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Potential Composite Bond Contamination By Contact Angle Fluids

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Materials Science and Engineering

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
  - ➢ Effect of measurements on bonding surface

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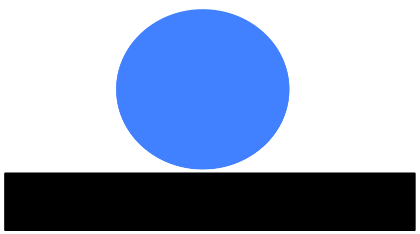
FAA Sponsored Project Information

- Principal Investigators & Researchers
  - Brian D. Flinn (PI)
  - Ashley C. Tracey (PhD student, UW-MSE)
  - Jonathan T. Morasch (undergraduate, UW-MSE)
  - Aaron Capps (UW-MSE)
- FAA Technical Monitor
  - David Westlund
- Other FAA Personnel Involved
  - Larry Ilcewicz
- Industry Participation
  - Toray Composites
  - Precision Fabrics, Richmond Aerospace & Airtech International
  - The Boeing Company (Marc Piehl, Kay Blohowiak, Pete VanVoast, Will Grace, Tony Belcher, Liz Castro)

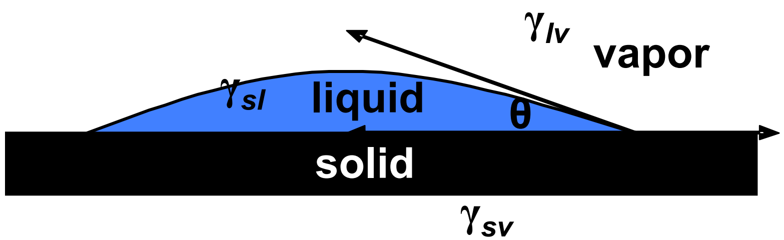
Surface Energy to Examine Surfaces

- 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°

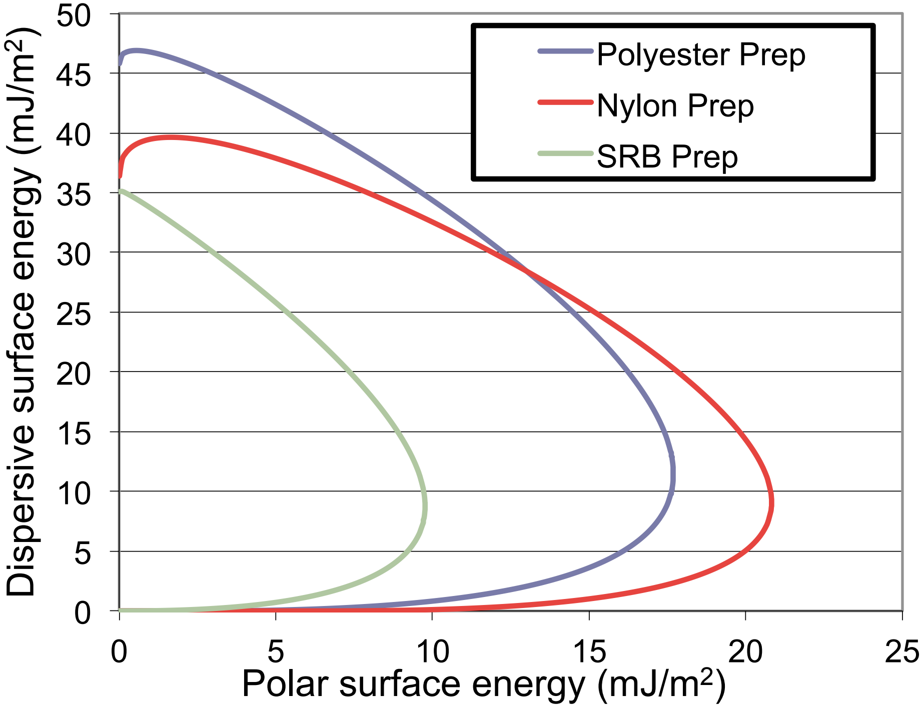


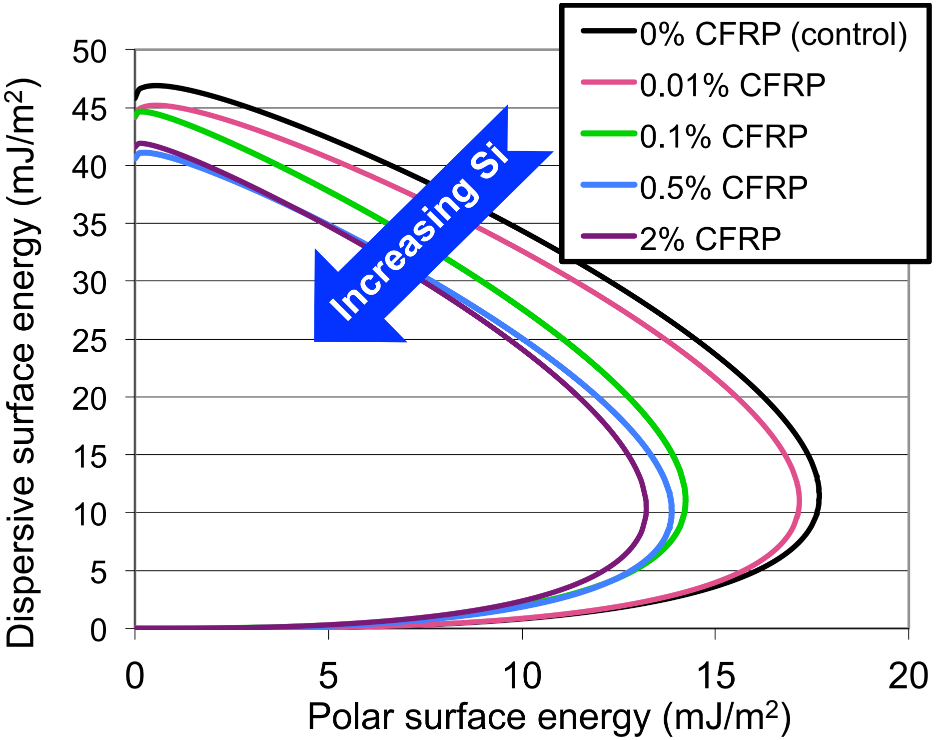
$$\gamma_{sl} = \gamma_{sv} - \gamma_{lv} \cos\theta$$

- 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

Contact Angle to Detect Surface Prep

- CA can detect surface prep and silicone contamination
  - Wettability envelopes: 2D representation of surface energy





➢ Need to understand how fluid affects bonding surface

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Experimental Overview

Investigate effect of CA fluid application on prepared composite surfaces and resulting bond quality

- Apply CA fluid on prepared CFRP surfaces followed by use of one of below methods:
  1. Dry wipe – fluid on surface 10-20 min
  2. Acetone wipe – fluid on surface 10-20 min
  3. Air dry (in fume hood) – fluid on surface 30 min-3 hr
  - Note: amount of fluid applied to surfaces much larger than would typically be exposed to in QA situations (100% vs <5% coverage)
- Fabricate Double Cantilever Beam (DCB) test specimens (bond within 4 hours of application)
  - Mode I strain energy release rate (G_{IC}) and failure mode

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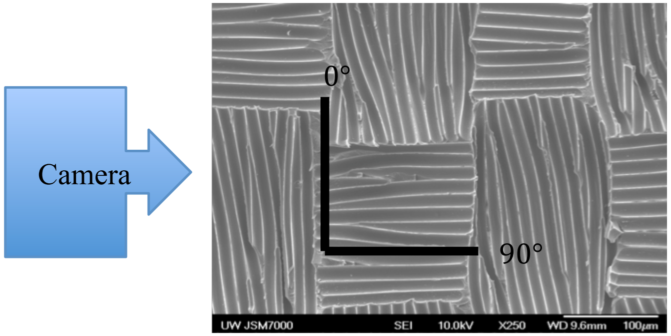
Materials and Process

- Toray 3900/T800 unidirectional laminates
  - Autoclave cure (350 °F, 89 psi)
- Peel ply surface prep
  - Precision Fabric Group 60001 polyester peel ply
- Contact angle fluid application
  - Fluids: DI H_2O, ethylene glycol (EG), glycerol (GLY), diiodomethane (DIM)
  - DuPont Sontara aerospace grade wipes
    - Application and removal of CA fluid

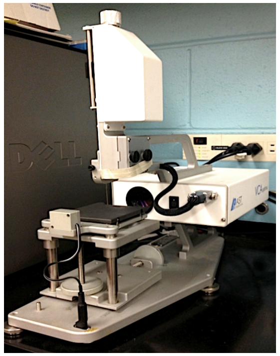
Materials and Process – CA Measurement

- Measure CAs of 1 µL sessile drops from side view using goniometer
  - 10 drops (20 CAs) per fluid
- Fluids: DI H_2O, EG, GLY, DIM
- Measure at 0 or 90° wrt peel ply texture

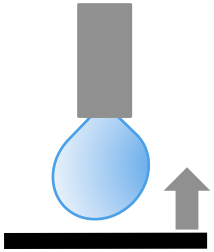


- Calculate CFRP surface energy from CAs

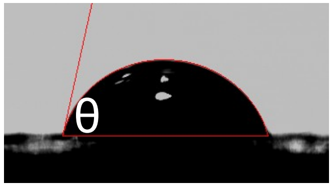
$$\frac{\gamma_{lv}(\cos\theta+1)}{2\sqrt{\gamma_{lv}^{p}}} = \sqrt{\gamma_{sv}^{d}}\left(\sqrt{\frac{\gamma_{lv}^{d}}{\gamma_{lv}^{p}}}\right)+\sqrt{\gamma_{sv}^{p}}$$



VCA Optima Goniometer



Drop application: dispense drop, raise surface



Side-view of drop as viewed from goniometer camera

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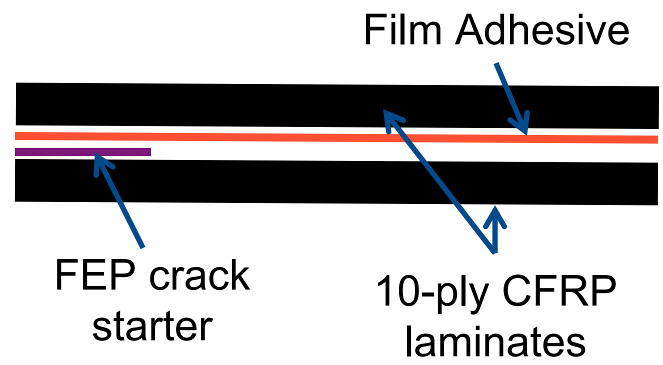
Materials and Process – DCB Testing

- AF 555M film adhesive
  - Aerial weight: 0.050 ± 0.005 lb/ft^2 [1]
  - Autoclave cure (350 °C, 89 psi)
  - Bondline thickness: 4.1 - 12.6 mils
- MB 1515-3M film adhesive
  - Aerial weight: 0.05 lb/ft^2 [2]
  - Autoclave cure (350 °F, 45 psi)
  - Bondline thickness: 7.4 - 11.8 mils



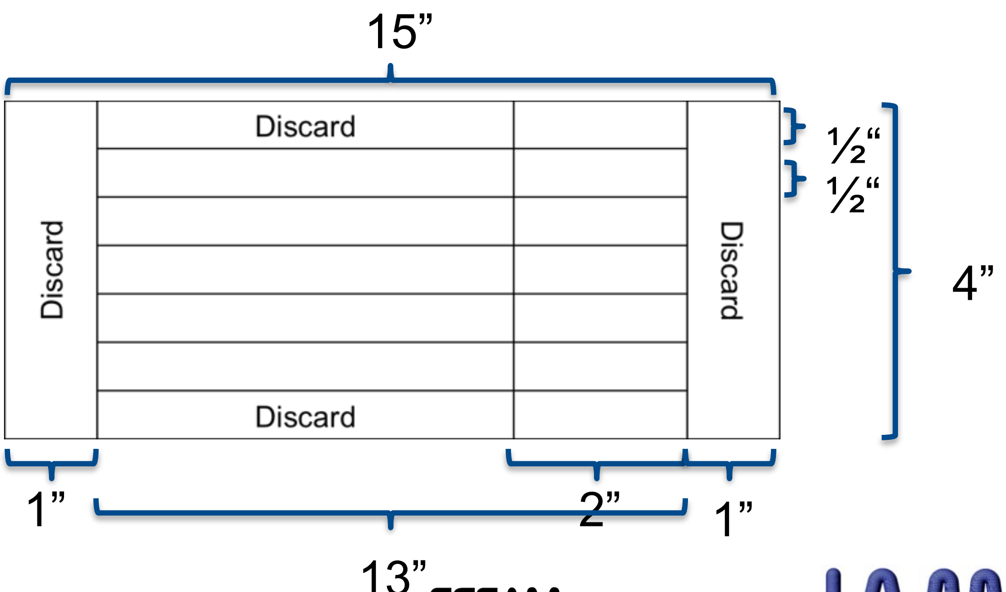
[1] “3M Scotch-Weld Structural Film Adhesive AF 555 Technical Data Sheet.” *3M Aerospace and Aircraft*. N.p., Oct 2007. Web. 4 Mar 2013. <http://www.3M.com/aerospace>.

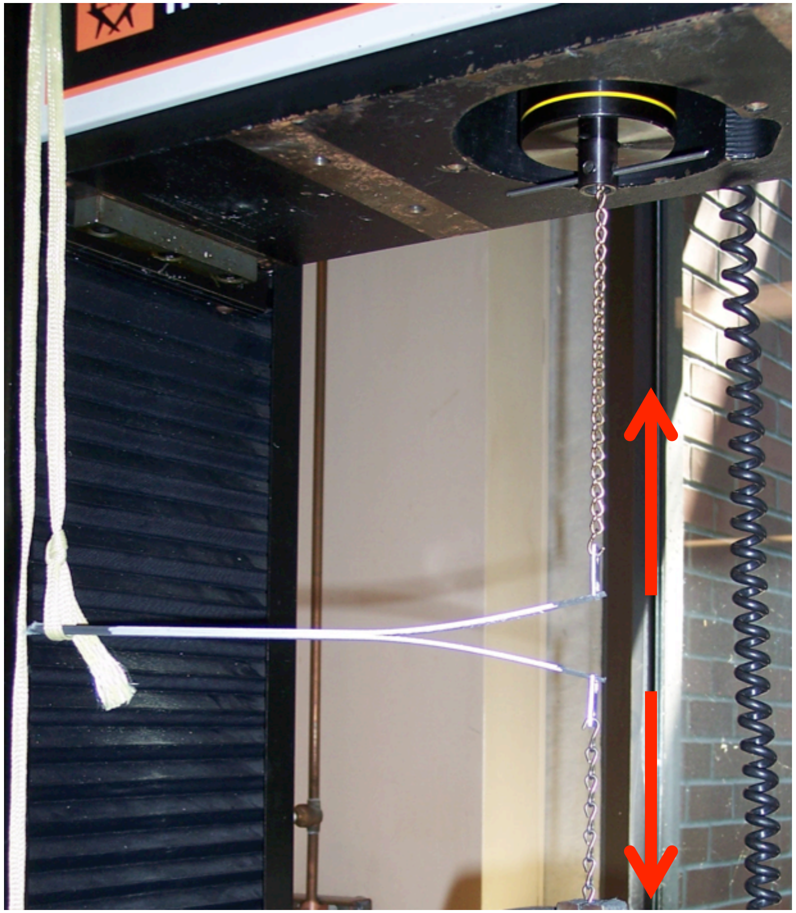
[2] “Cytec Metlbond 1515-3 Film Adhesive Technical Data Sheet.” *Cytec Engineered Materials*. N.p., 12 Aug 2010. Web. 8 Mar 2013. <http://www.cytec.com/>.

Materials and Process – DCB Testing

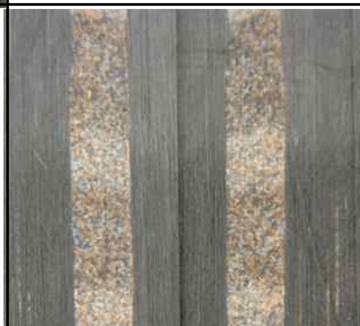
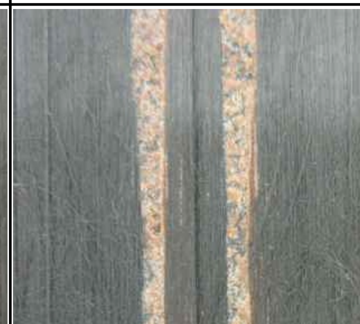
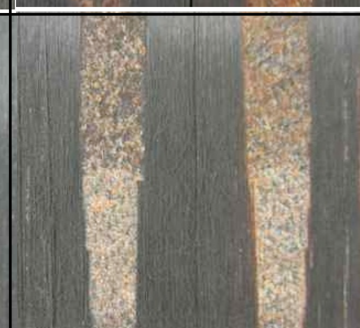
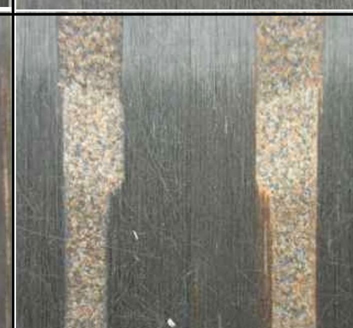
- Bonded panels cut into (5) ½" x 13" specimens
- Used area method
  - E: area of curve
  - A: crack length
  - B: specimen width

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

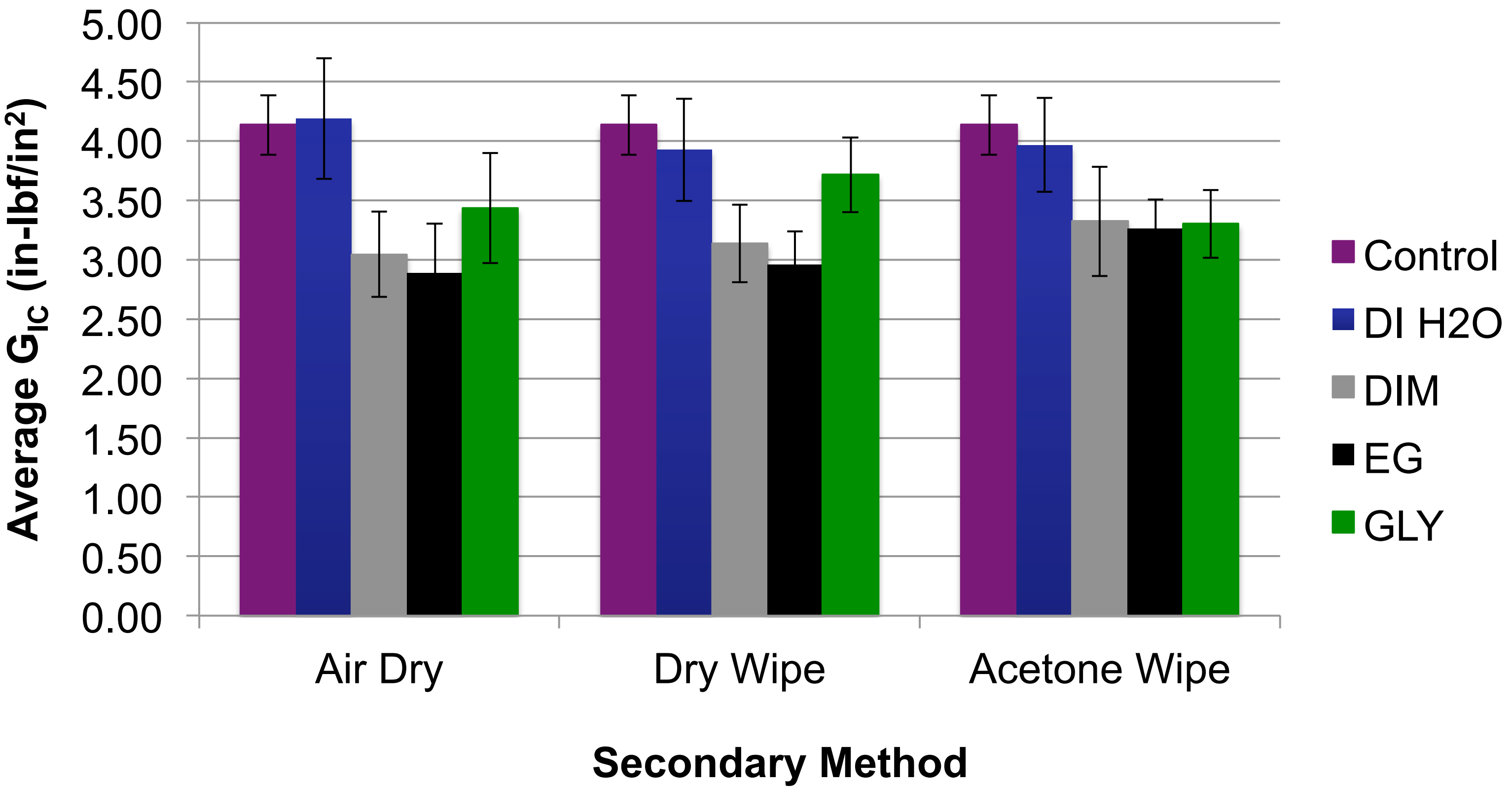




DCB Failure Modes – AF 555M Adhesive

Control 	DI H_2O	DIM	EG	GLY
Air Dry				
Dry Wipe				
Acetone Wipe				

DCB Mode I Strain Energy Release Rates – AF 555M Adhesive



- DI H_2O did not degrade G_{IC}
- DIM and EG decreased G_{IC} 20-30%
- GLY decreased G_{IC} 10-20%

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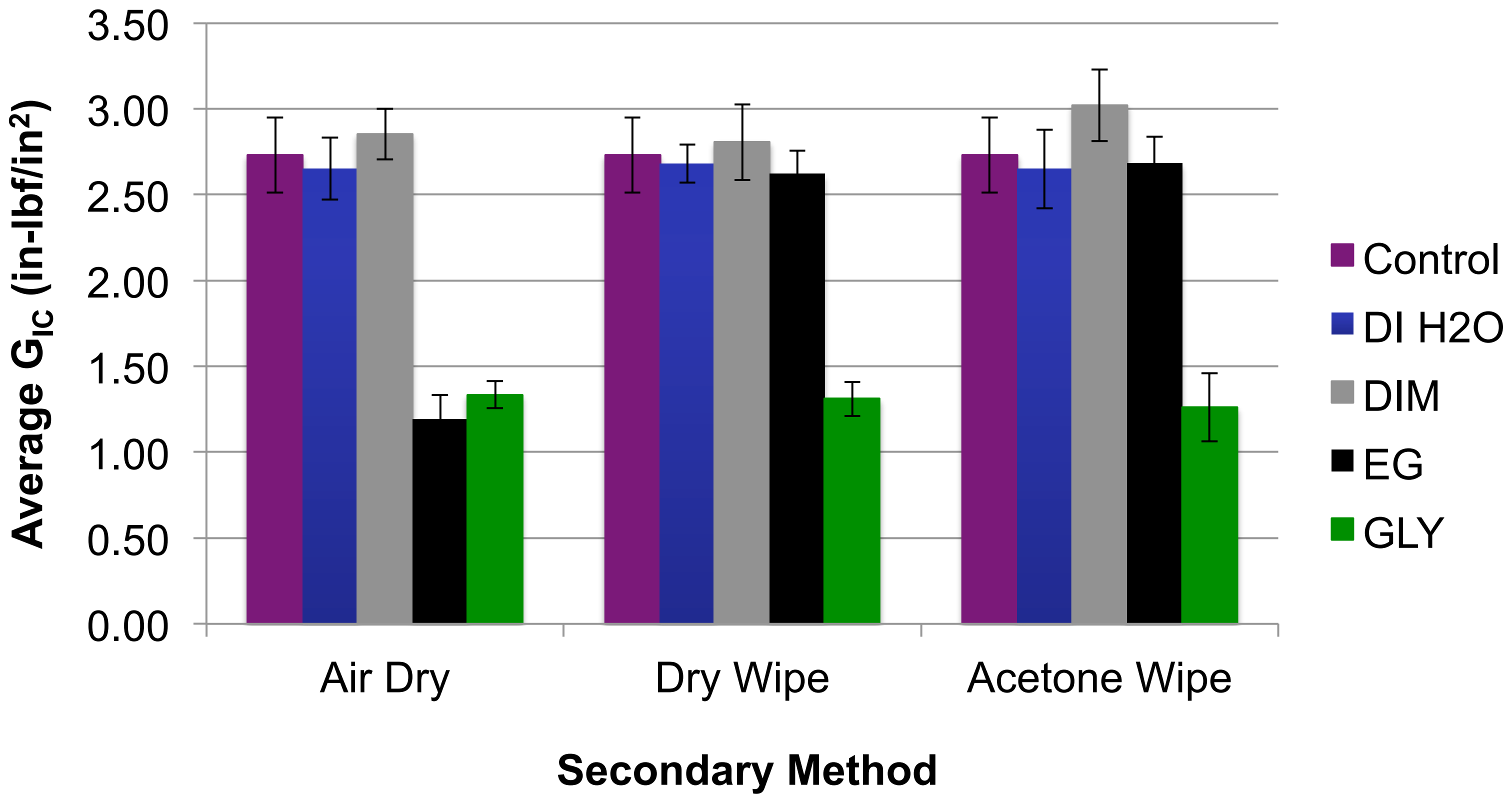
DCB Observations – AF 555M Adhesive

- DI H_2O did not degrade failure mode or G_{IC} compared to control samples
- DIM did not change failure mode but decreased G_{IC} 20-30%
  - Interaction of DIM with substrate and/or adhesive?
- EG decreased G_{IC} 20-30%
  - EG + Air Dry and EG + Dry Wipe mostly interlaminar failure → may explain decrease
  - EG + Acetone Wipe similar failure to control samples
- GLY decreased G_{IC} 10-20%
  - Unexpected as fracture mode mostly cohesive
  - Interaction between GLY and substrate and/or adhesive?

DCB Failure Modes – MB 1515-3M Adhesive

Control 	DI H_2O	DIM	EG	GLY
Air Dry				
Dry Wipe				
Acetone Wipe				

DCB Mode I Strain Energy Release Rates – MB 1515-3M Adhesive



- DI H_2O and DIM did not significantly change G_{IC}
- EG showed variable results
- GLY decreased G_{IC} 50-55%

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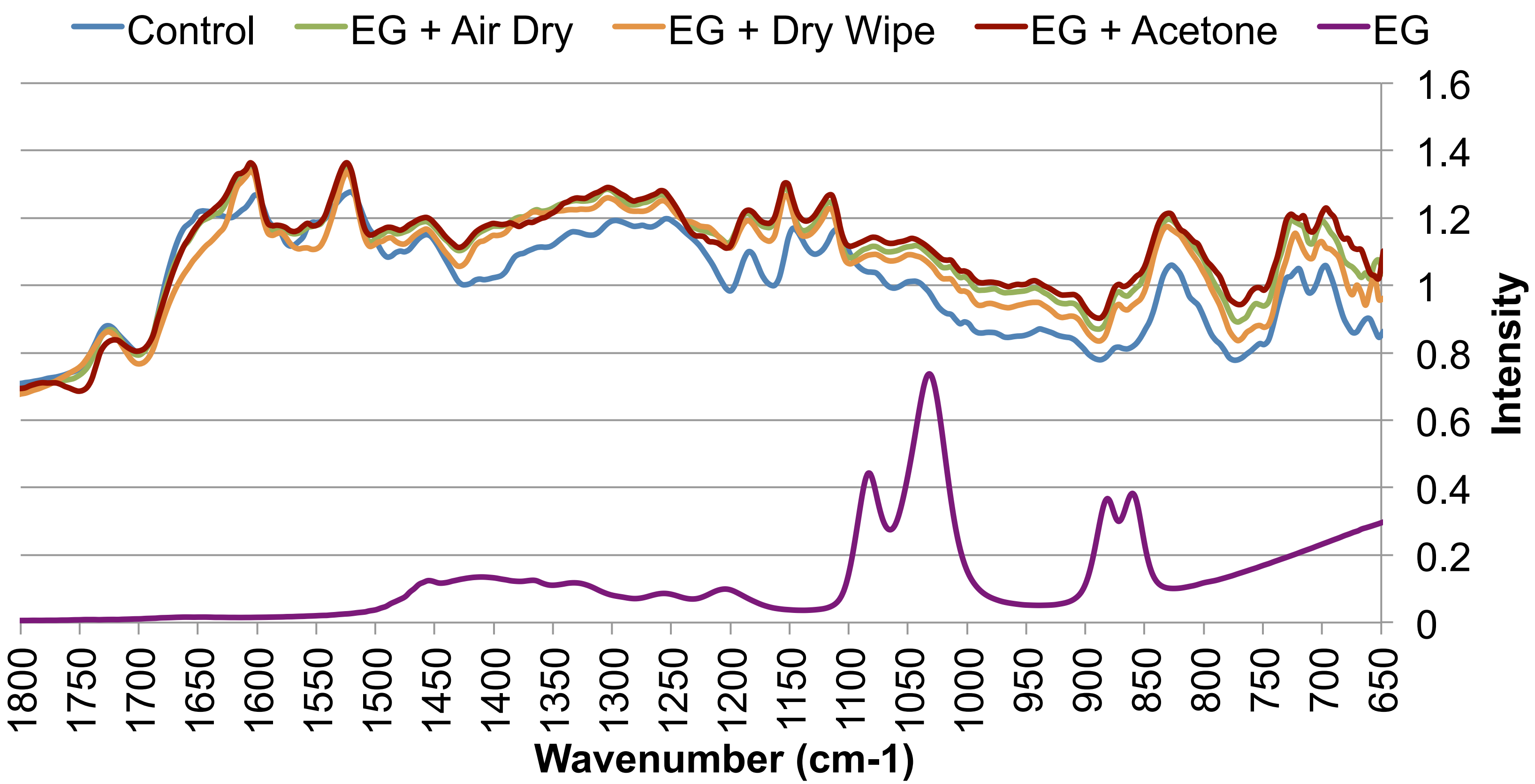
DCB Observations – MB 1515-3M Adhesive

- DI H_2O and DIM did not degrade failure mode or G_{IC} compared to control samples
- EG showed variable fracture surfaces and G_{IC} measurements
  - FTIR or CA detect differences?
- GLY decreased G_{IC} 50-55%
  - Unexpected as fracture mode mostly cohesive
  - Interaction between GLY and substrate and/or adhesive?

Diffuse Reflectance FTIR Analysis of EG Surfaces



- Slight spectral differences between EG samples but not due to EG on surface

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GLY Fracture Surfaces

- GLY fracture surface showed significant bondline porosity compared to control and all other "contaminated" surfaces

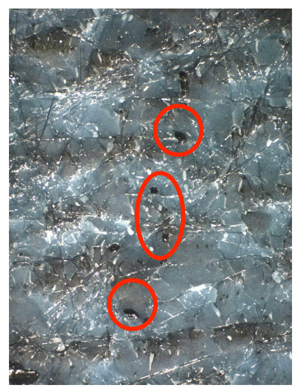
Control

GLY + Air Dry

GLY + Dry Wipe

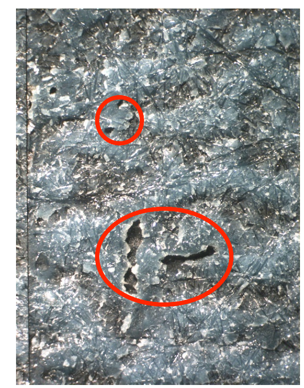
GLY + Acetone Wipe

Control



GLY + Air Dry

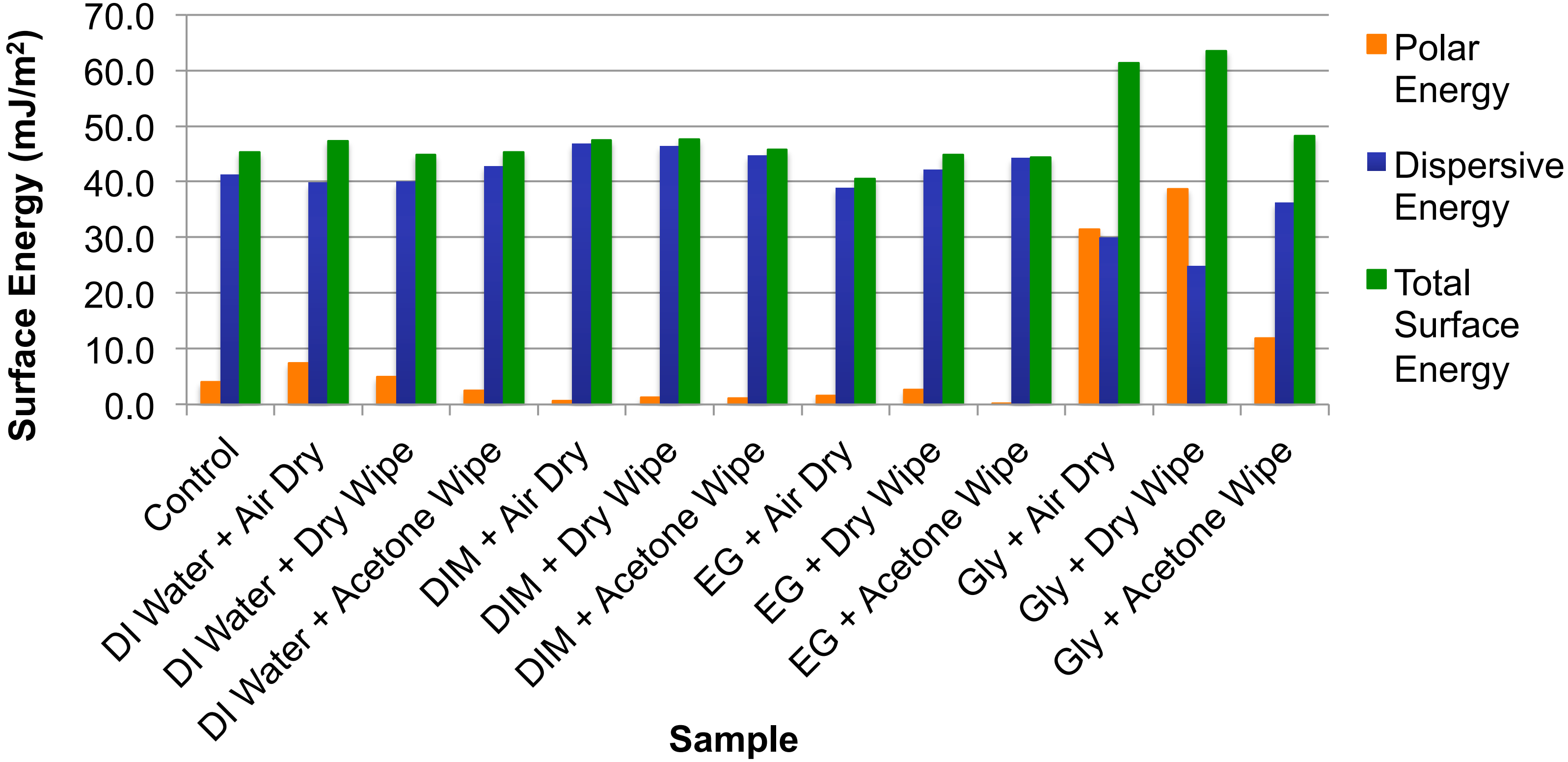
GLY + Dry Wipe



GLY + Acetone Wipe

CA Measurements

- Some CA probe fluids affect G_{IC} and fracture mode → CA analysis of "contaminated" surfaces



- DI H_2O, DIM and EG did not significantly change surface energy
- GLY showed largest difference in surface energies
  - GLY + Air Dry and GLY + Dry Wipe samples approaching surface energy of GLY itself (γ^p = 30 mJ/m^2, γ^d = 34 mJ/m^2)

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Summary

- Contact angle used to measure bonding surfaces → effect of measurement on surface?
- All DCBs showed acceptable failure modes – no adhesion failure
- Some observations of decrease in G_{IC} and change in failure mode
  - CA analysis showed surface energy differences for GLY substrates
  - More research necessary to understand other G_{IC} and failure mode differences
  - Note: amount of fluid applied to bonding surfaces much larger than would typically be exposed to in QA situations

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Looking Forward

- Benefit to Aviation
  - Guide development of QA methods for surface prep
  - Greater confidence in adhesive bonds
- Future needs
  - Application to other composite/surface prep/adhesive systems (repair, paste adhesive, etc.)
  - Model to guide bonding based on characterization, surface prep and material properties
  - QA methods to ensure proper surface for bonding

Acknowledgements

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  - Marc Piehl, Kay Blohowiak, Pete VanVoast, Will Grace, Tony Belcher, Liz Castro
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- UW MSE

Thank you!

Questions and comments welcome.

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