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Improving Adhesive Bonding of Composites Through Surface Characterization

November 1, 2011

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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 preparation
- Objective
  - Develop QA technique for surface preparation
- Approach
  - Investigate different surface preparations and process variables using laboratory and handheld devices

FAA Sponsored Project Information

- Principal Investigators & Researchers
  - Brian D. Flinn (PI)
  - Ashley Tracey (PhD student, UW-MSE)
  - Lisa Carlson(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 (Kay Blohowiak, Peter Van Voast, William Grace, Liz Castro, John Spalding, Mary Vargas & Paul Shelley)

2010-2011 Statement of Work

	Surface Characterization/QA Technique			
	Contact Angle		**FTIR**	
	Goniometer	**Surface Analyst**	**ATR**	**Exoscan**
Cure Temp and Dwell Time	✔	✔		
Peel Ply Prep	✔	✔	✔	✔
Si Contaminants	✔	✔	✔ (Boeing)	
Peel Ply Orientation	✔	✔ No effect	N/A	
Abraded Texture	✔			
Scarfed Surfaces/ Repair				

✔ = work completed

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Surface Characterization

For a good bond: 1) Adhesive must wet substrate and 2) strong chemical bonds between adhesive and substrate

Surface Energy

- Ability of adhesive to wet substrate
- Characterized by contact angle
- Contamination can lower surface energy

Surface Chemistry

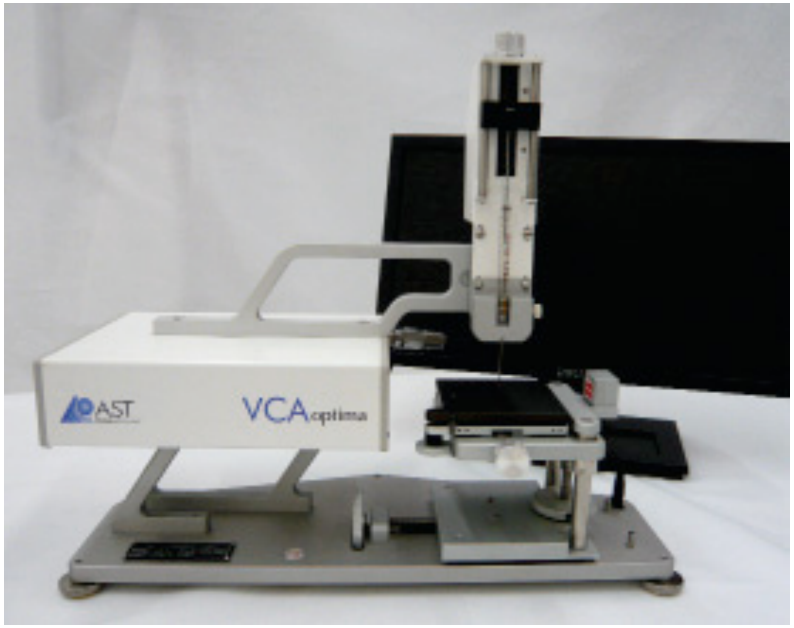
- Availability of chemical bonds at the surface
- Characterized by FTIR
- Contamination changes surface chemistry

Surface preparation influences the energetics and chemistry of a substrate

Contact Angle Methodology (Surface Energy)

VCA Optima Goniometer

- Bench top device
- Lab research



http://www.astp.com/contact-angle/optima

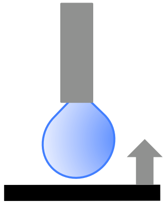
Brighton Surface Analyst™

- Handheld device
- In-field inspection

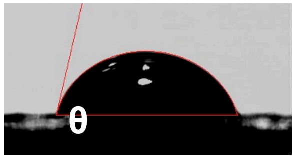
http://www.btgnow.com

Contact Angle Methodology

Goniometer



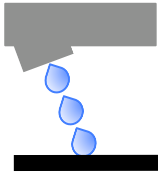
Drop application: dispense drop, raise surface



Side-view of drop as viewed from goniometer camera

- 4 fluids
- 10 drops/fluid/substrate
- Calculate surface energy

Surface Analyst™



Drop application: dynamically apply 20 small drops to form 1 drop

Top-view of drop as viewed from Surface Analyst camera

Note: rectangle is reflection from camera light

- Water only
- 20 drops/substrate
- Average contact angle

FTIR Methodology

Interaction between IR beam and material produce spectra displaying chemical bonds in material

Bruker Vertex 70 FTIR

- Bench top device
- Attenuated total reflectance (ATR)

http://www.aoc.kit.edu/english/612.php

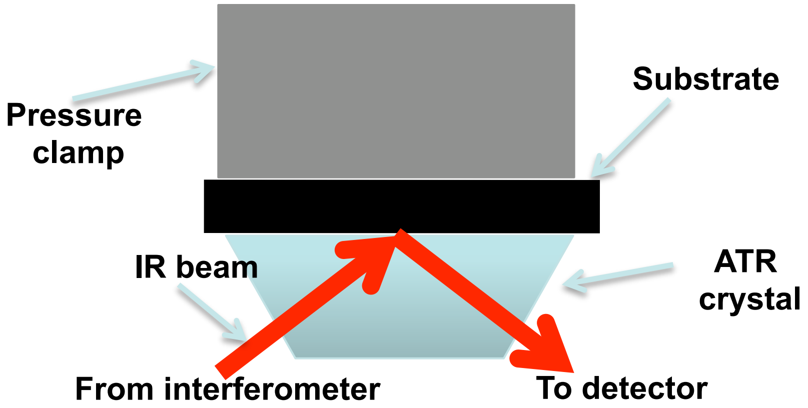
Agilent Technologies Exoscan™ FTIR

- Handheld device
- Specular reflectance

https://www.chem.agilent.com

FTIR Methodology

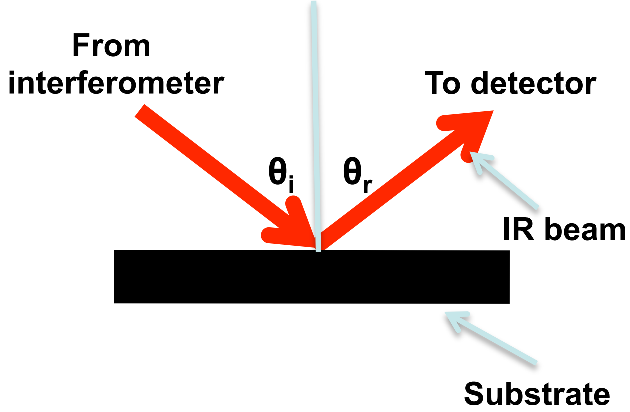
Vertex 70



An IR beam path for single bounce ATR

- Single bounce MIR diamond ATR
- Use pressure clamp to ensure good contact with substrate

Exoscan™



An IR beam path of specular reflectance

- MIR specular reflectance
- Non-contact

Experimental Overview

Assess potential QA methods ability to identify variations in process conditions

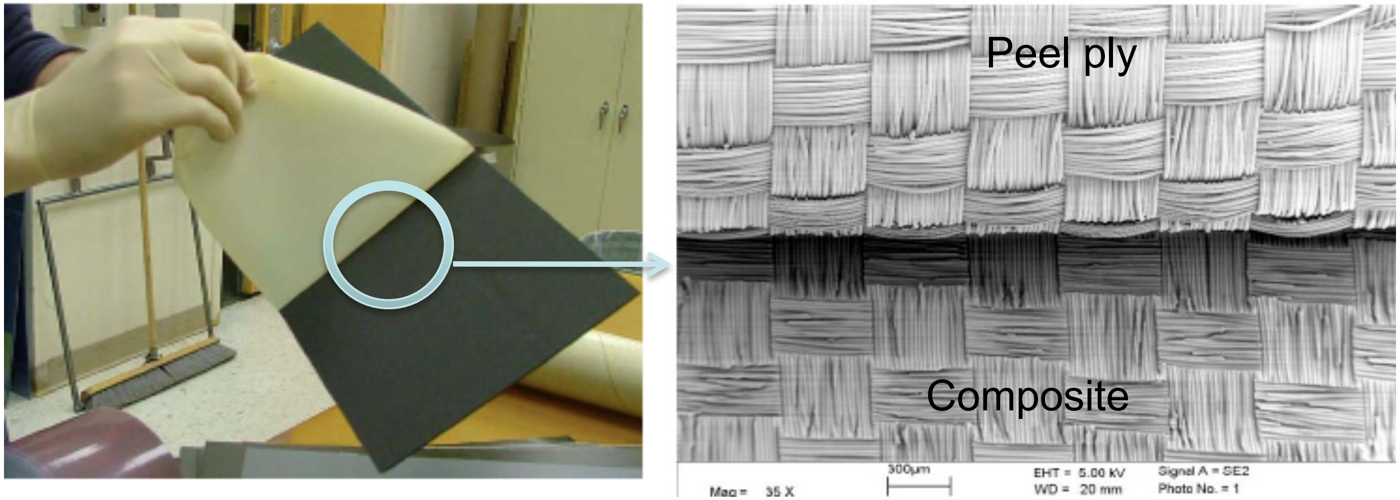
- Surface Preparations:
  - Polyester peel ply, nylon peel ply, SRB release ply
- Peel Ply Contamination:
  - Various levels of siloxane contamination
- Abrasion Variables:
  - Grit size: 80, 220, 400
  - Orientation

Materials and Process

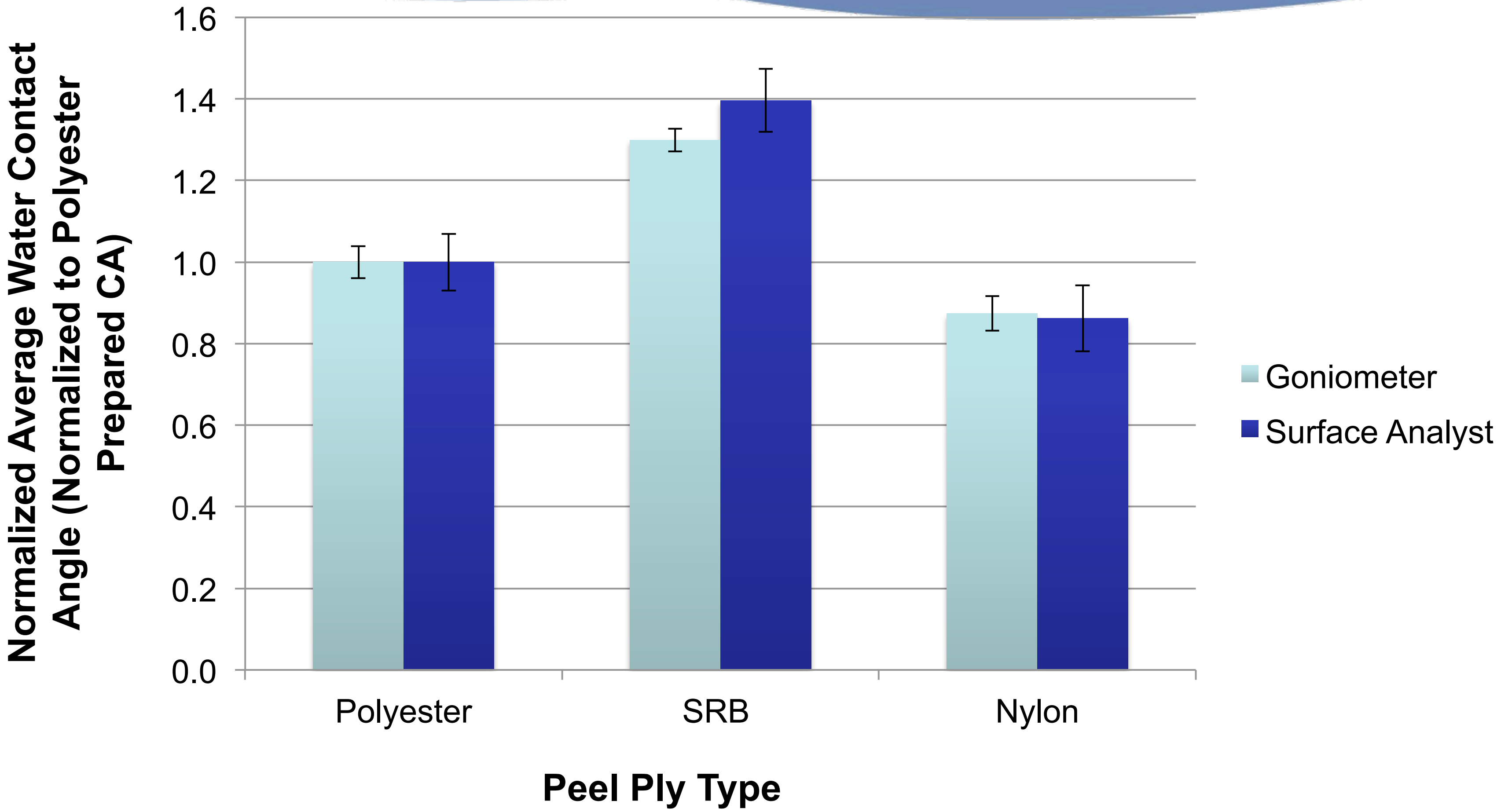
- Toray 3900/T800 unidirectional laminates
- Peel ply surface prep
  - Precision Fabric Group 60001 polyester peel ply
  - Precision Fabric Group 52006 nylon peel ply
  - Precision Fabric Group SRB release ply
- Autoclave cure (177°C, 0.6MPa)
- Fluids used for contact angle analysis:
  - De-ionized water (DI water)
  - Ethylene Glycol (EG)
  - Glycerol (Gly)
  - Diiodomethane (DIM)
  - Formamide (Form)
- 3M Al_2O_3 grit abrasive cloth

Characterization of Peel Ply Preparation

- Affect of peel ply type on surface characteristics
  - Polyester peel ply
  - Nylon peel ply
  - SRB release ply

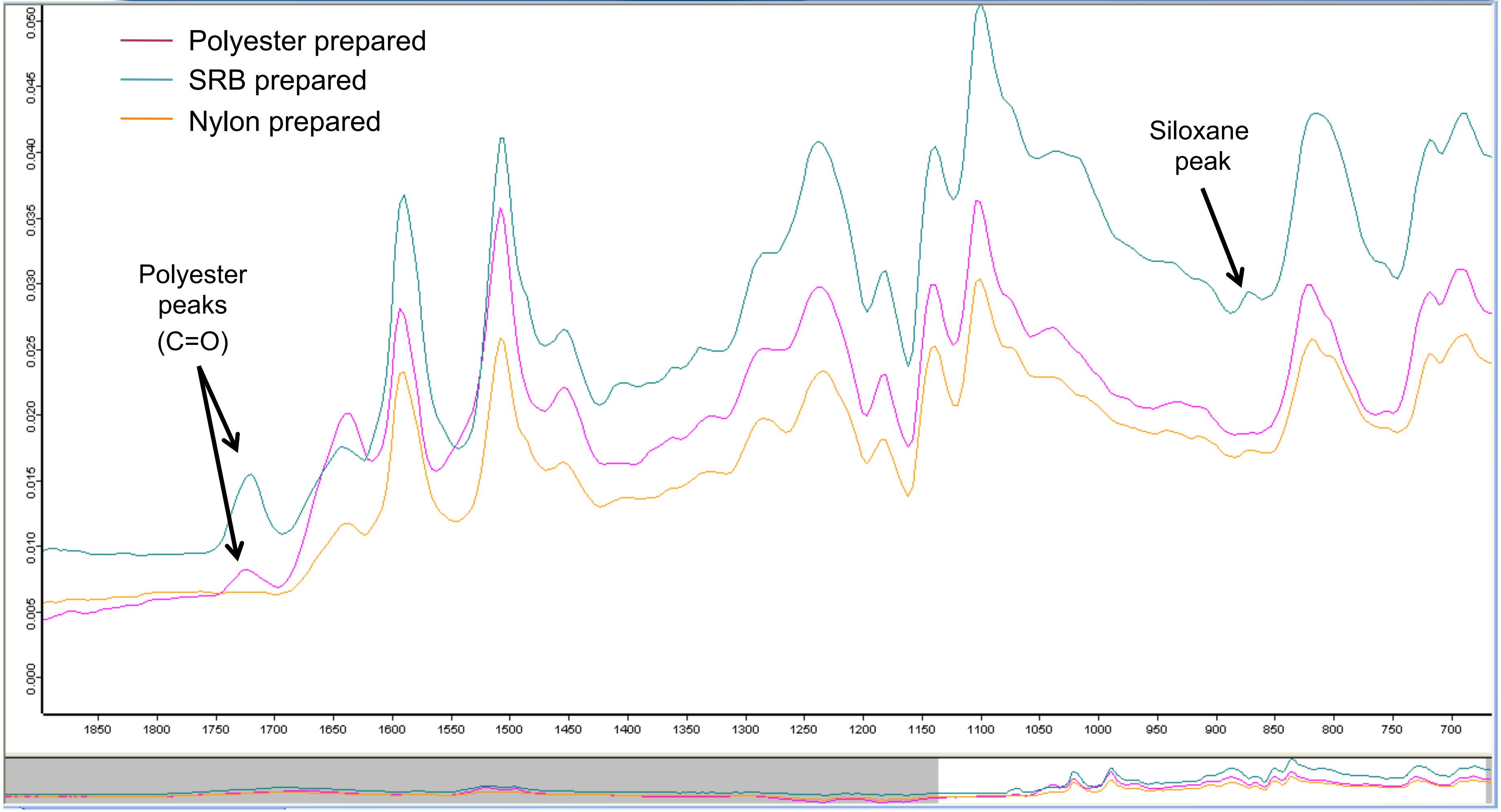


Contact Angle Results (DI H_2O)



✓ Both methods detect differences in peel ply type

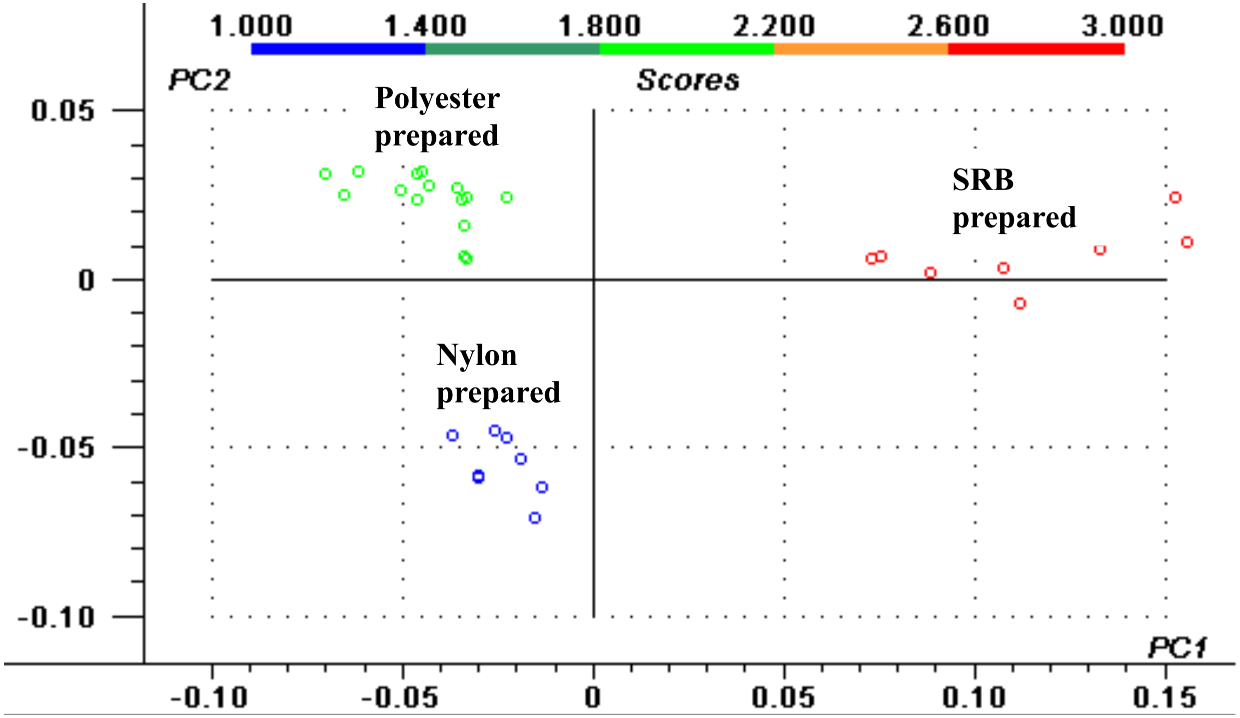
FTIR-ATR Surface Chemistry Results



- ✓ Differences in surface chemistry are evident

FTIR-Exoscan™ Surface Chemistry Results

Multivariate Analysis of Specular Reflectance Spectra



Partial least squares with Savitsky-Golay first derivative and 5 smoothing point preprocessing 2-principal component model

- Method written for Exoscan™ to identify peel ply type
- ✓ Exoscan™ can be used to identify different peel ply/release ply surface preps

In collaboration with Paul Shelley, Boeing

Characterization of Surface Preparation

Effect of Peel Ply Contamination

- Contaminants are detrimental to bonding
- Previous research at Boeing showed that FTIR-ATR can detect contamination levels >0.5% on the cured laminate[1]
  - Can contact angle be used to identify surface contamination?

Mix Solids Target Level (% siloxane)
0% (control)
0.0001%
0.001%
0.01%
0.05%
0.1%
0.2%
0.3%
0.4%
0.5%
1%
2%

Detrimental to Bonding[1]

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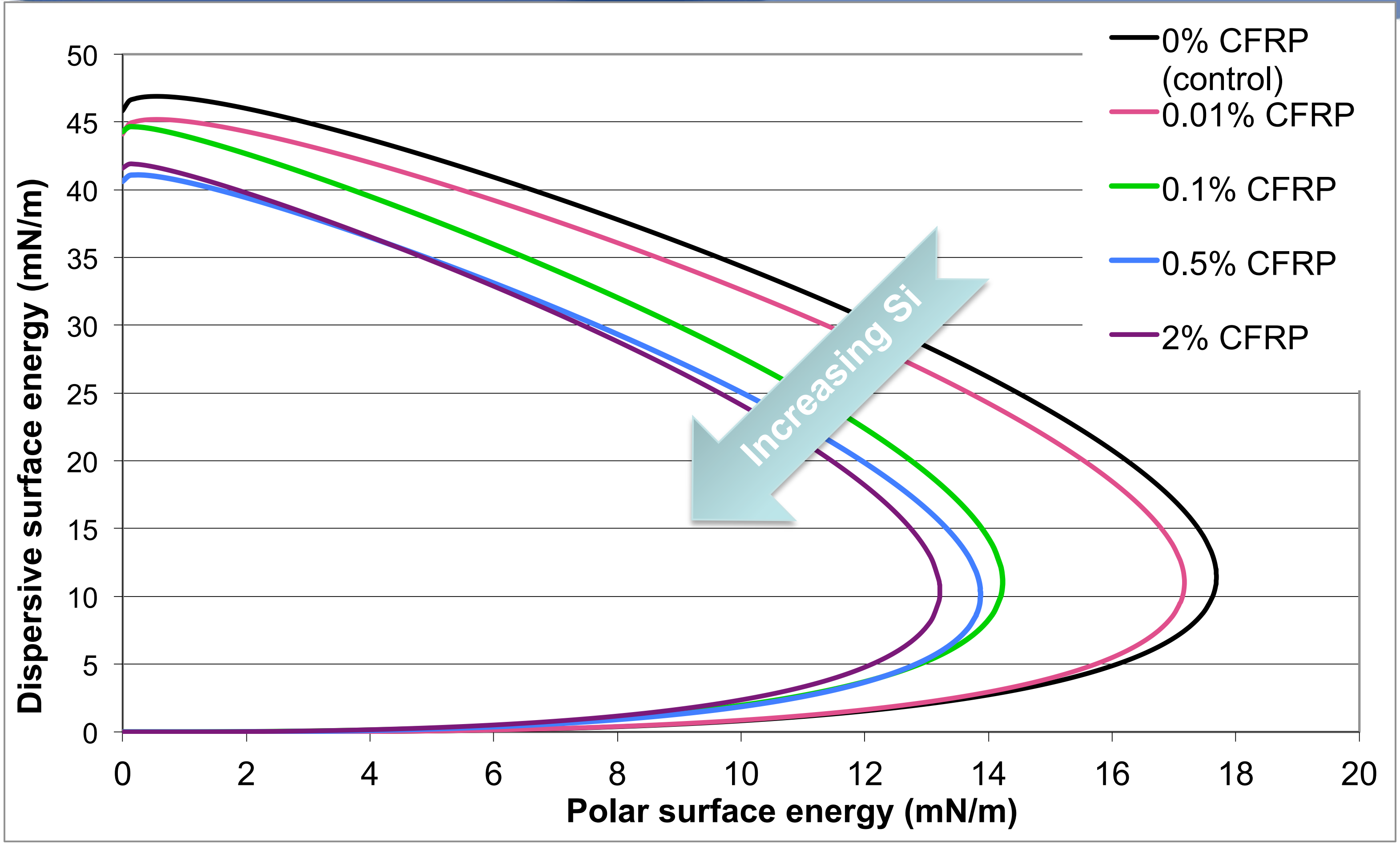
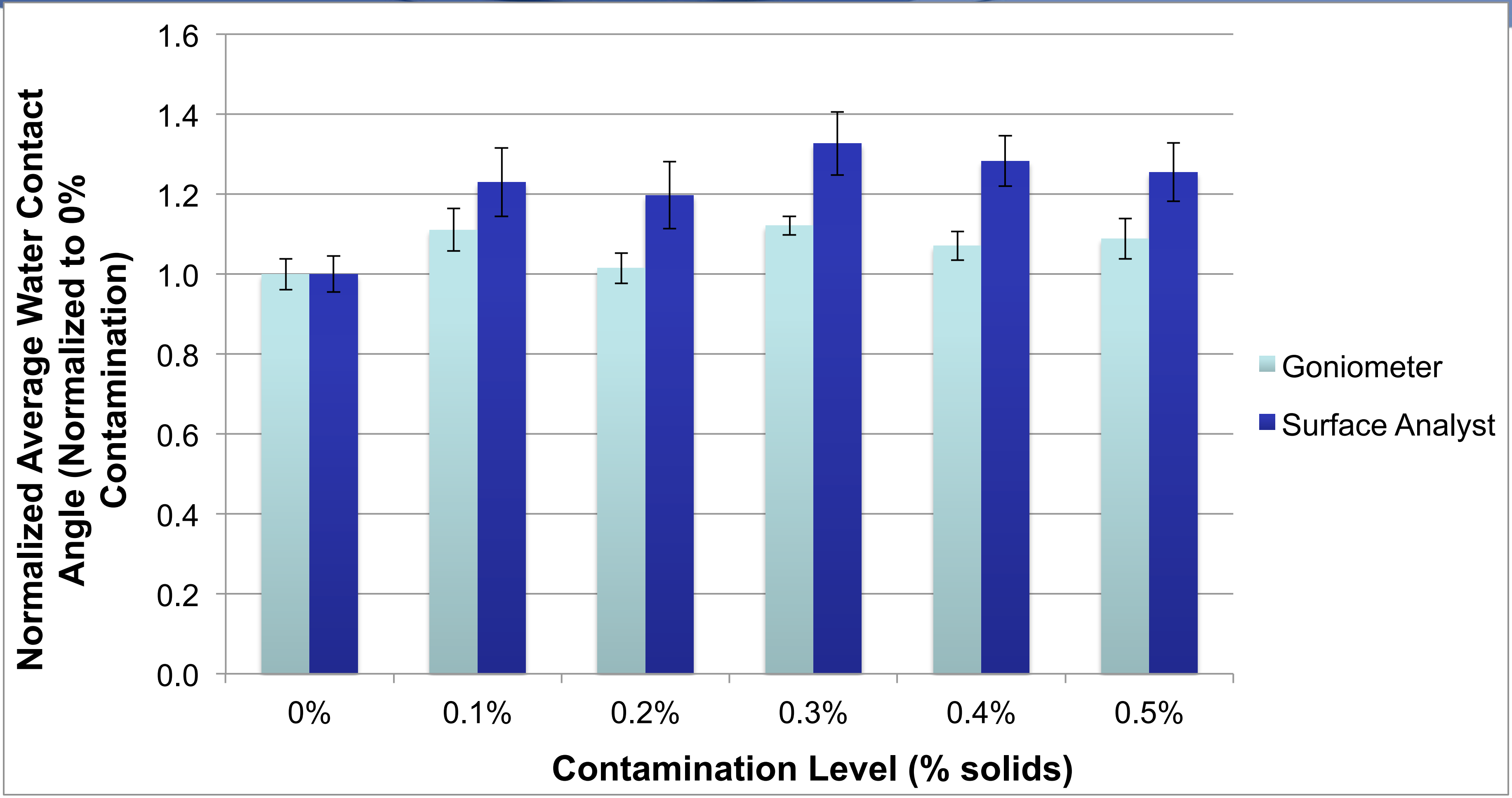
[1] VanVoast, P.J., P.H. Shelley, R.L. Blakely, C.B. Smith, M.P. Jones, A.C. Tracey, B.D. Flinn, G. Dillingham, B. Oakley. “Effect of Varying Levels of Peel Ply Contamination on Adhesion Threshold.” SAMPE 2010 – Seattle, WA May 17-20, 2010.

Goniometer Results: Wettability Envelopes



- ✓ Contact angle sensitive to < 0.1% Si contamination

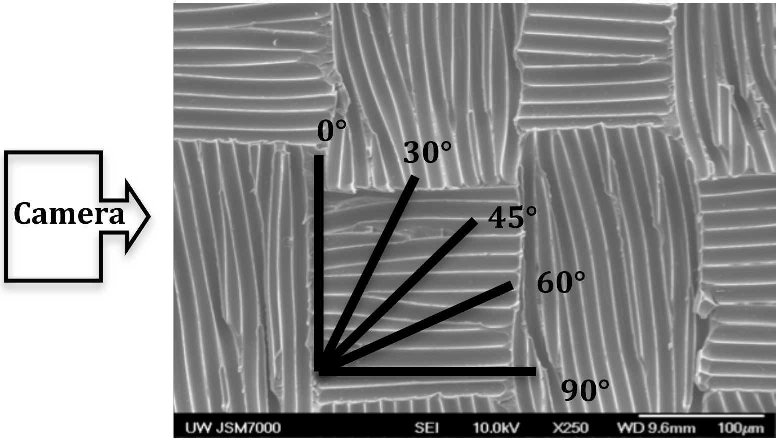
Contact Angle Results

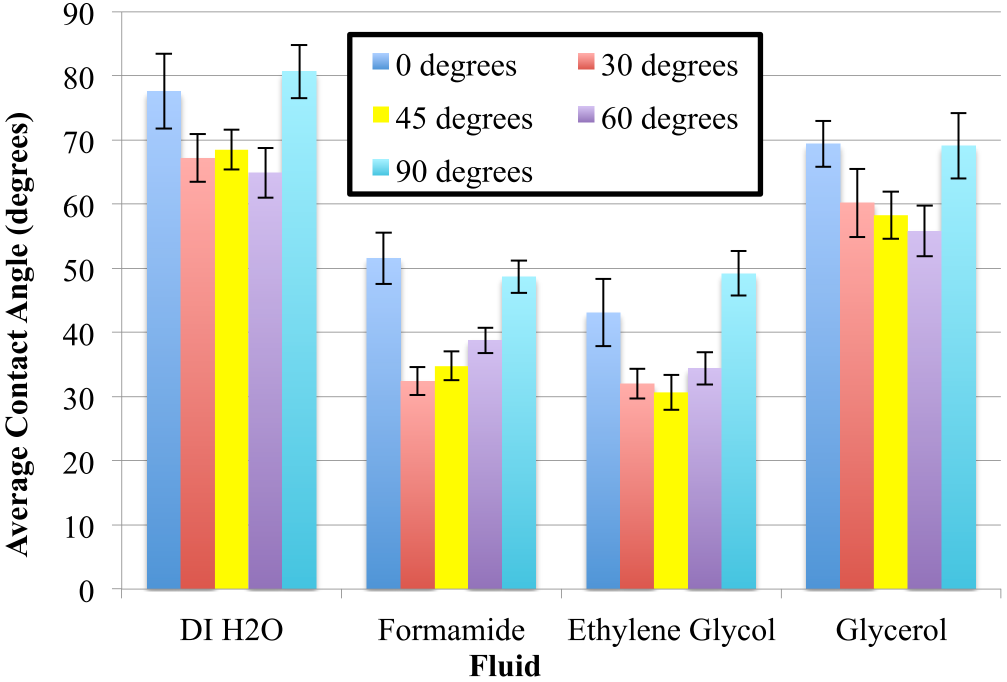


✓ Both methods detect contamination below that which affects bonding (1%)

Surface Roughness

- Previous research showed contact angle changes at different peel ply angles with respect to goniometer camera due to noncircular drops

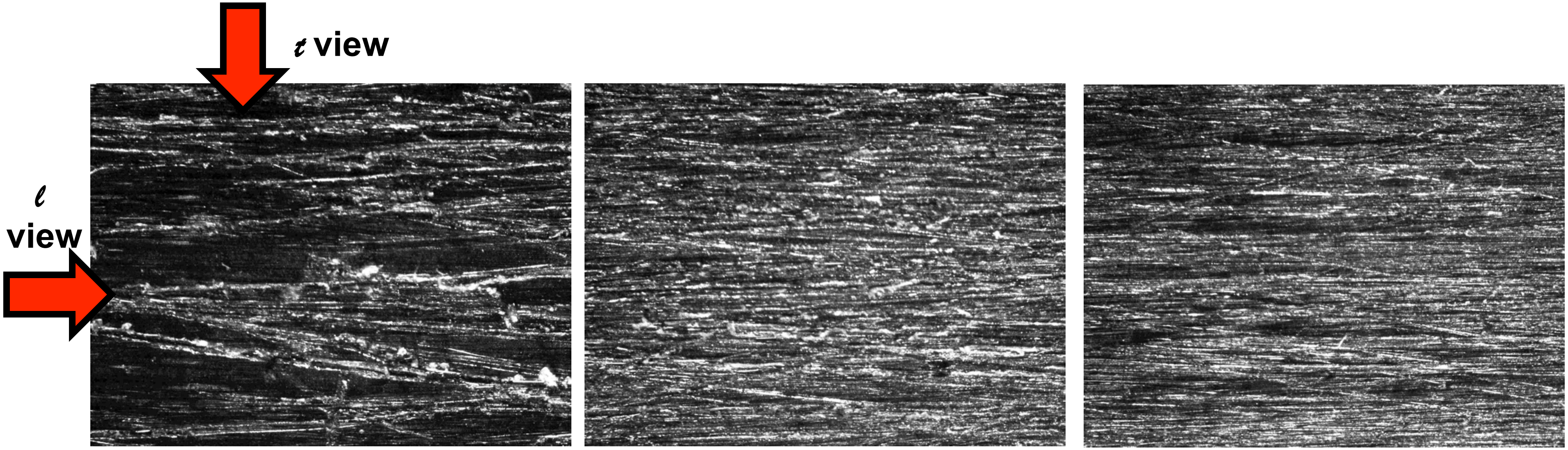




- Contact angles highest at 0° or 90° orientation, lower at all other orientations
  - Measure contact angle at 0° or 90° orientation

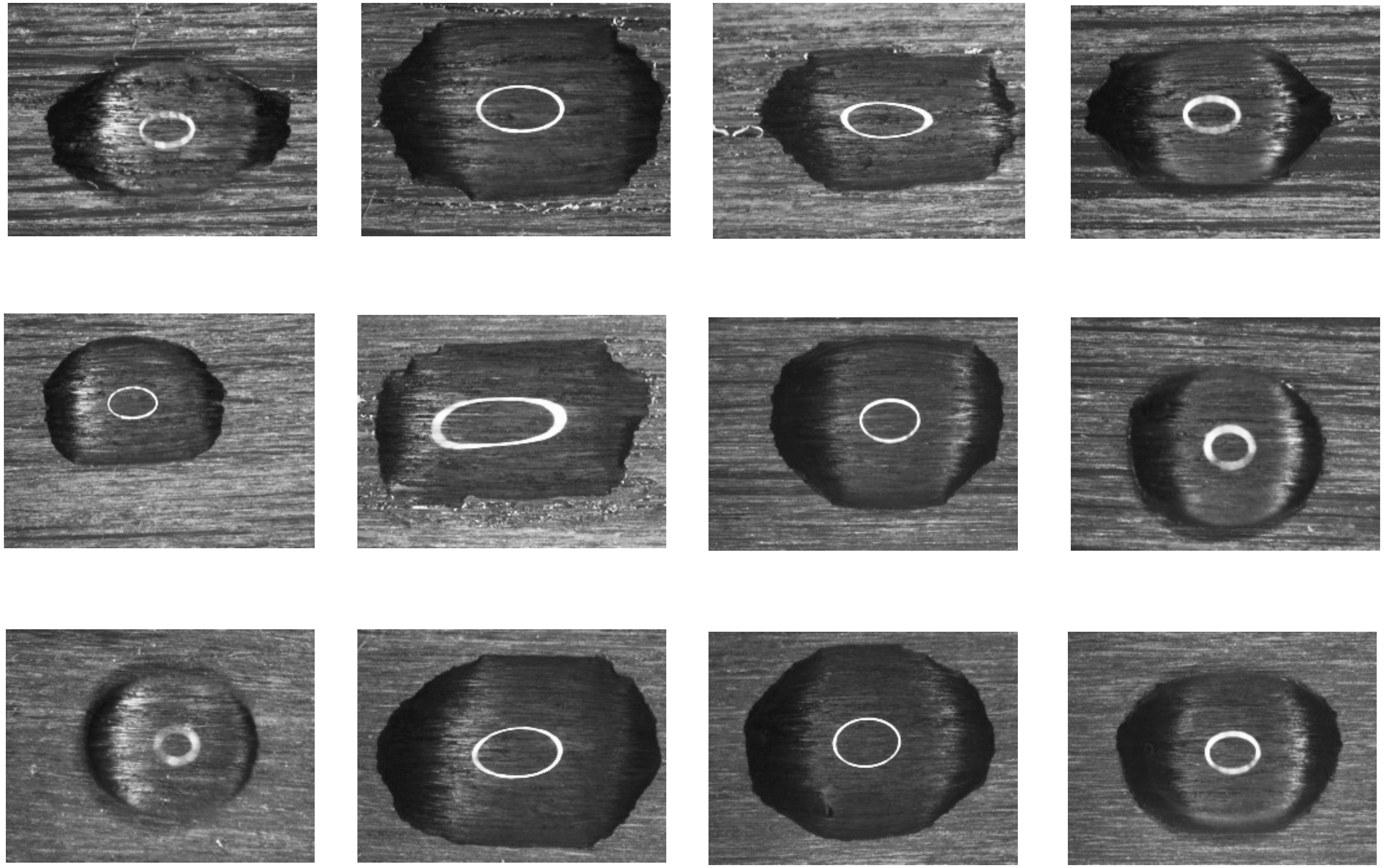
Abrasion Texture: Stereoscope Images

- Want to further understand effect of roughness
  - Manual abrasion (surface prep)
  - As tooled surfaces abraded with 80, 220 and 400 grit abrasive cloth parallel to fiber (ℓ) direction
  - Measure contact angle at longitudinal (ℓ) and transverse (𝓉) views



80 grit **220 grit** **400 grit**

Effect of Surface Roughness on Contact Angle



DI H_2O | Ethylene Glycol | Formamide | Glycerol

80 grit
$R_a(t) = 94.6 \pm 16.6$ µin
$R_a(\ell) = 43.6 \pm 20.8$ µin

220 grit
$R_a(t) = 63.4 \pm 9.1$ µin
$R_a(\ell) = 40.7 \pm 8.7$µin

400 grit
$R_a(t) = 35.0 \pm 4.9$ µin
$R_a(\ell) = 19.8 \pm 3.3$ µin

Note: white circles on drops are reflection of camera light

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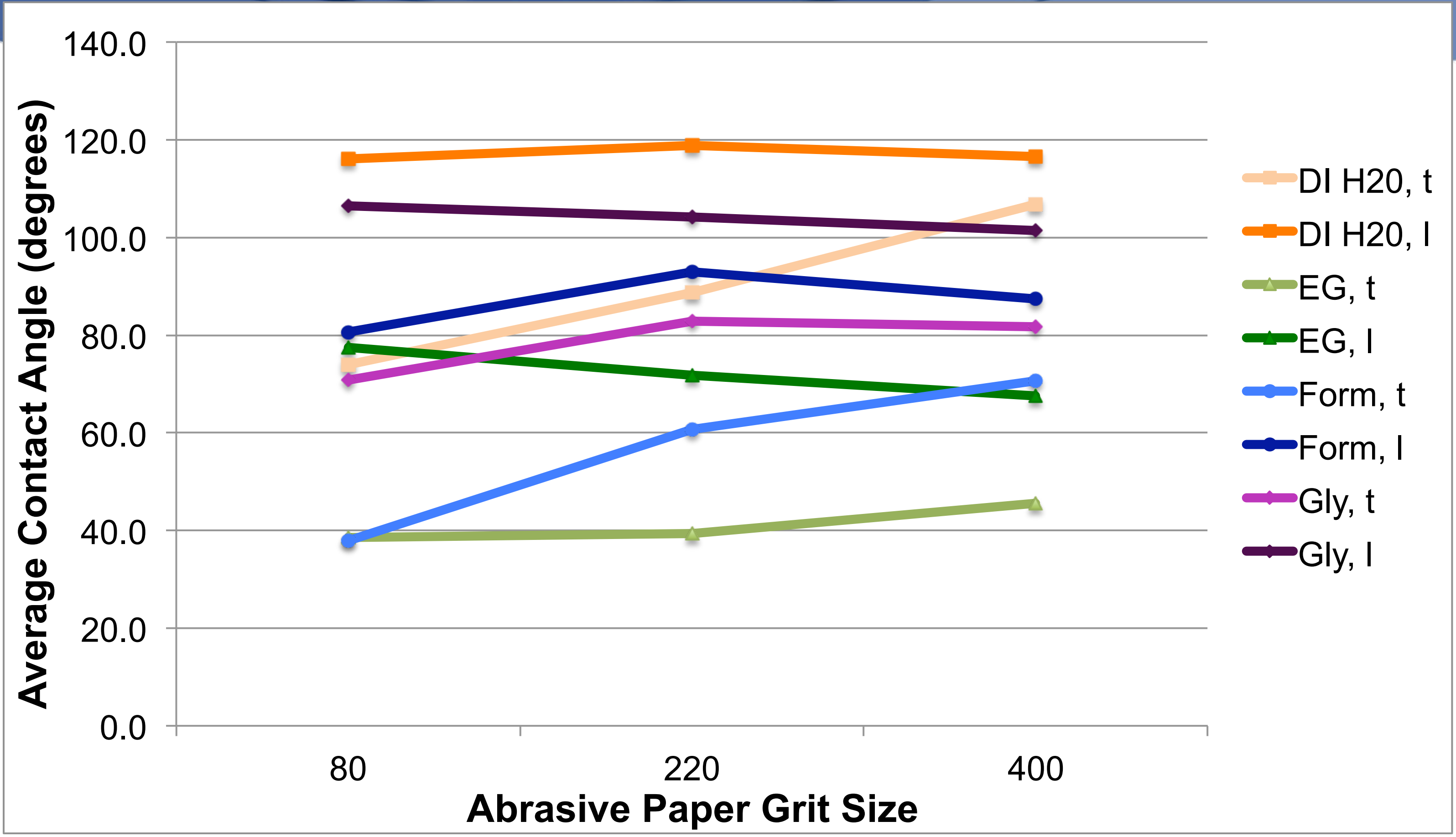
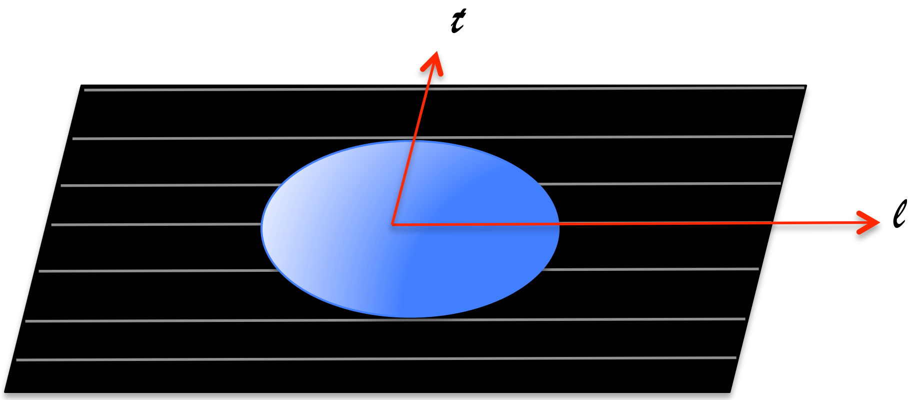
Contact Angle: Effect of Surface Roughness

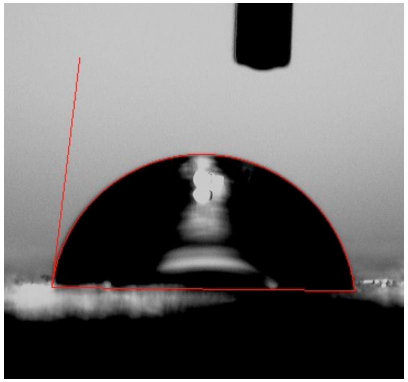


- ✓ Transverse (*t*) contact angle lower than longitudinal (*ℓ*)
- Contact angle decreases with increased roughness
  - Exception: EG – viscosity effect?

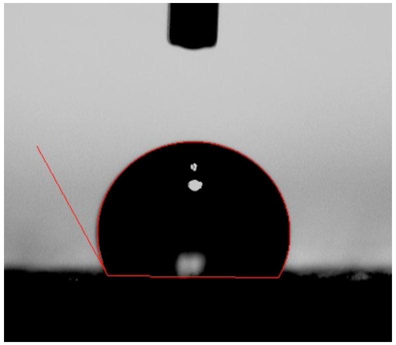
Surface Roughness and Contact Angle

- Fluids form noncircular drops on surfaces
  - Fluid flows down path of least resistance ➔ t contact angle lower
  - Fluid arrested at peaks between grooves ➔ ℓ contact angle higher





DI H2O on 220 grit surface viewing along t



DI H2O on 220 grit surface viewing along ℓ

Summary

	Surface Characterization/QA Technique			
	Contact Angle		**FTIR**	
	Goniometer	**Surface Analyst**	**ATR**	**Exoscan**
Cure Temp and Dwell Time	✔	✔	TBD	TBD
Peel Ply Prep	✔	✔	✔	✔
Si Contaminants	✔	✔	✔ (Boeing)	TBD
Peel Ply Orientation	✔	✔ No effect	N/A	TBD
Abraded Texture	✔			
Scarfed Surfaces/ Repair	TBD	TBD	TBD	TBD

More work is necessary, but contact angle and FTIR have a potential for QA methods

Looking 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
  - – 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
- Precision Fabric Group
- Richmond Aircraft Products
- Airtech International
- Prof. Mark Tuttle (UW)
- Paul Shelley (Boeing)

Thank you

Questions and comments welcome

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