Improving Adhesive Bonding of Composites Through Surface Characterization

Ashley Tracey, Jake Plummer & Brian D. Flinn

October 21, 2010
FAA Sponsored Project Information

- **Principal Investigators & Researchers**
  - Brian D. Flinn (PI)
  - 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
  – 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

VCA Optima Goniometer
- Desktop device
  - Lab research

http://www.btgnow.com
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 $\theta$ approaches zero
- Contaminants usually lower the solid’s surface energy (increase $\theta$)
- Surface preparations try to increase the solid’s surface energy and clean off contaminants
• 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

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

The Joint Advanced Materials and Structures Center of Excellence
Different cure cycles resulted in different wettability envelopes.
Surface Energies

<table>
<thead>
<tr>
<th>Cure Cycle</th>
<th>Polar Energy</th>
<th>Dispersive Energy</th>
<th>Total Surface Energy</th>
</tr>
</thead>
<tbody>
<tr>
<td>350 F, 2 hr hold</td>
<td>5</td>
<td>40</td>
<td>45</td>
</tr>
<tr>
<td>350 F, 8 hr hold</td>
<td>5</td>
<td>40</td>
<td>45</td>
</tr>
<tr>
<td>380 F, 2 hr hold</td>
<td>5</td>
<td>40</td>
<td>45</td>
</tr>
</tbody>
</table>
Brighton Device Preliminary Results

- Brighton measurements have larger standard deviations (new operator?)
SEM Images of Substrates

350 °F, 2 hour dwell, 100X

350 °F, 8 hour dwell, 100X

380 °F, 2 hour dwell, 100X
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

The Joint Advanced Materials and Structures Center of Excellence
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.
  – 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

- FAA, JAMS, AMTAS
- Boeing Company
- Precision Fabric Group
- Richmond Aircraft Products
- Airtech International
- Prof. Mark Tuttle (UW)
QUESTIONS ?
COMMENTS?
SUGGESTIONS?