Improving Adhesive Bonding of Composites Through Surface Characterization

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FAA Sponsored Project Information

- Principal Investigators & Researchers
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  - Ashley Tracey (PhD student, UW-MSE)
  - Jake Plummer (undergraduate, UW-MSE)
- FAA Technical Monitor
  - David Westlund
- Other FAA Personnel Involved
  - Larry Ilcewicz
- Industry Participation
  - Toray Composites
  - Henkel International
  - Precision Fabrics & Richmond Aerospace & Airtech International
  - The Boeing Company (Kay Blohowiak, Peter Van Voast, and William Grace)

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 variables that affect contact angle measurements
  - Verify technique on intentionally contaminated surfaces

2010-2011 Statement of Work

- Literature review to understand state of composite bonding and surface analysis techniques
- Map and characterize bonding processing steps to locate highest risk factors in process
- Determine locations to incorporate in-line Quality Control (QC) methods
  - Contact angle (CA)
  - Fourier transform infrared spectroscopy (FTIR)
- Use QC assessment methods at identified critical processing steps to evaluate process conditions and reliability of bonded joint
- Assess tool's ability to identify less-than-desirable process conditions to determine their suitability for QC
- Correlate surface conditions to bond strength and durability
- Support of other AMTAS bonding research
  - FIU (bond durability)
  - U of Utah (metal bond wedge test)

Summary of Progress

- Variables that affect contact angle measurement:
  - Time to measure contact angle
    - Increase in time resulted in a decrease in contact angle => ALWAYS freeze image after 5 seconds
  - Peel ply orientation
    - Different peel ply orientations resulted in different contact angle measurements => ALWAYS measure contact angle at the same orientation (0 degrees)
  - Siloxane Contamination
    - Increase in contamination resulted in an increase in contact angle
    - Current research determining detection limit
  - **Cure Cycle (different temperatures and dwell times)**

Summary of Progress

- Variables that did not have a significant effect on contact angle measurement:
  - Material Lot (different dates of manufacture)
  - Cure Run (same cure cycle, different run)
  - Exposure After Peel Ply Removal (ranging from 0-48 hour exposure to ambient lab conditions)

Surface Energy to Probe Surfaces

- Why use surface energy to probe the surface preparation method applied to the composite for bonding?
  - One requirement of adhesion is the adhesive must wet the substrate
    - This is controlled by surface energy

Low surface energy High surface energy

  - Contact angle is influenced by surface prep.

Materials

- Toray 3900/T800 unidirectional laminates
- Precision Fabric Group 60001 polyester peel ply
- Autoclave cure of composites
- Fluids used for contact angle analysis:
  - De-ionized water (DI water)
  - Dimethlysufoxide (DMSO)
  - Ethylene Glycol (EG)
  - Glycerol (Gly)
  - Formamide (Form)
  - Diiodomethane (DIM)

Methodology

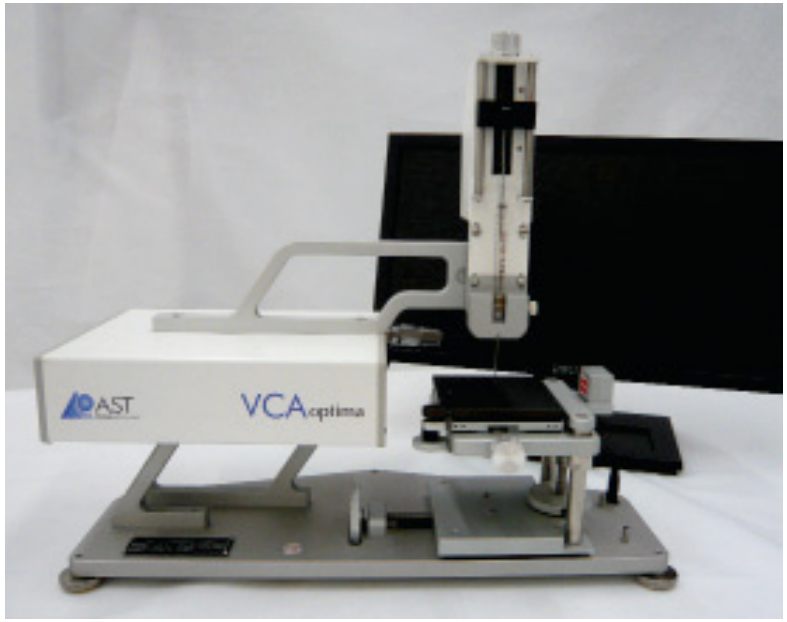
Brighton Surface Analyst

- Handheld device
  - In-field inspection

http://www.btgnow.com

VCA Optima Goniometer

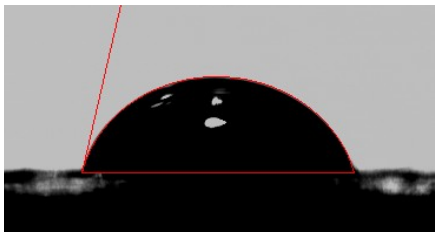
- Desktop device
  - Lab research

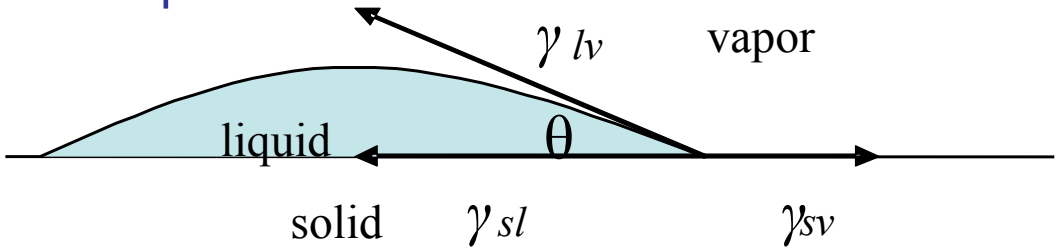


http://www.astp.com/contact-angle/optima

Goniometer Methodology

- Using a goniometer, the contact angle of a 1µL drop of fluid is measured – side view
  - Peel ply removed and contact angles measured within 1 hour
  - Four fluids, 10 drops per fluid were evaluated on each surface
  - Average contact angle and standard deviation were calculated to determine surface energies and generate wettability envelopes

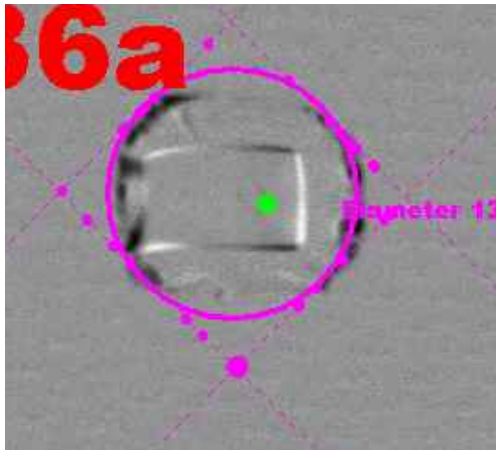




- Complete wetting when θ approaches zero
- Contaminants usually lower the solid's surface energy (increase θ)
- Surface preparations try to increase the solid's surface energy and clean off contaminants

Brighton Methodology

- Using the Brighton Surface Analyst, the contact angle of a 1.38µL drop (20 69nL drops) of water is measured – top view
  - Contact angle is calculated by fitting the circumference to the volume of the drop
  - Average contact angle and standard deviation were calculated for comparison to water CAs measured with use of goniometry



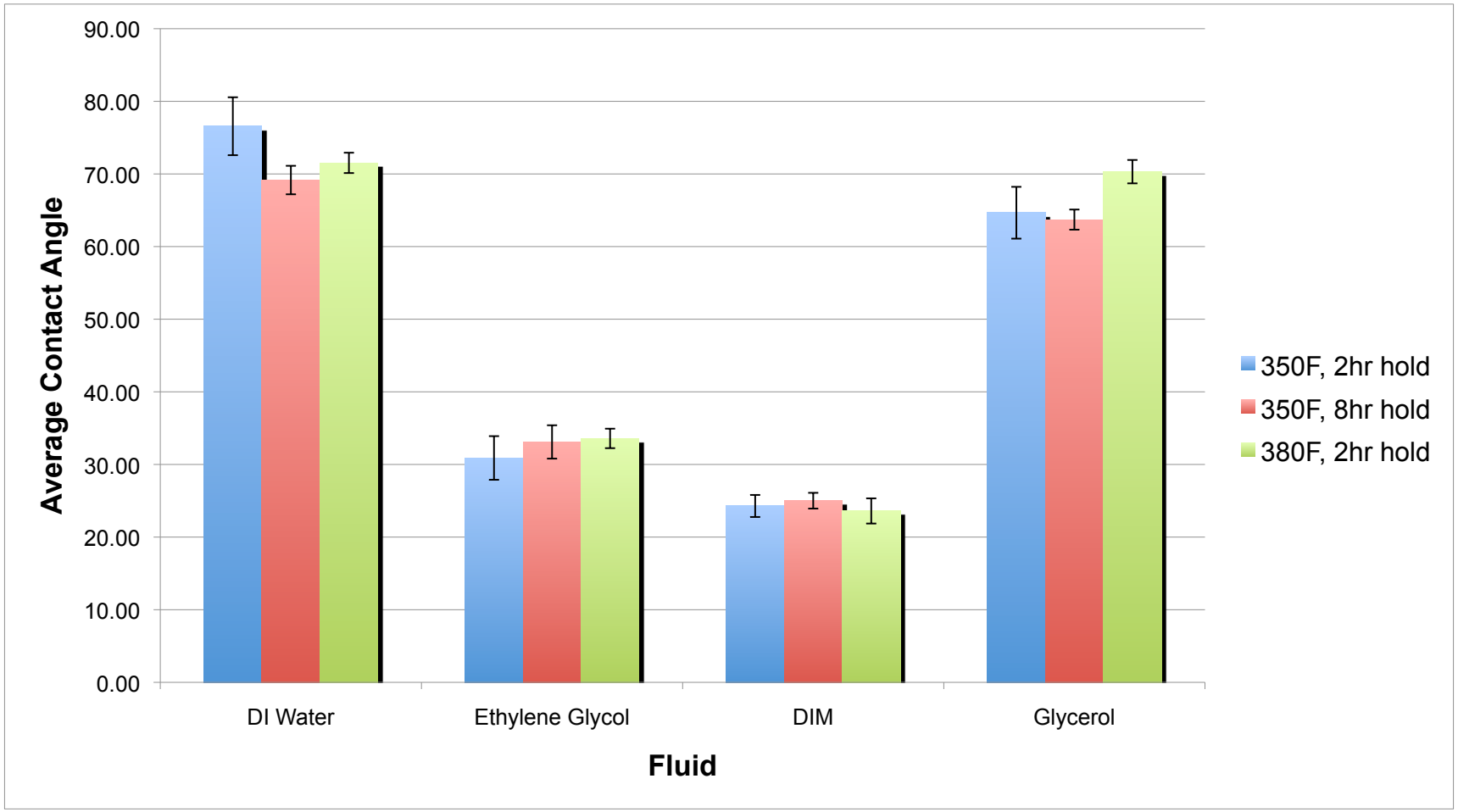
• **Note: rectangle in image is a reflection of light from camera**

http://www.btgnow.com/SEP.html

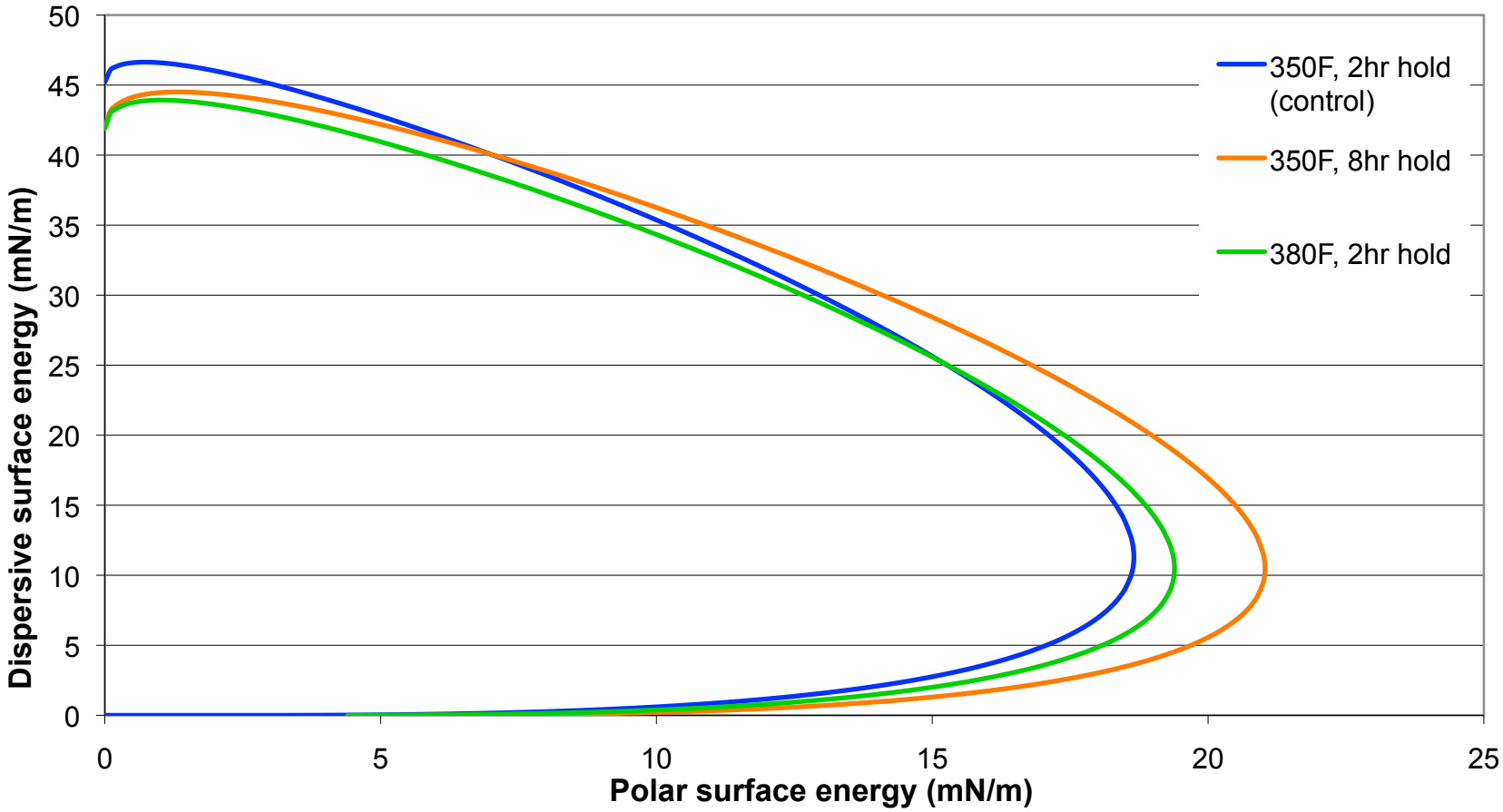
Cure Cycle

- Does temperature and dwell time affect contact angle measurement and/or bondability?
  - Previous research from Boeing has shown that increased temperatures and dwell times during autoclave cure decrease fracture energy

Effect of Cure Cycle on Contact Angle Measurement

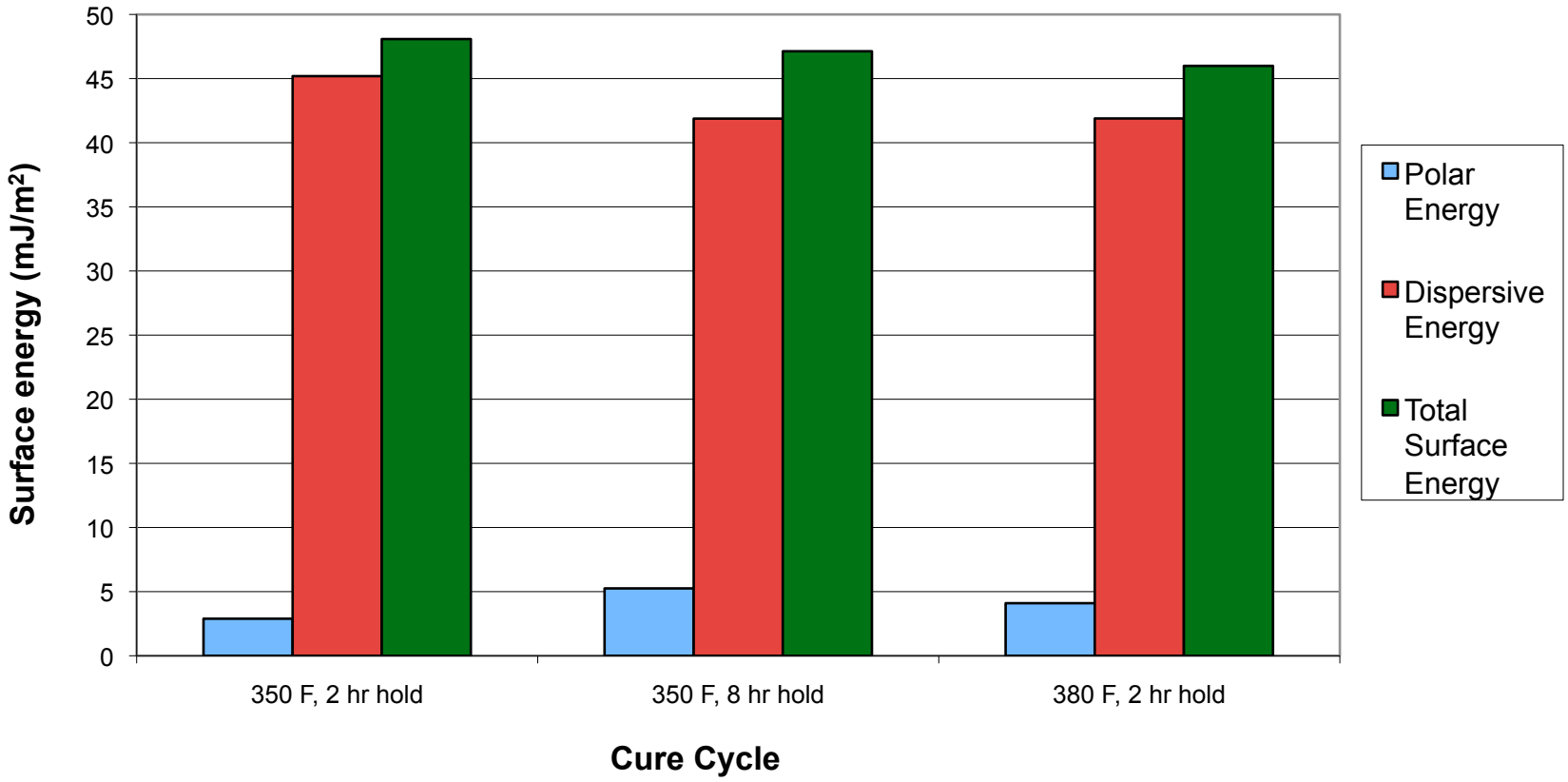


Wettability Envelopes

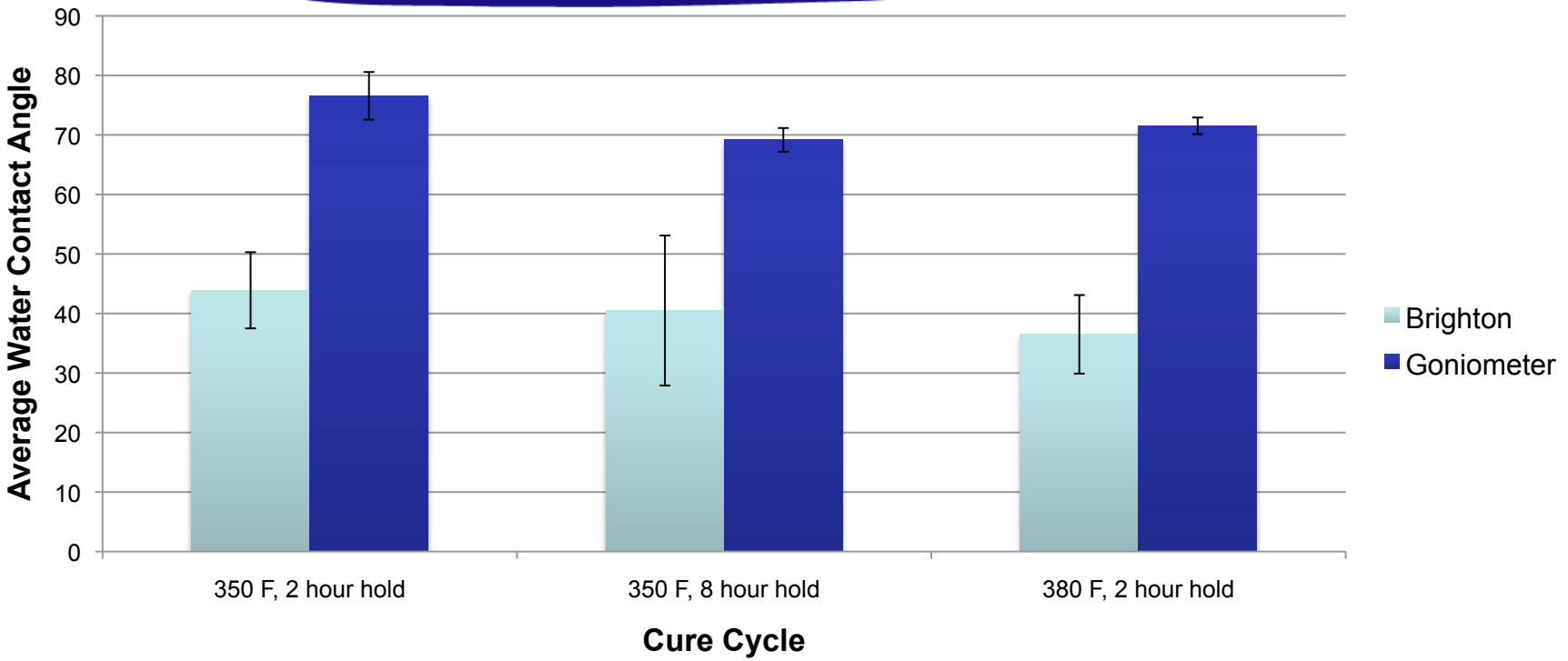


- **Different cure cycles resulted in different wettability envelopes**

Surface Energies

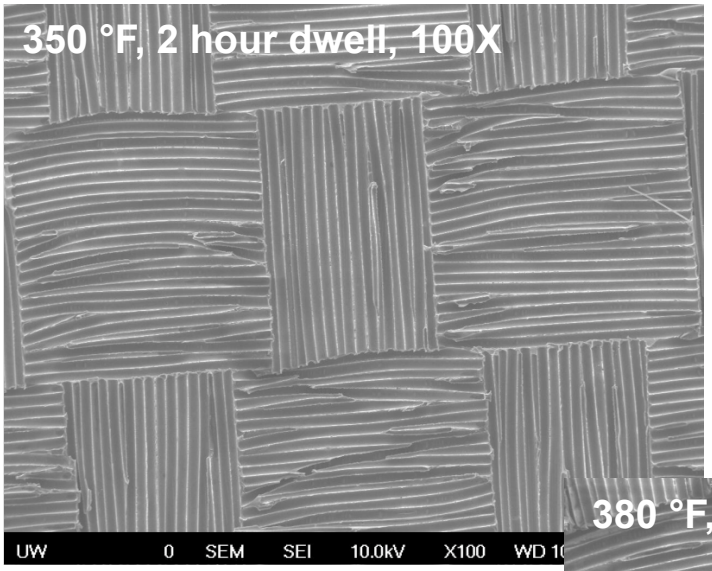


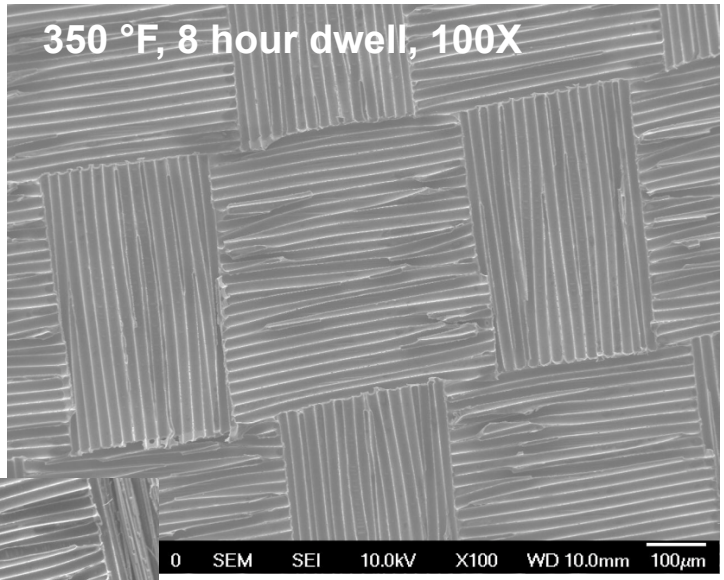
Brighton Device Preliminary Results

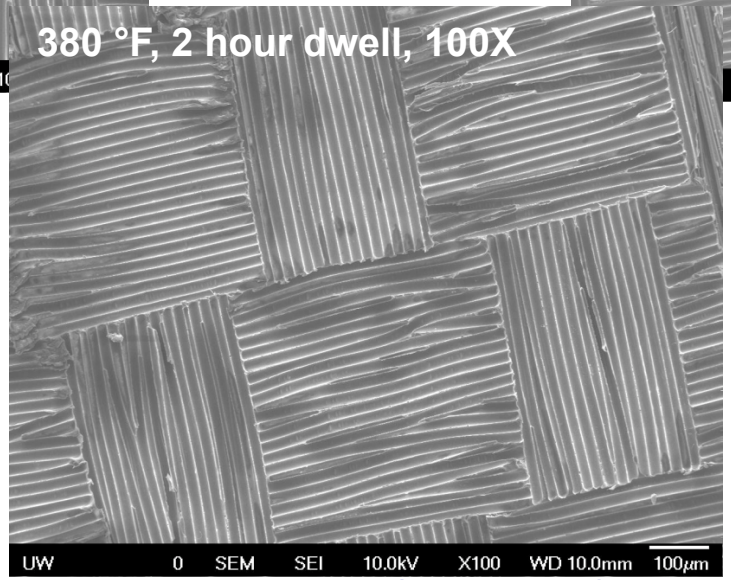


- Brighton measurements have larger standard deviations (new operator?)

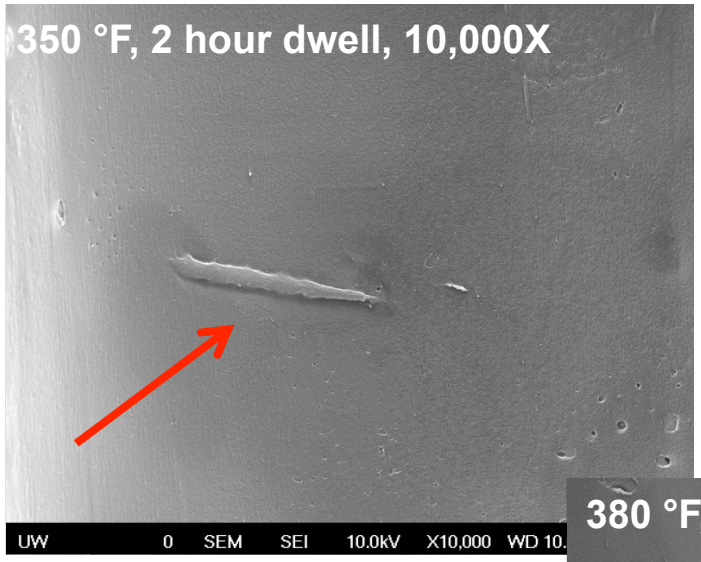
SEM Images of Substrates

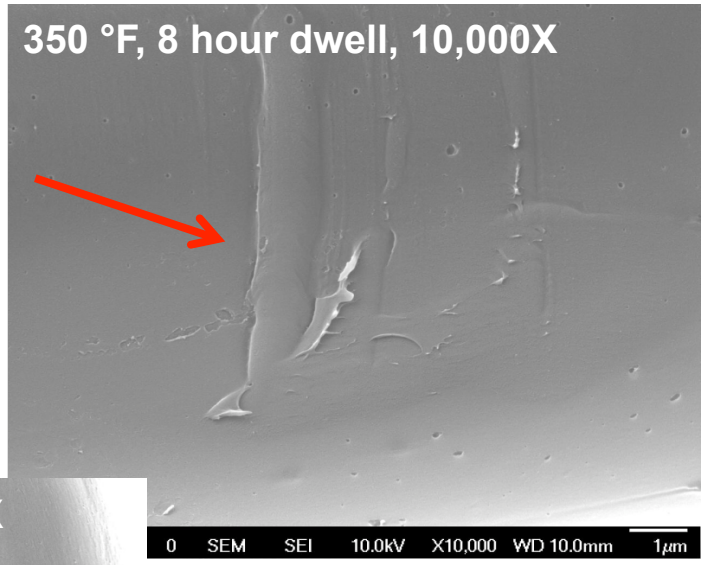


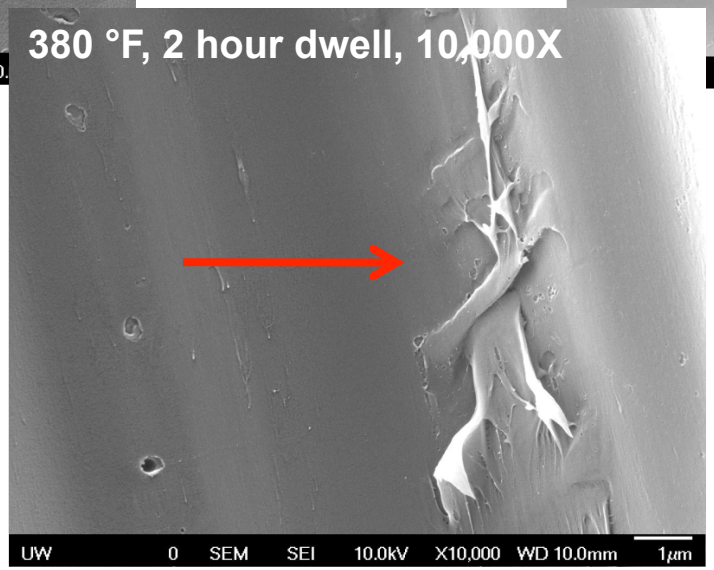




SEM Images of Substrates







- 350 °F, 2 hour dwell shows least amount of peel ply transferred to surface

- 380 °F, 2 hour dwell shows largest amount of peel ply transferred to surface

Conclusions

- Different cure cycles affect contact angle and hence wettability envelopes
  - SEM images show greatest peel ply transfer on 380 °F, 2 hour dwell and least amount of transfer on 350 °F, 2 hour dwell
  - Need data on fracture energy to see if can correlate contact angle measurements/wettability envelopes to bondability
- Brighton - potential QA technique for surface preparation

A Look 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
  - Surface characterization vs. bond quality model
  - QA method to ensure proper surface for bonding
  - Applicability to other composite and adhesive systems
  - Model to guide bonding based on characterization, surface prep. and material properties

Acknowledgements

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- Airtech International
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QUESTIONS ?
COMMENTS?
SUGGESTIONS?