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# Improving Adhesive Bonding of Composites Through Surface Characterization

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# Improving Adhesive Bonding of Composites 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 and examine effect of measurements on bonding surface

# FAA Sponsored Project Information

- Principal Investigators & Researchers
  - Brian D. Flinn (PI)
  - Ashley Tracey (PhD student, UW-MSE)
  - Elise Santa Maria (undergraduate, 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, Peter VanVoast, William Grace, Tony Belcher, Liz Castro)

# 2011-2012 Statement of Work

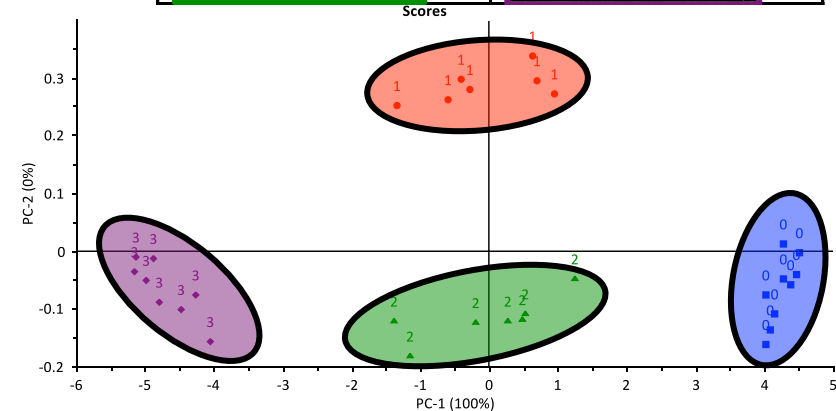
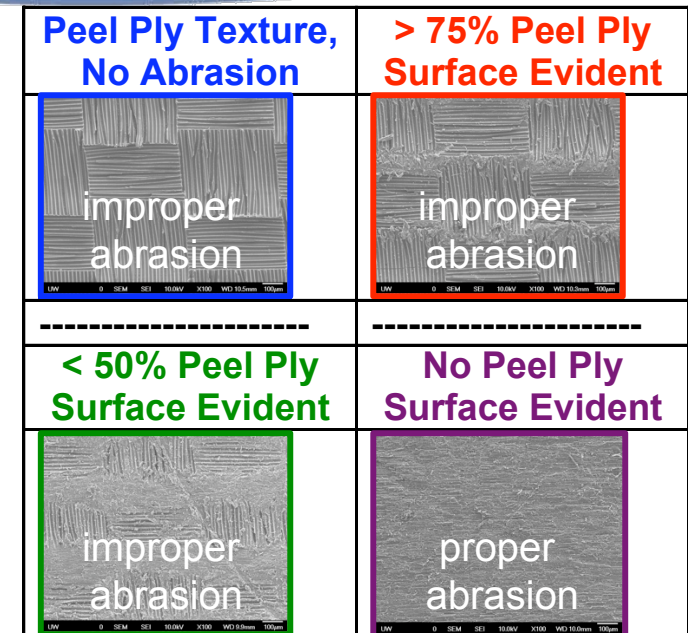
	Surface Characterization/QA Technique			
	Contact Angle		FTIR	
	Goniometer	Surface Analyst	DATR	Diffuse Reflectance
Cure Temp and Dwell Time	✓	✓	In progress	In progress
Peel Ply Prep	✓	✓	✓	✓
Si Contaminants	✓	✓	✓ (Boeing)	
Peel Ply Orientation	✓	✓ No effect	N/A	In progress
Peel Ply + Abrasion	✓		In progress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

✓ = work completed



# Recent Progress

- 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 removed
  - Diffuse reflectance FTIR can detect proper vs. improper abrasion to remove peel ply surface
    - Correlate to bond quality?

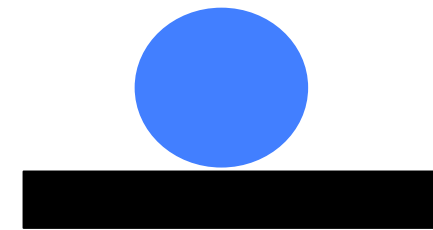


# Recent Progress

- Scarfed surfaces/repair surfaces
  - Motivation: examine repair surfaces prior to bonding to ensure proper abrasion
  - Variables: reinforcement fiber orientation, fiber type, resin type, fiber arrangement (tape vs. weave)
  - Contact angle (CA) data collected – analysis in progress
- Effect of CA fluid measurement on bond quality
  - Motivation: does measurement affect bonding surface?
  - Bond quality of intentionally contaminated samples

# Surface Energy to Examine Surfaces

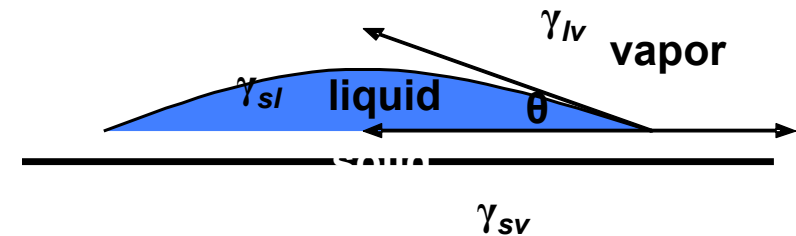
- Adhesive must wet substrate – controlled by surface energy
- Surface energy = measure of energy associated with unsatisfied bonds at the surface [free energy/unit area]
- CAs used to measure surface energy



Non-wetting,  $\theta = 180^\circ$



Wetting,  $\theta = 0^\circ$



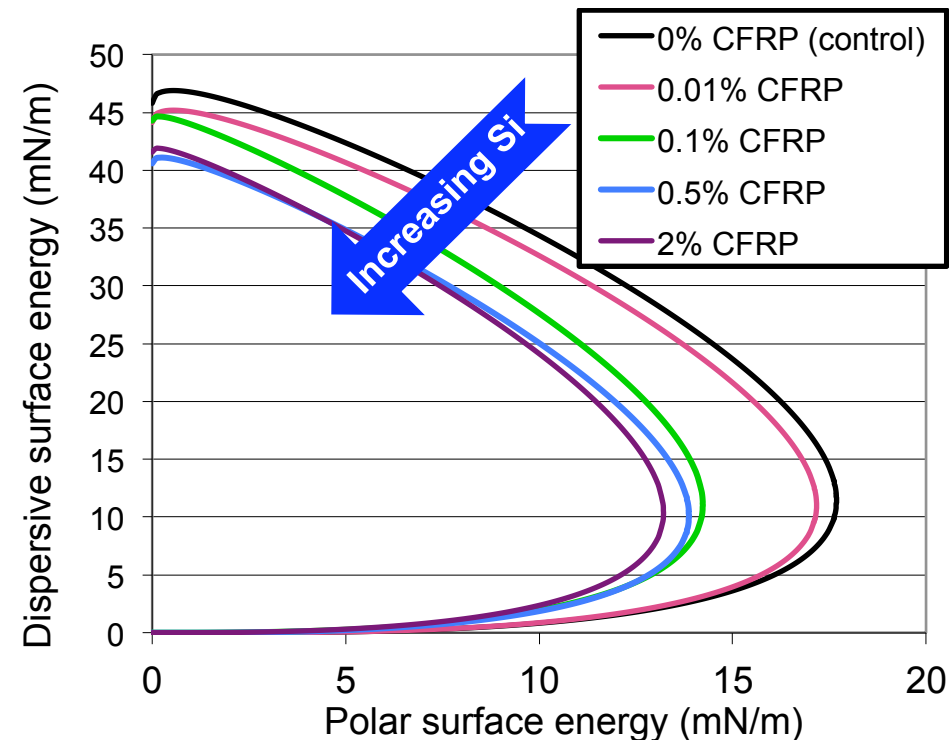
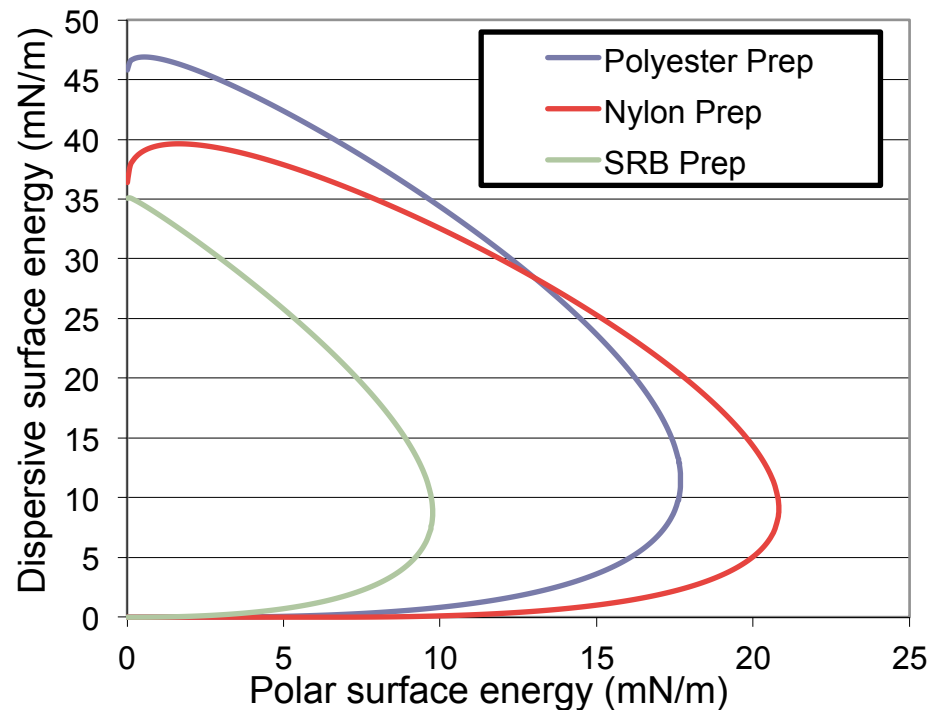
$$\gamma_{sl} = \gamma_{sv} - \gamma_{lv} \cos \theta$$

- 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



# Experimental Overview

## Investigate effect of contact angle fluid contamination on bond quality

- Contaminate CFRP surfaces with contact angle fluid followed by use of one of below methods:
  - Dry wipe
  - Acetone wipe
  - Air dry (in fume hood)
- Fabricate bonded specimens (bond within 4 hours)
  - Backing-Rapid Adhesion Test (B-RAT)/Climbing Drum Peel (CDP) Test
    - Failure mode
  - Double Cantilever Beam (DCB) Test
    - Mode I strain energy release rate ( $G_{IC}$ )

# 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 contamination
  - Fluids: DI water, ethylene glycol (EG), glycerol (Gly), diiodomethane (DIM)
  - BMS8-15 aerospace wipers
    - Application and removal of contamination

# Materials and Process

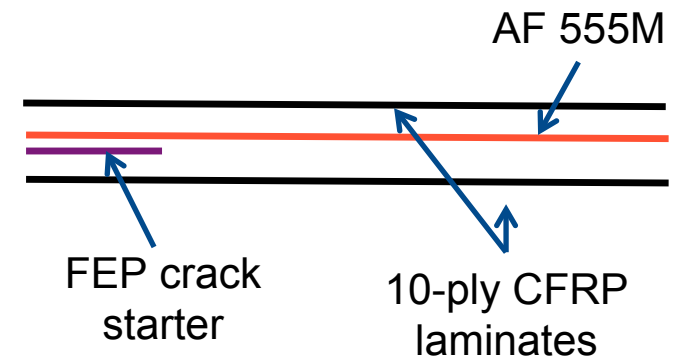
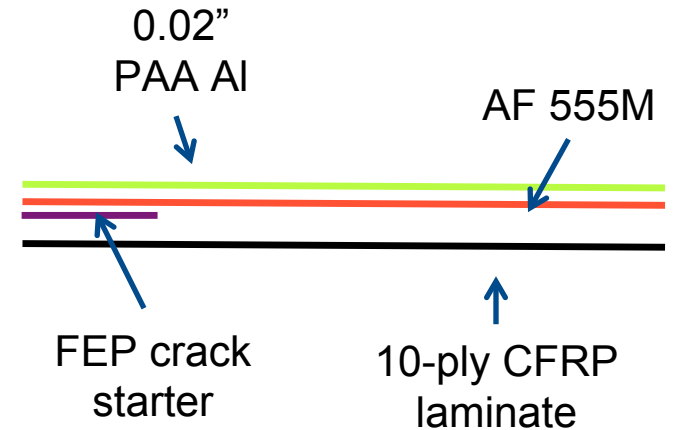
- Secondary Bonding

- B-RAT/CDP specimens

- 3M AF 555M film adhesive
    - 0.02" phosphate acid anodized Al
    - Autoclave cure (350 °F, 45 psi)

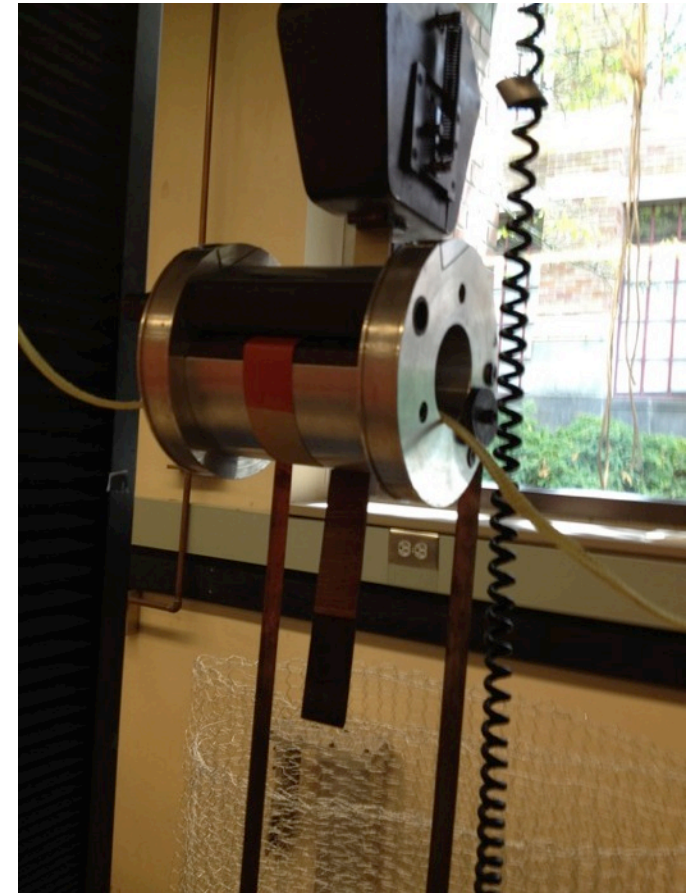
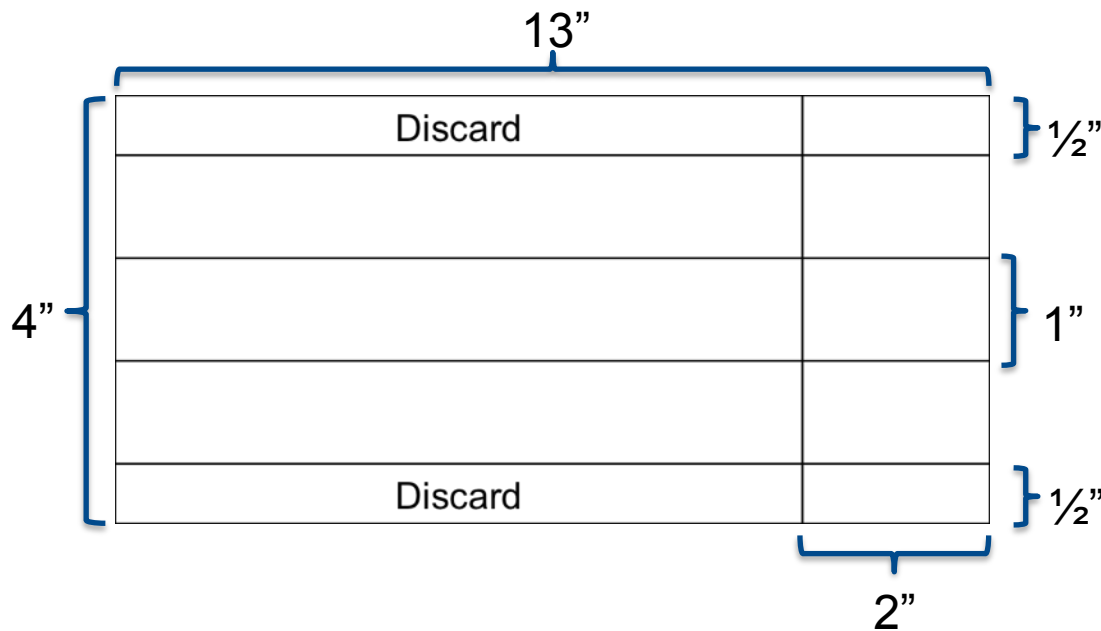
- DCB specimens

- 3M AF 555M film adhesive
    - Autoclave cure (350 °F, 89 psi)



# Climbing Drum Peel Test

- Bonded panels cut into (3) 1" x 13" specimens
- Test per ASTM D1781





# CDP/B-RAT Failure Modes

Control	DI Water		DIM		Gly		EG	
	AI	CFRP	AI	CFRP	AI	CFRP	AI	CFRP
Air Dry								
Dry Wipe								
Acetone Wipe								



- All cohesive failure within adhesive

# CDP/B-RAT Observations

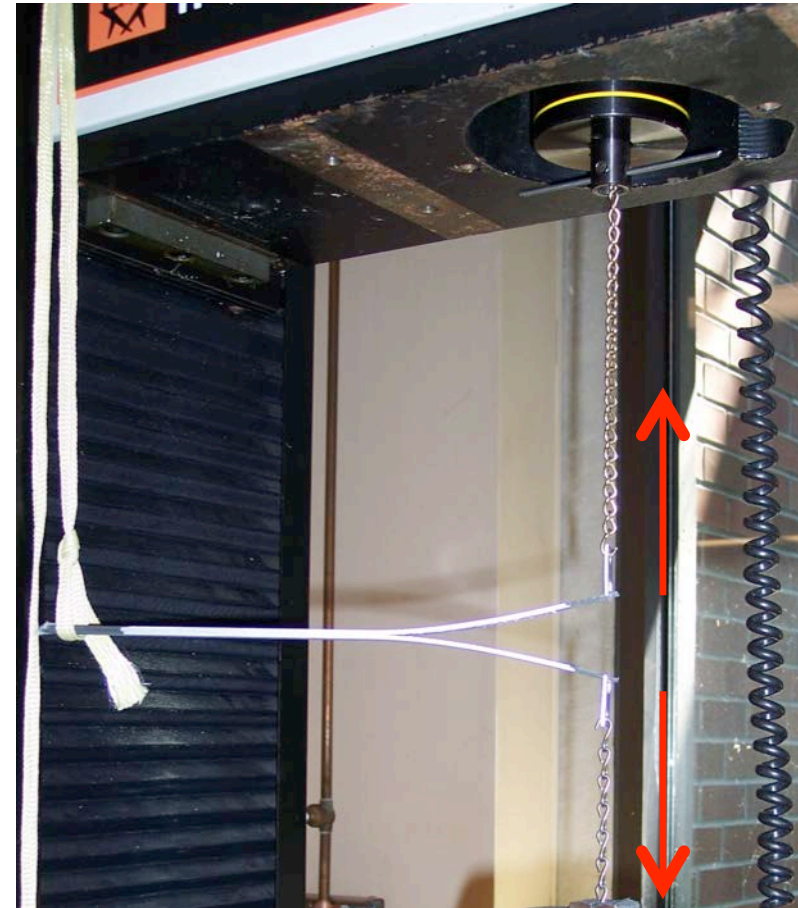
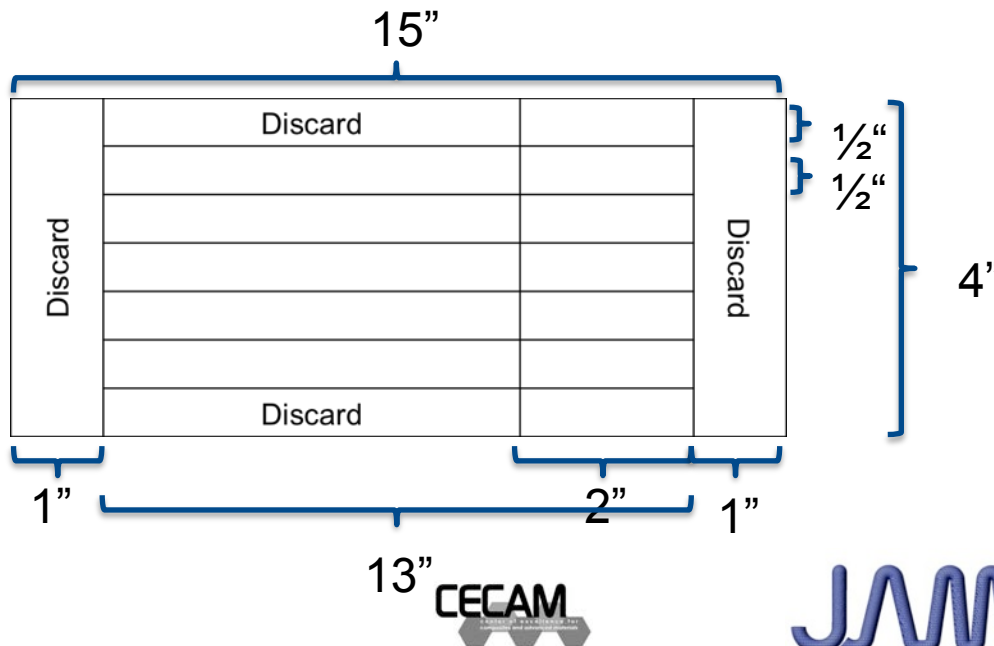
- Visual: cohesive failure in the adhesive
  - Desired failure mode
- Results suggest:
  - Failure mode not influenced by CA fluid contamination
  - CDP/B-RAT not sensitive to CA fluid contamination
  - Confirm results with DCB test?







# DCB Test

- Bonded panels cut into (5) 1/2" x 13" specimens
- Used area method
  - E: area of curve
  - A: crack length
  - B: specimen width

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



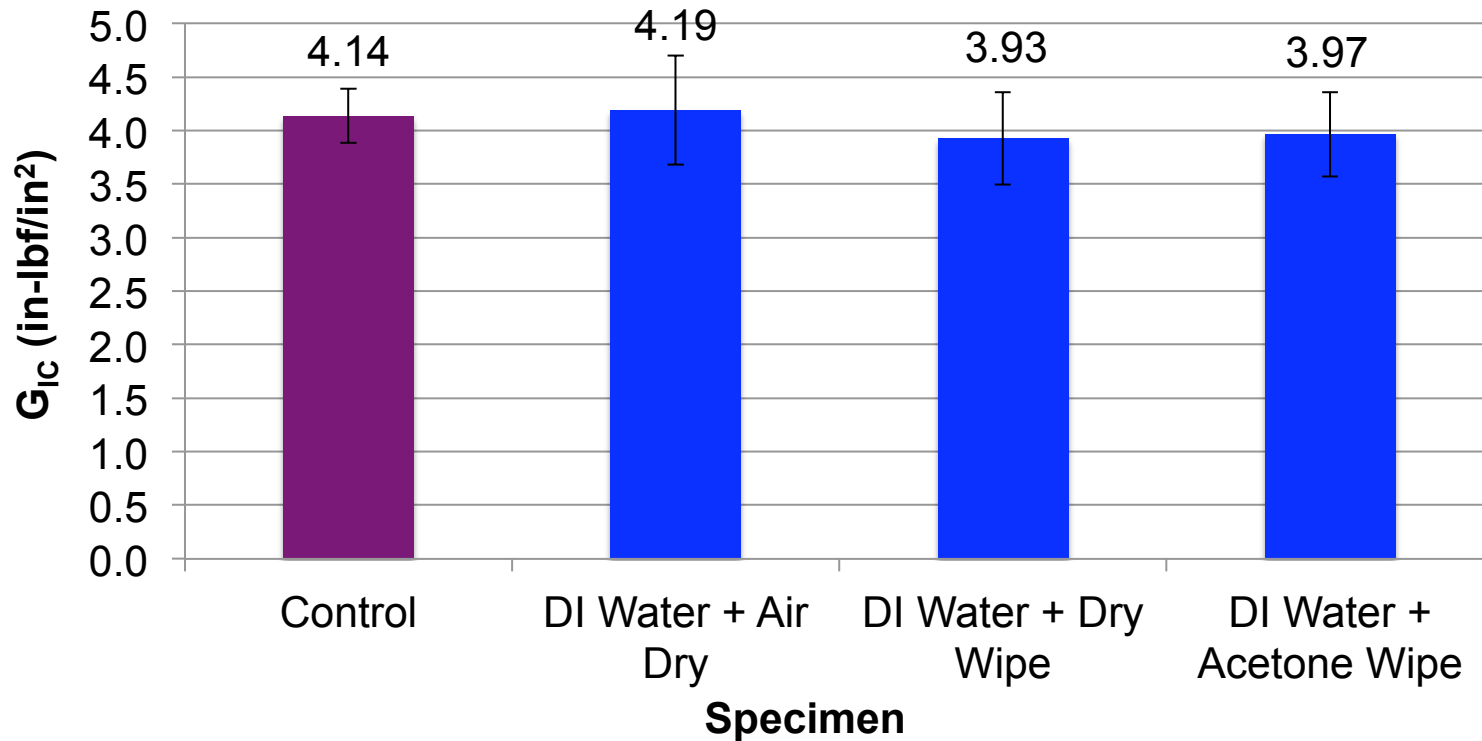
# DCB Failure Modes

Control	DI Water + Air Dry	DI Water + Dry Wipe	DI Water + Acetone Wipe
			

- All mixed failure modes: cohesive within adhesive and interlaminar

# DCB Mode I Strain Energy Release Rate

- Results to date: control and DI water contaminated samples
  - DCBs with gly, EG, DIM contamination?



# DCB Observations

- Visual: mixed failure modes → cohesive within adhesive + interlaminar
  - Desired failure modes
- No significant difference in  $G_{IC}$  values for DI water contaminated samples and control samples
- No degradation in bond quality found by DCB ( $G_{IC}$  or failure mode) for DI water contamination

# Summary

- Contact angle used to measure bonding surfaces → effect of measurement on surface?
  - CDP/B-RAT → no degradation in failure mode
  - DCB → no degradation in  $G_{IC}$  or failure mode for DI water contaminated samples
    - Need to examine other CA fluid contaminations, other systems (composite, surface prep, adhesive)

Bond Quality Test	Contact Angle Fluid Contamination			
	DI Water	DIM	EG	Gly
CDP/B-RAT	✓	✓	✓	✓
DCB	✓	TBD	TBD	TBD



# Summary

- Contact angle used to measure bonding surfaces → effect of measurement on surface?
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Bond Quality Test	Contact Angle Fluid Contamination			
	DI Water	DIM	EG	Gly
CDP/B-RAT	✓	✓	✓	✓
DCB	✓	TBD	TBD	TBD

**Contact angle is potential QA method**



# 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

- FAA, JAMS, AMTAS



- Boeing Company

- Marc Piehl, Kay Blohowiak, Will Grace, Pete VanVoast, Tony Belcher, Liz Castro, Paul Vahey, Paul Shelly, Greg Werner



- Precision Fabric Group



- Richmond Aircraft Products



- Airtech International



- UW MSE



Thank you

Questions and comments welcome

