Improving Adhesive Bonding of Composites Through Surface Characterization

Proposed FAA Sponsored Research for JAMS/AMTAS 2008-09

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Executive Summary

- Proposed research supports the FAA's mission
- Industrial interactions in place - more welcome!
  - Support of relevance of research
  - Industry input on research results and directions
- 4 sub-topics proposed relate bond quality to physical characteristics of composite surfaces prepared for bonding
- Contribute to fundamental understanding of surface chemistry and bond quality relationships
- Apply results towards an inspection technique for surface preparation of composites
- Modest budget supports 1 Grad. Student & 1/12th Fac.

FAA Sponsored Project Information

- Principal Investigators & Researchers
  - Brian D. Flinn (PI)
  - Fumio Ohuchi (Co-PI)
  - Molly Phariss (Ph.D. Candidate, UW)
  - Jeff Satterwhite (Masters student, UW)
  - Curtis Hickmott (senior, UW)
  - John “Jack” Aubin (MS 2008, UW)
  - Conor Keenen (BSc 2008, UW)
- FAA Technical Monitor
  - Curtis Davies
- Other FAA Personnel Involved
  - Larry Ilcewicz
- Industry Participation
  - Boeing: Peter Van Voast, William Grace, Paul Shelly
  - Precision Fabrics Group, Cytec, Toray, 3M, Henkel
- JAMS Participation
  - Mark Tuttle: Technical Discussions, Wettability Envelopes

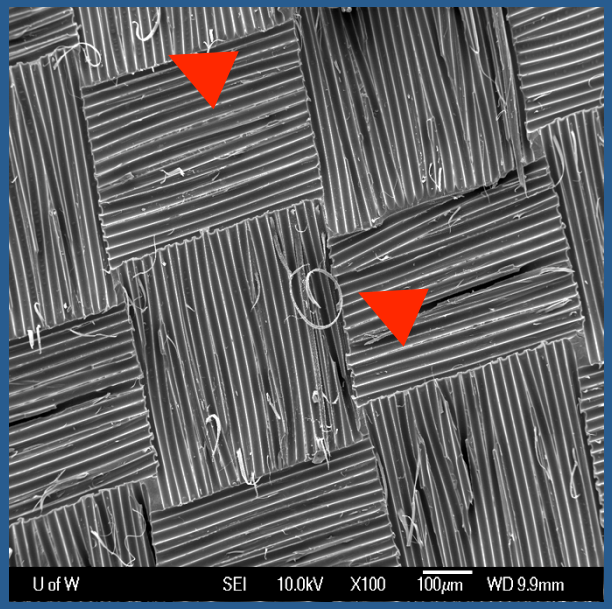
Improving Adhesive Bonding of Composites

- Research to date (years 1-3)
  - Investigated the effect of various surface preparation procedures and material systems on the adherend surface chemistry/structure and relate to subsequent bond performance
  - Materials & Methods:
    - Carbon Fiber Epoxies 127º C (260º F) & 176º C (350º F)
    - Glass Fiber Epoxies 127º C (260º F)
    - Surface Preparation: Sanding and Peel Plies
    - Adhesives: paste & film (127ºC (260ºF) and 176ºC (350ºF))
  - Characterization
    - Surface Chemistry, SEM, Contact Angle, Mechanical Testing and Fractography

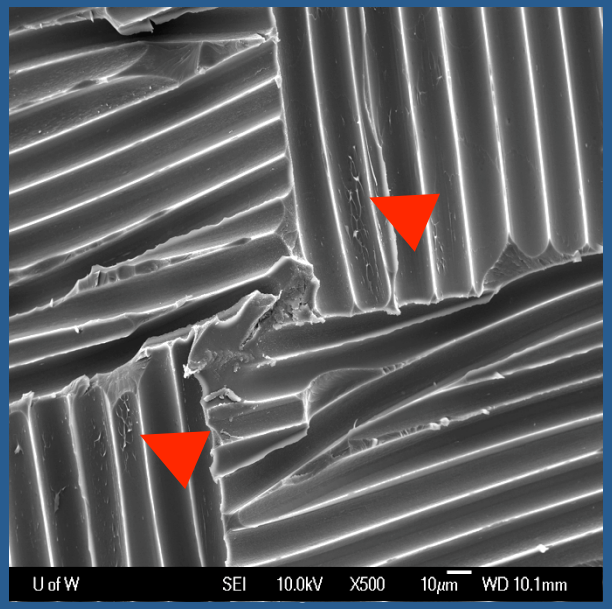
Surface Appearance

- Scanning Electron Microscope (SEM)
  - Detailed view of surface (10-100,000x)
    - Microscopic remnants of peel ply present? –**BAD**
    - Amount of “fresh” matrix exposed- **Good**
  - Limited to lab/process development settings
    - Not portable, trained operator, not cheap

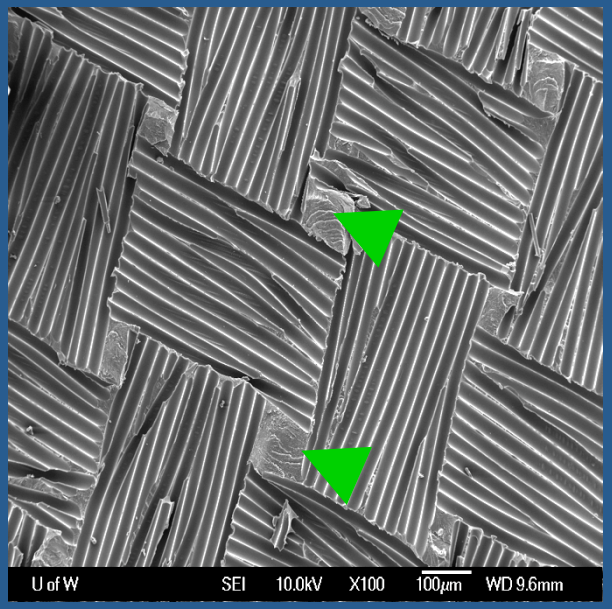
Cytec 970-dry polyester PP



Cytec 970-dry nylon PP

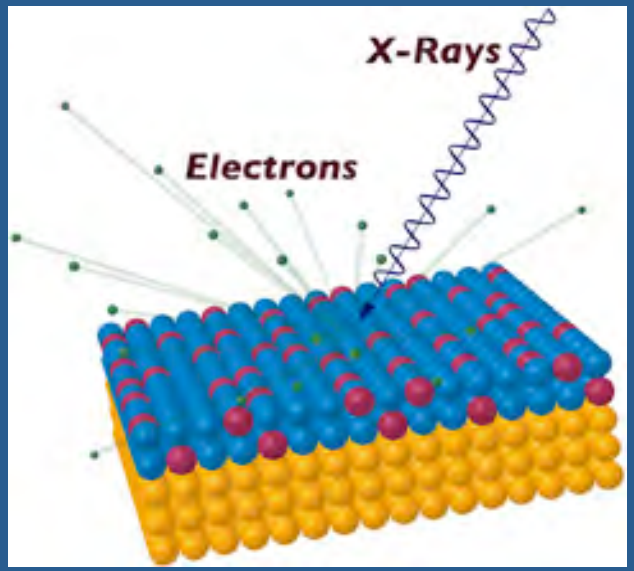


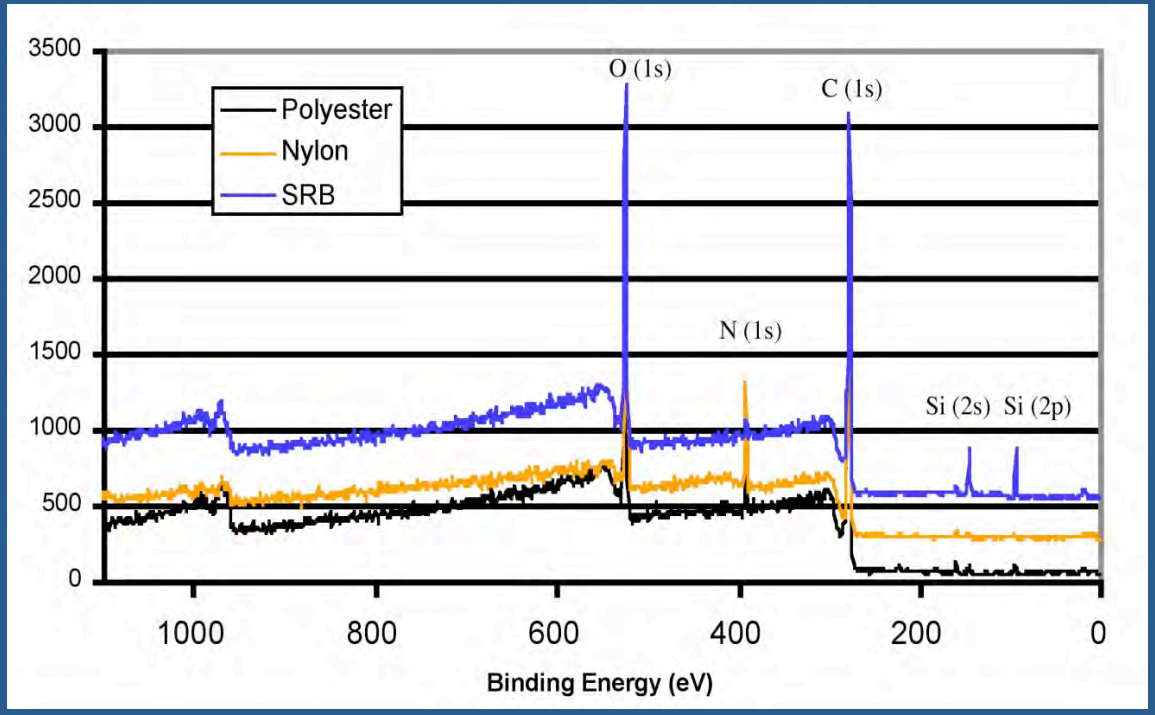
Cytec 970-EA9895



Surface Chemistry

- X-Ray Photo Spectroscopy (XPS/ESCA)
  - Chemical composition & functional groups of surface
  - Sensitive to small amounts of contamination
  - Critical tool for research- NOT for manufacture/repair
  - Expensive, time consuming, small specimen size

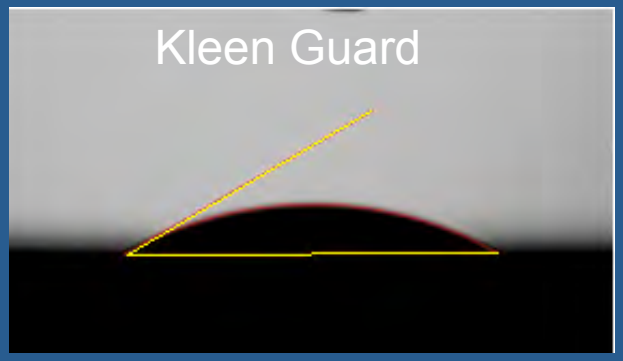


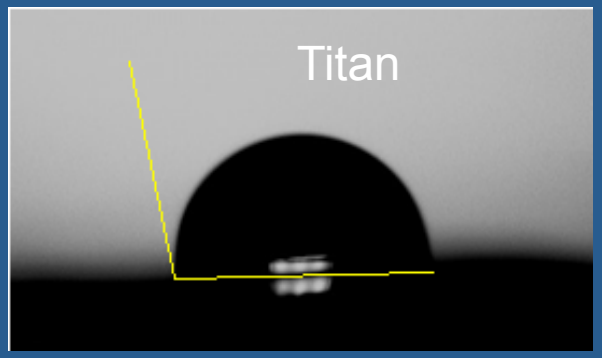


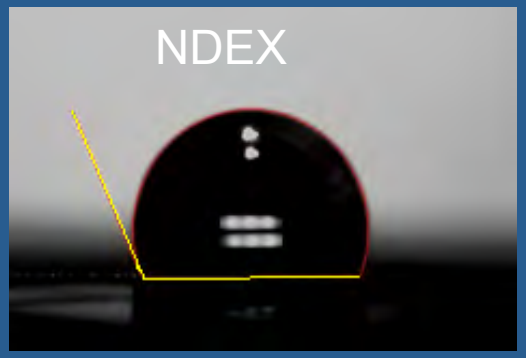
Video-detection of contamination with contact angle

Contact Angle has been shown to detect changes in surface energy due to contamination

	No Glove	Kleen Guard	Titan	NDEX
Glove surface	NA	29.1º	92.2º	104.2º
Composite surface	78.9º	69.8º	109.0º	115.3º



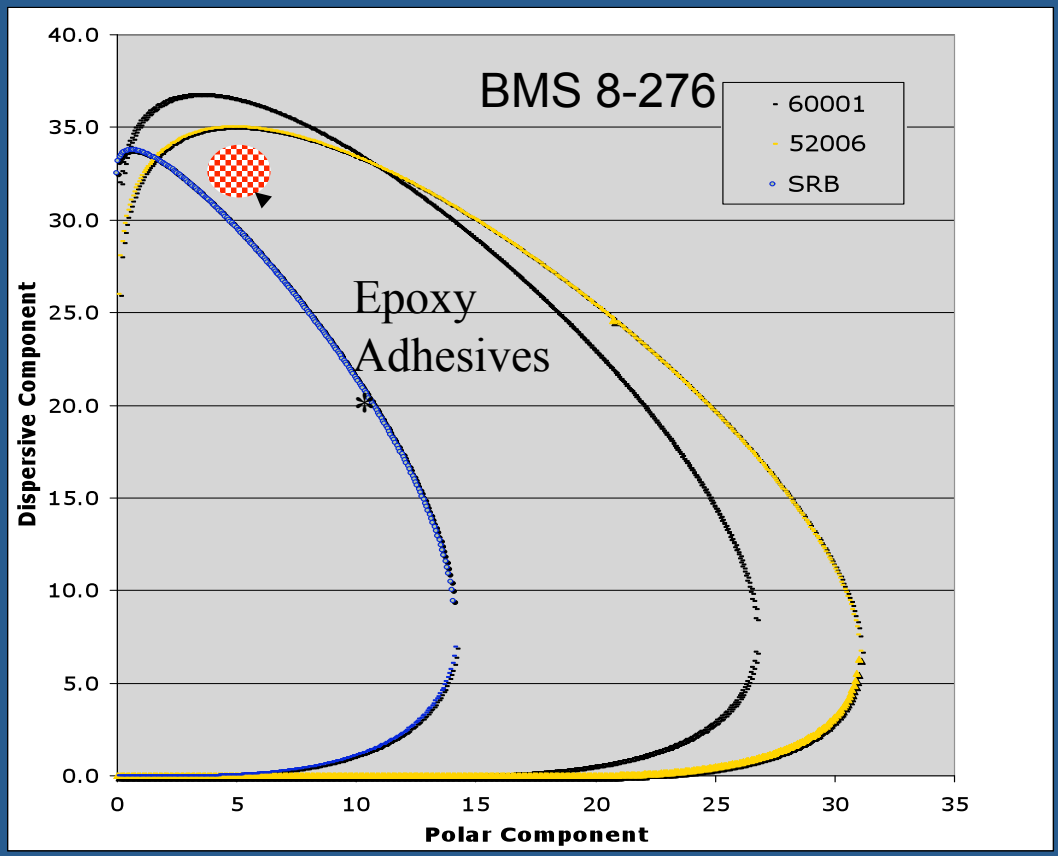




Contact angle of DI water on surfaces rubbed with different gloves.

Wettability envelopes showed the difference in the prepared surfaces.

- Fluids inside the envelope will wet spontaneously
  - Critical condition for bonding?
- Wettability envelopes a potential method to determine suitability of a surface for bonding
- Epoxy adhesives* on boundary for nylon prepared BMS8-276 surfaces
  - Predicted wetting is a necessary but not always sufficient condition for a strong

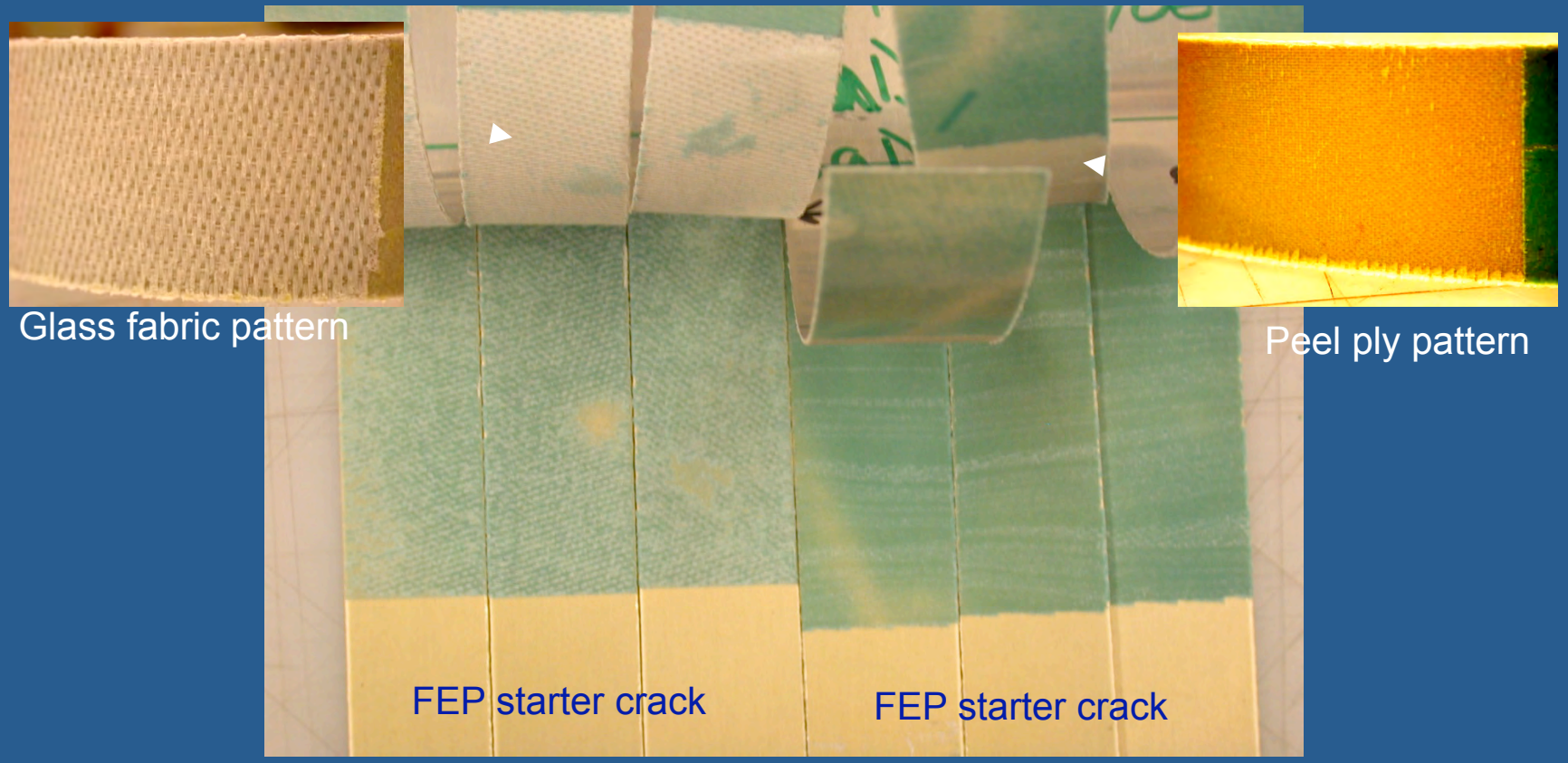


* Literature values for aerospace epoxies
- Curves generated using WET program (M. Tuttle)

Fracture Evaluation

- The only sure way to measure bond quality is to break it
  - Not very good business plan
  - Use process control to ensure reproducible bond quality
  - Standard Specimens: Lap Shear and DCB
    - Time & material consuming, requires test frame
  - "Quick" tests
    - Rapid Adhesion Test (RAT) method
    - Instrumented RAT (*i*-RAT)

RAT Method Assessment



Cohesive failure (left) vs. Adhesion failure (right)

Research To Date Summary

- No Single method to characterize surfaces to ensure a good bond
- XPS and SEM are valuable tools
- Contact angle/wetting can NOT predict bond quality
- Contact angle/wetting can detect contamination
- Strict process control is required (even type of gloves)
- Simple peel tests can be used to quickly (and at much lower cost) evaluate bond quality
- Of course… more research is needed

On to Proposed research…..

4 Proposed Topics

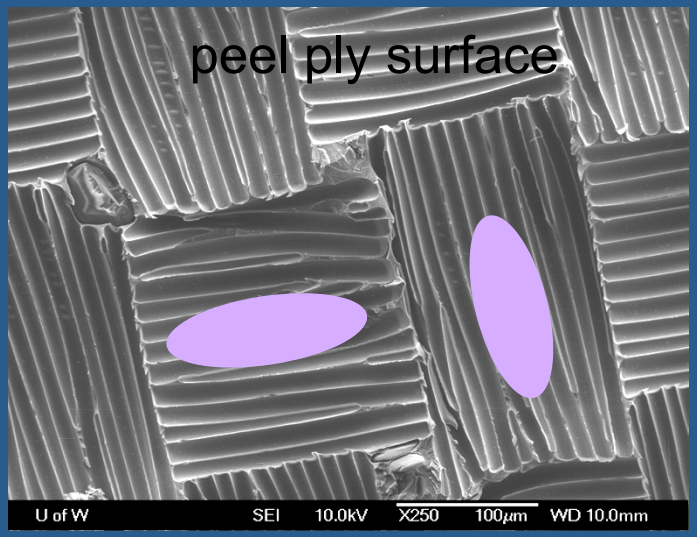
1. Variables that influence the measured surface energy/wettability envelopes
2. Surface and bonding characteristics of General Aviation sector (AGATE) materials
3. Evaluation of Brighton Technology Surface Energy Probe
4. Potential for contamination of surfaces by contact angle fluids

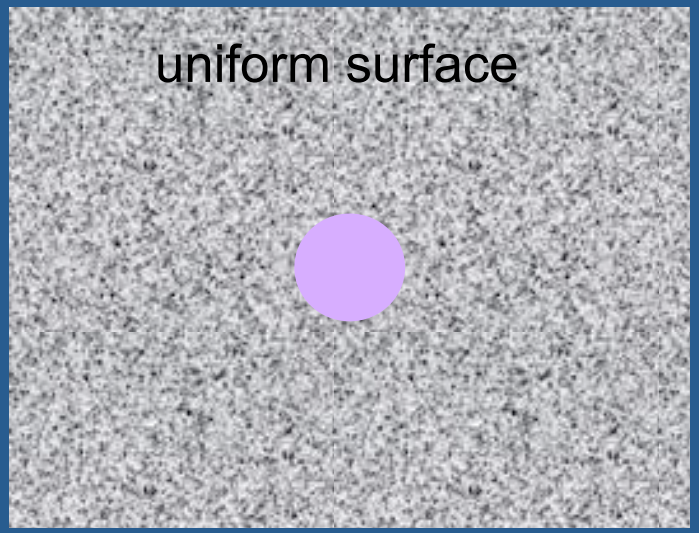
1. Variables that influence the measured surface energy/wettability envelopes

- Correlation between bond quality and surface energy and wettability envelopes has been marginal - WHY?
- Investigate possible effects of:
  - Effect of temperature on surface energy
    - Room Temp. Measurements vs. Elevated Temp. Cures
  - Analysis of contact angle measurements using a different wetting theory (Lewis Acid-Base model vs. Owens polar dispersive model)
  - Surface texture (especially peel ply) on measured contact angles
  - Different methods of determining Surface Energy

Surface Energy

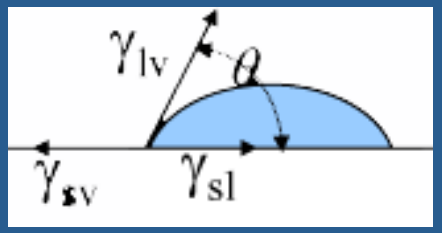
- Determine role of surface texture and orientation on measured contact angles

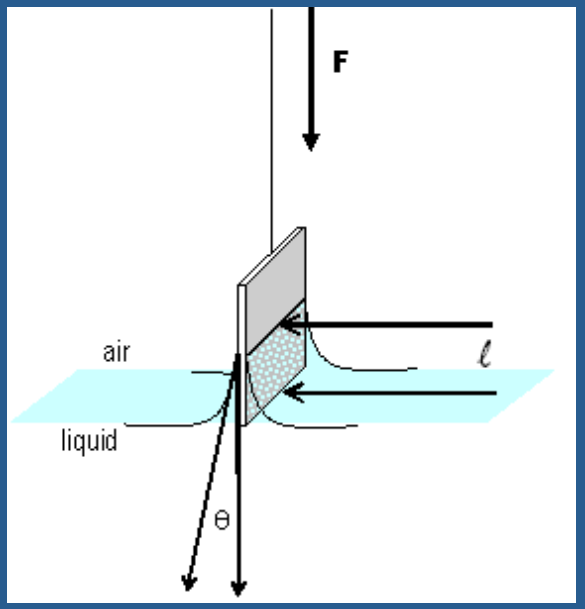




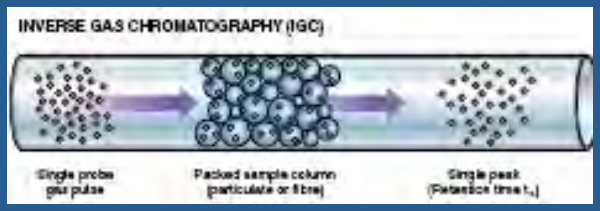
- How much error/scatter is introduced by texture?
- Can it be accounted for ?

Surface Energy Measurement





- Contact Angle
  - quick & easy
  - small area
- Wilhelmy plate (tensiometry)
  - larger surface area measured
- Inverse Gas Chromatography (IGC)
  - different measurement principle



- How similar are results?
- Is one technique more applicable to bonding?

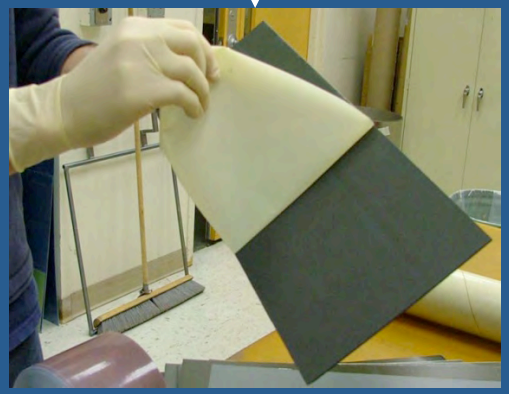
2. Surface and Bonding Characteristics of General Aviation Sector (AGATE) Materials

(Toray 2510 resin based prepreg)

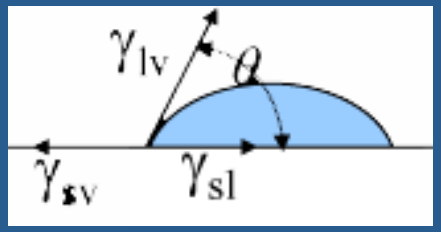
- Our research has concentrated on commercial aircraft materials (mostly BMS) and film adhesives
- Apply research protocols to different class of composites- Out of Autoclave, paste adhesives
- Benefits:
  - Increase knowledge base and test applicability of findings- better fundamental understanding
  - Safety and reliability of bonded structures in

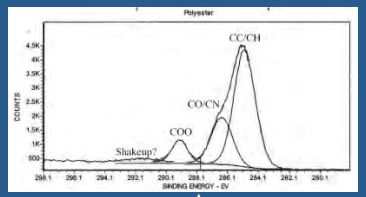
Research Protocol

Cure

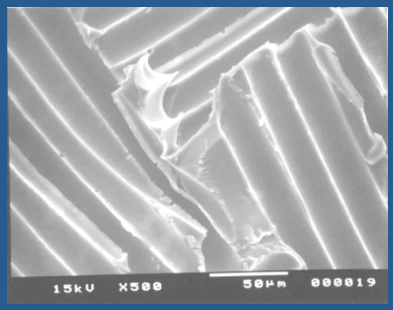


Surface Preparation



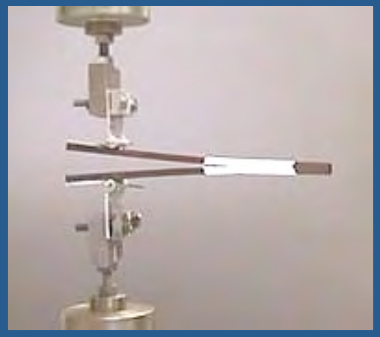


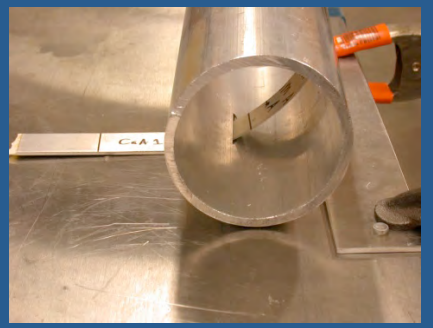
Characterization Via XPS, SEM, Contact Angle



Bonding

Cure





Mode I testing

3. Evaluation of Brighton Technology Probe

- Prototype hand held surface energy probe has been developed
- Surface energy measurement determined from contact angle of sessile drops
  - Top down view of drops
  - Used to characterize surface
  - Proposed as a inspection technique
- Device needs to be evaluated for sensitivity to:
  - Process deviations (e.g. wrong peel ply)
  - Detection of contamination
  - Choice of FIuids
  - Operator

4. Contamination by Contact Angle Fluids

If contact angle is used as an inspection technique:

- Are the fluids used to make contact angle measurements detrimental to bond quality?
- What level of cleaning after CA measurement is required?
- Evaluate effect of several common fluids using established research protocols and materials
  - Surface energy
  - XPS
  - Bond quality

Industry Interactions

1) Detailed technical interactions will continue with Peter Van Voast and Will Grace of the Boeing company concerning the fundamental understanding of surface characterization and bond quality
2) Investigate bonding to GA composite materials by working with composite material suppliers. Specifically Toray Composite America, Cytec Engineered Materials, 3M and Henkel have supplied materials and are willing work cooperatively on this research.
3) Input on materials, processes, and relevant issues will be solicited from General Aviation OEM's that use composite bonding. OEMS will also be invited to review and cooperate on the research.
4) Cooperate with Brighton Technologies Group and evaluate the technology they are developing to assess surface preparation for composite material bonding.
5) Future opportunities

YOUR TURN !

- QUESTIONS
- COMMENTS
- SUGGESTIONS
- DISCUSSION