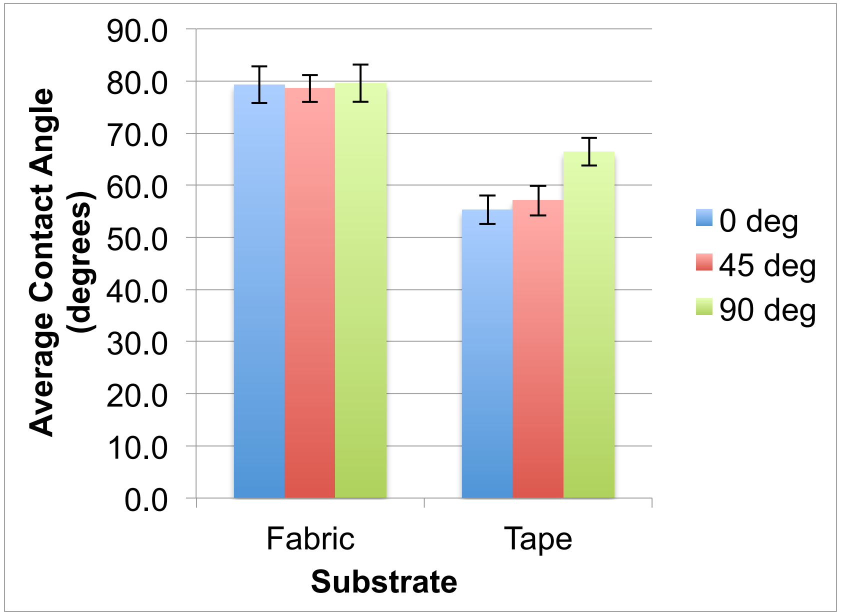
GLY



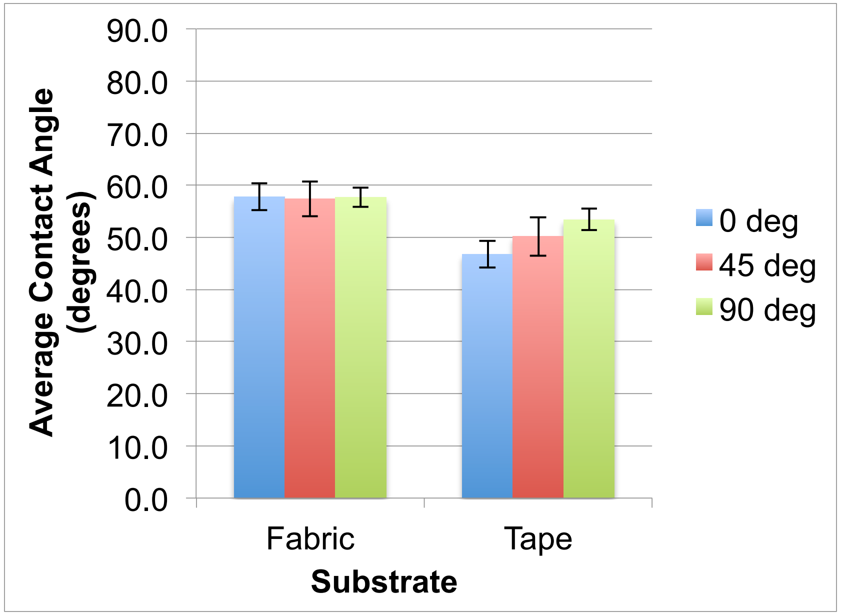
- DI H_2O and GLY CAs measured on tape surfaces higher at 90° orientations
  - Measure CAs at 90° for most conservative measurement
- Fabric surfaces do not show trend due to fiber orientation
- DIM and EG CAs not significantly different and low (most CAs ≤20°)

Tape vs. Fabric

DI H_2O



GLY



DI H_2O and GLY CAs on Toray T800/3900 tape surfaces lower than on fabric surfaces

- Due to resin content on fabric surface? Epoxy lower surface energy than CF

EG and DIM CAs on Toray T800/3900 tape and fabric surfaces not significantly different but very low (<20°)

Summary: Scarfed/ Sanded Surfaces

Fiber Orientation:

- DI H_2O and GLY CAs are a function of fiber orientation on tape surfaces
    - Measure CAs at 90° for most conservative measurement
- Fabric surfaces do not show trend due to fiber orientation

Tape vs. Fabric:

- DI H_2O and GLY CAs on Toray T800/3900 tape surfaces lower than on fabric surfaces
    - Due to resin on fabric surface?

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Peel Ply + Abrasion

Motivation: examine surfaces prior to bonding to ensure removal of peel ply texture

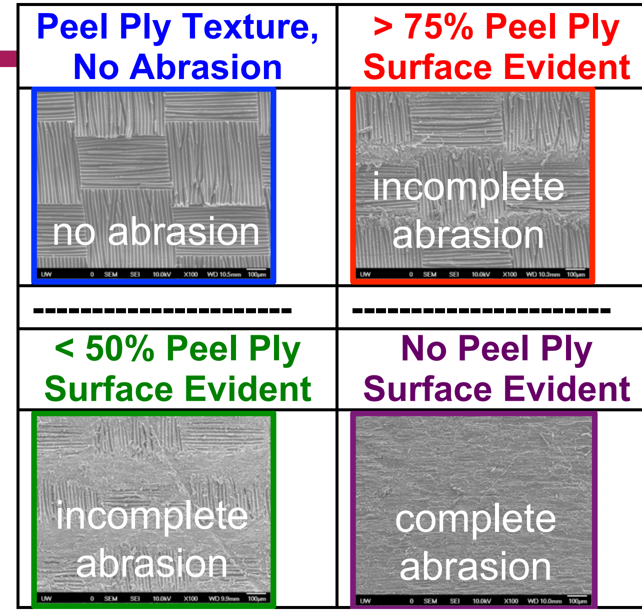
- Application: bonding with paste adhesives

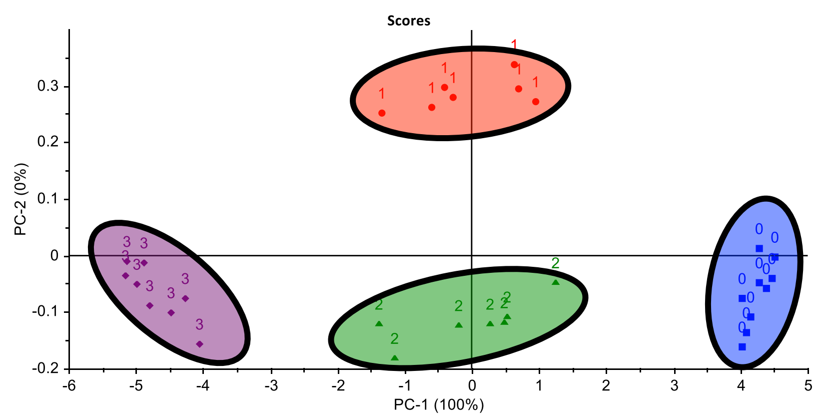
Variables:

- peel ply type before abrasion
- directional vs. random abrasion
- amount of peel ply texture removed

Diffuse reflectance FTIR can detect complete vs. incomplete abrasion to remove peel ply surface

- ➢ Correlate to bond quality?





Experimental Overview: Peel Ply + Abrasion

Investigate effect of complete vs. incomplete sanding to remove peel ply texture on bond quality

Three surface preparation conditions

- No sanding/peel ply surface
- Incomplete sanding/ <50% peel ply texture evident
- Complete sanding/no peel ply texture evident

Fabricate bonded specimens (bond within 4 hours)

- Double Cantilever Beam (DCB) Test
  - Mode I strain energy release rate (G_{IC})
  - Failure mode

Materials and Process

Toray T800/3900 unidirectional laminates

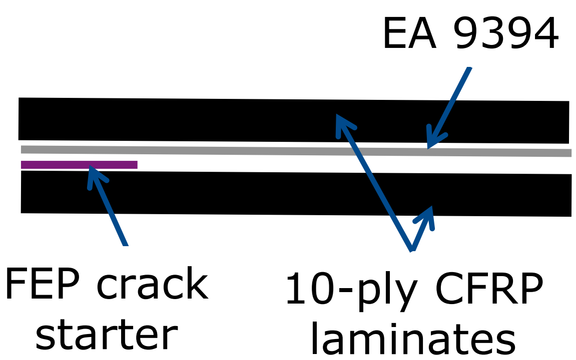
- Autoclave cure (350 °F, 89 psi)

Surface Preparation

- Precision Fabrics Group 60001 polyester peel ply
- Orbital sander, Merit 180 grit Al_2O_3
- Acetone wipe, double wipe method

Secondary Bonding

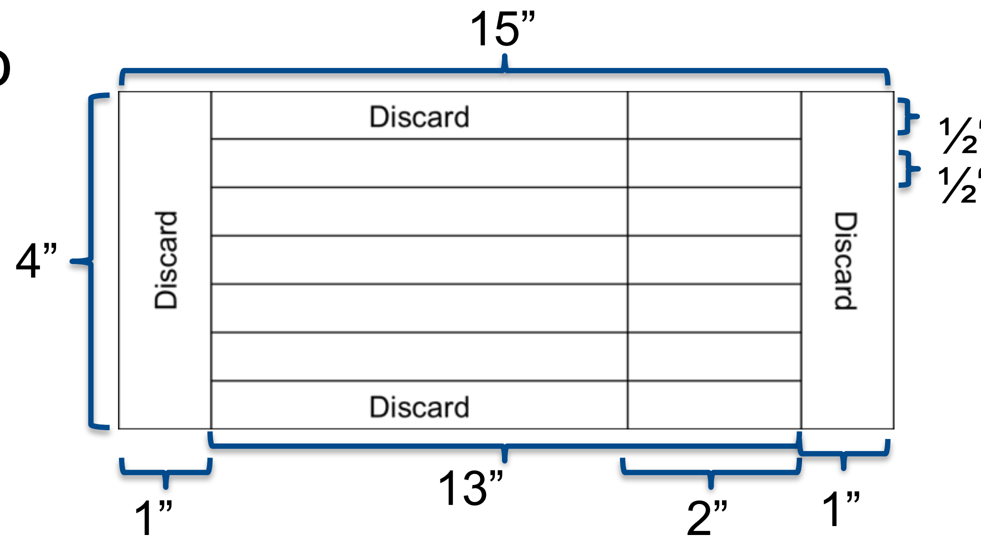
- Henkel EA9394 paste adhesive
- Autoclave cure (150 °F, 25 psi)
- Bondline thickness range: 11.3 ± 1.5 mils



DCB Test

Bonded panels cut into (8) ½" x 13" specimens

Used area method



$$G_{IC} = \frac{E}{A \times B}$$

- E: area of curve
- A: crack length
- B: specimen width

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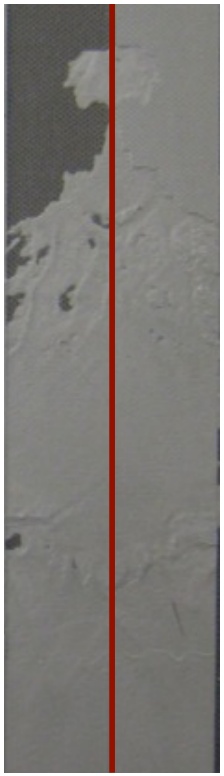
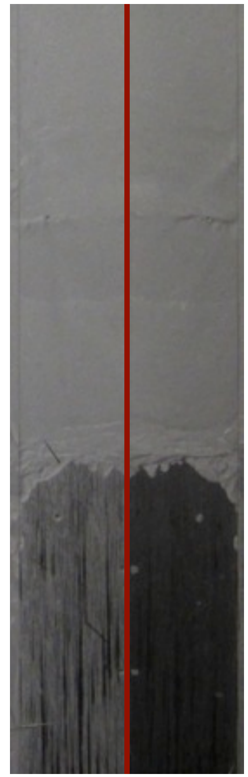
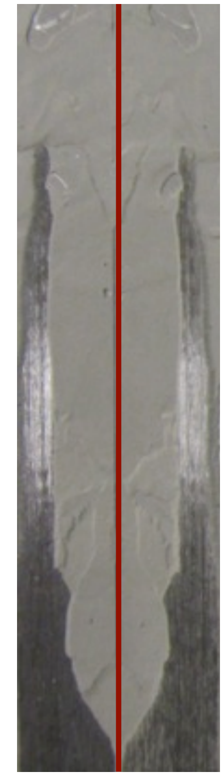
Failure Modes



Peel Ply Surface: mostly cohesive, some adhesion

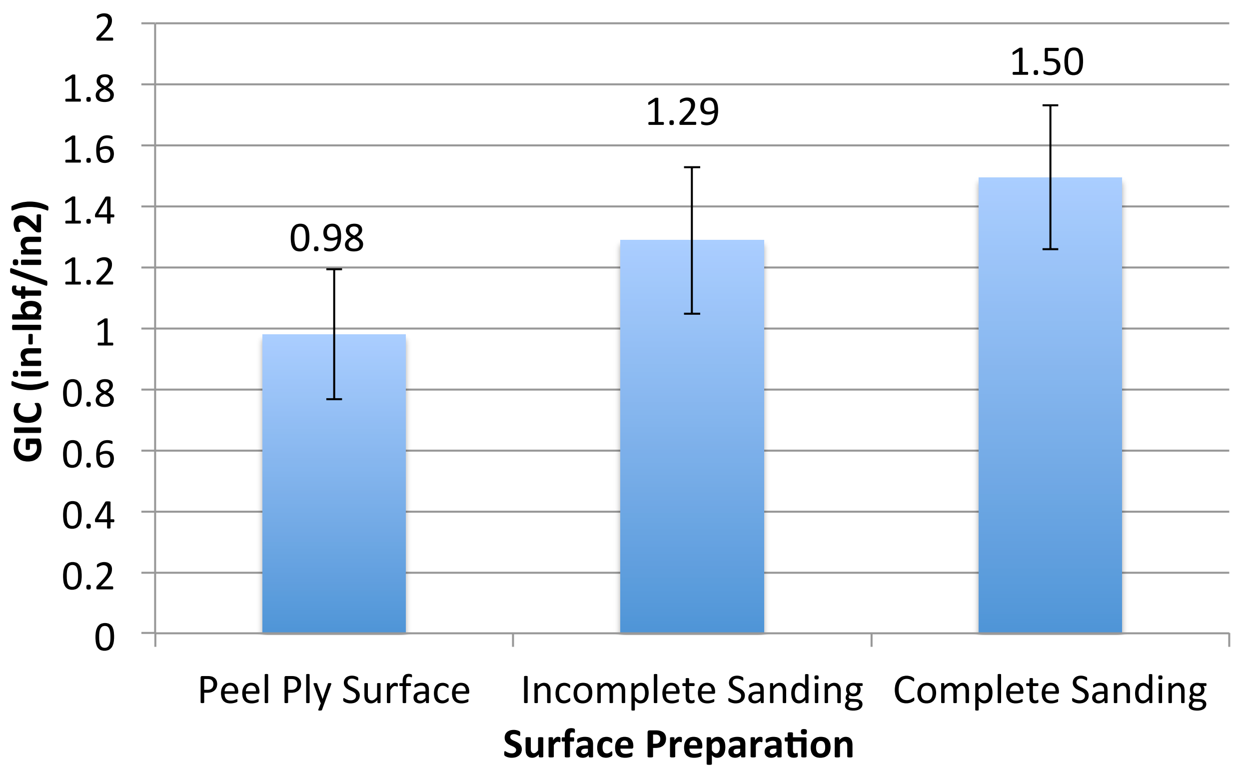


Incomplete Sanding: mostly cohesive, some interlaminar, adhesion



Complete Sanding: mostly cohesive, some interlaminar

Mode I Strain Energy Release Rate



Fracture energies highest for sanded surfaces and lowest for peel ply surfaces

Summary: Peel Ply + Abrasion

FTIR can detect complete vs. incomplete abrasion to remove peel ply texture

Samples with peel ply texture present before bonding showed some adhesion failure

Samples without peel ply texture present before bonding only showed acceptable failure modes (cohesive, interlaminar)

Fracture energy highest for sanded adherends and lowest for peel ply prepared adherends

Large bondline thickness variation

- Hot press

Future Work

Investigate effect of sanding variables on QA methods

- CA, include Brighton Surface Analyst (ballistic drop deposition)
- FTIR

Quantification of proper vs. improper sanding to remove peel ply texture

- Only one level of abrasion to remove peel ply texture examined with DCB test

Looking Forward

Benefit to Aviation

- Guide development of QA methods for surface preparation
- Greater confidence in adhesive bonds

Future needs

- Application to other composite/surface prep/adhesive systems
- Model to guide bonding based on characterization, surface preparation and material properties
- QA methods to ensure proper surface for bonding

Acknowledgements

FAA, JAMS, AMTAS

Boeing Company

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Precision Fabrics Group

Richmond Aircraft Products

Airtech International

UW MSE

Thank you!

Questions?