# Improving Adhesive Bonding of Composites

- Principal Investigator and Researchers
  - Brian Flinn (PI)
  - Rita Johnson (UW MSE/Boeing)
  - Marc Staiger (UW MSE)
  - Seth Nichols, Dylan Faherty (UW MSE)
- FAA Technical Monitor
  - Curtis Davies and David Westlund
- Other FAA Personnel
  - Larry Illecwicz
  - Cindy Ashforth
- Industry Participation
  -

# Two projects will presented today

1. Amine Blush in Epoxy Paste Adhesives
   - Characterization of Bondline

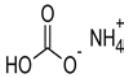
# Amine Blush in Epoxy Paste Adhesives

- Project Background
- Test Methods and Design
- Current Results
- Upcoming Work

# Motivation and Key Issues

- Bond failures have been attributed to amine blush
  - Cessna incident (2010)

- What are the conditions for blush formation?
- What are the effects on bond quality?
- Investigate blush NDI methods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# Project Outline

1. Expose epoxy paste bonds to a range of environmental conditions and analyze the effect on bond strength.

2. Perform FTIR analysis on traveler coupons to determine if blush formation can be detected and quantified.

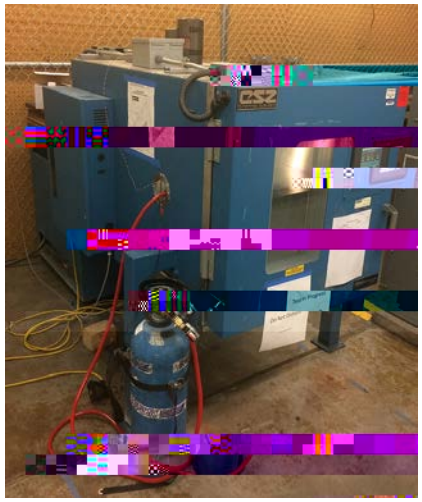
3. Relate the mechanical response of the bond relative to any measured blush formation.

# Project Test Plan

1. Adhesive: Henkel Loctite EA 9360
   - Two part epoxy – amine system

2. Experimental Exposure Variables
   - Temperature (60 – 90 F)
   - Humidity (50 – 85 %RH)
   - $CO_2$ (500 – 5000 ppm)
   - A & B component exposure time (0 – 3 weeks)
   - Bondline exposure time (20 – 50 min)
   - $2^{5-1}$ Factorial DOE

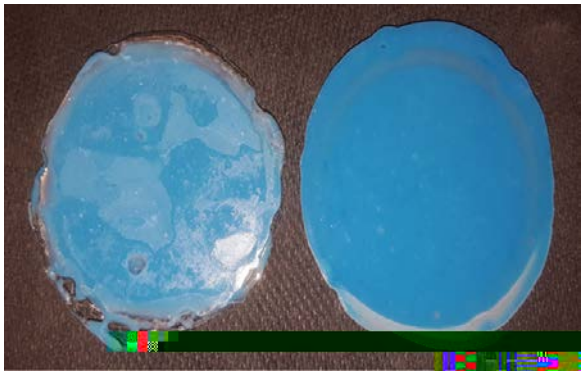
3. Test Methods
   - FTIR – Amine Blush Measurement
   - T-Peel – Bond Quality Measurement
   - DCB – Bond Quality (In progress)

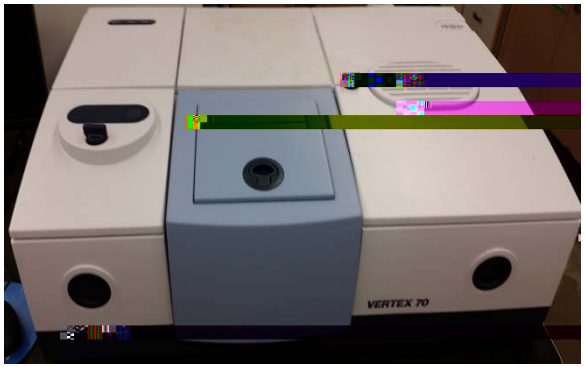


Exposed Amine Component (Left)
Environmental Conditioning Chamber (Right)

# FTIR Analysis

- Testing with benchtop ATR FTIR
  - Samples exposed alongside mechanical samples
  - Samples closed out after designated exposure time
- Establishment of quantification procedures
- Comparisons to reference literature





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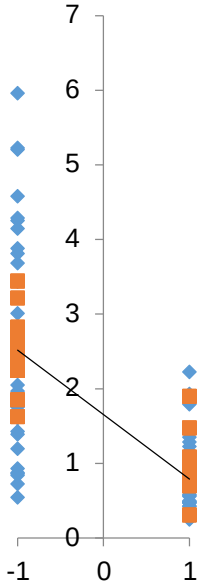
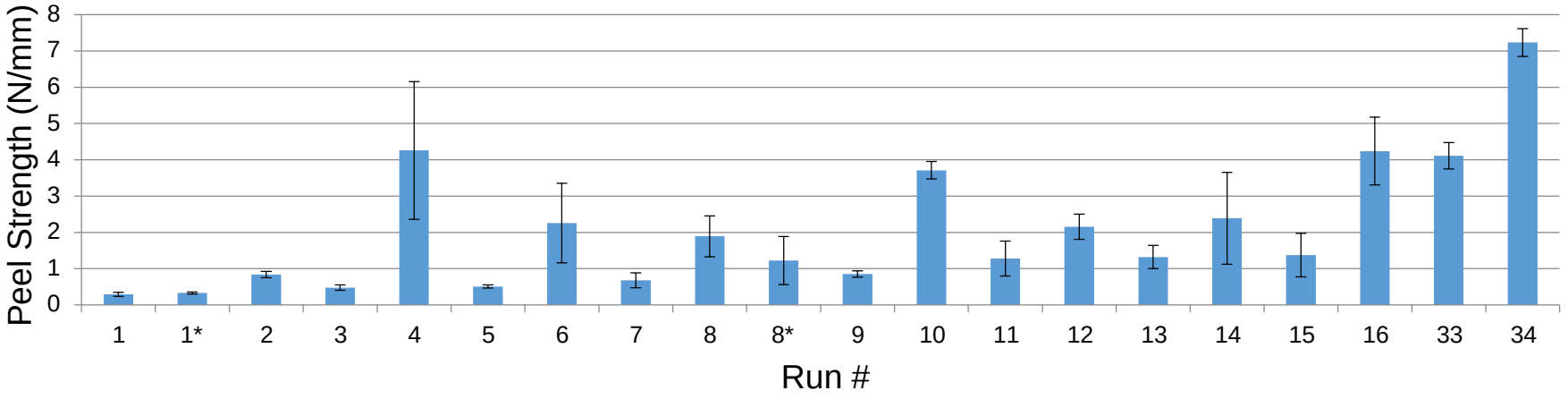
# Mechanical Testing Results

T-Peel e B 92.136 334.68 5.04 -5.04 re B 27.



# Mechanical Testing Results

1. Temperature was strongest factor for decrease in strength
2. Other significant negative factors:
   - Bondline Time Exposure
   - Part A&B Time Exposure
3. $CO_2$ had no measureable effect on t-peel strength
4. Humidity (50-85%) had no measurable effect on t-peel strength.
5. Combination effects are being analyzed.

| Regression Statistics | |
|---|---|
| Multiple R | 0.759 |
| R Square | 0.576 |
| Adjusted R Square | 0.543 |

# FTIR Results

- **Notable difference between control and exposed samples**

# FTIR Results

# Discussion

- How can we correlate FTIR measurements to mechanical properties?
  - Can FTIR Traveler coupons represent bondline?
    - Direct measurement of fracture surface.
    - Measure FTIR before and after cure.
  - Blush Thickness
    - FTIR detection may not reflect the amount (thickness) of blush.
    - May be detecting thin layers that are not detrimental to bond quality.
  - Bonding Procedure
    - Adhesive squeeze out may break up thin layers of blush

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# Looking forward- Amine Blush

# MOTIVATION AND KEY ISSUES

- Most important step for bonding is surface preparation
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# MEASURING SURFACE ENERGY

# IGC VS. CONTACT ANGLE

Contact Angle (CA)

- Small drops (1 ml) of 3-5 known liquids placed on surface
- Surface energy calculated over small area (order of mm2)
- Can be affected by surface texture (non-circular drops)
- Quick, inexpensive, can be portable

Inverse Gas Chromatography (IGC)

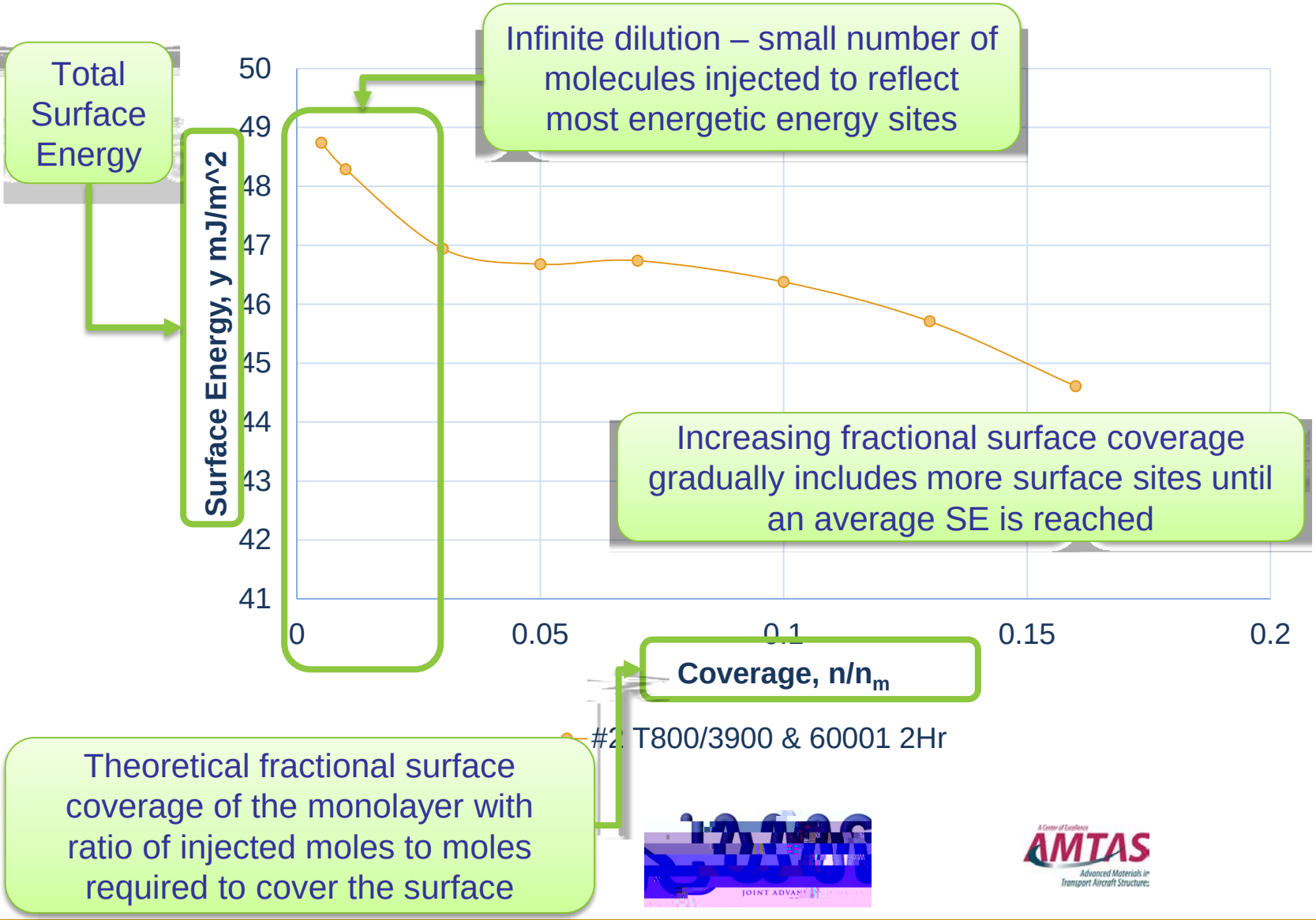
- 8-10 Known gases flow over surface
- Larger area sampled (2”X8”)
- More information obtained (higher fidelity data)
- Distribution of surface energy
- Greater sensitivity to subtle changes
- Expensive equipment, skilled operator

# IGC METHODOLOGY

| Variable | Description |
|---|---|
| Probe Gases | Undecane, Decane, Nonane, Heptane, Dichloromethane, Ethyl Acetate, Acetonitrile, Acetone |
| Targeted Fractional Surface Coverage | |

0.01,

# IGC Surface Energy Profiles



# PILOT STUDY EXPERIMENTATION

## Test Specimens:

| Panel ID# | Adherend (Fabric, Prepreg) | Peel Ply | Cure Dwell |
|---|---|---|---|
| 1 | 3900/T800 | 60001 Polyeste l | S Q q 1 |
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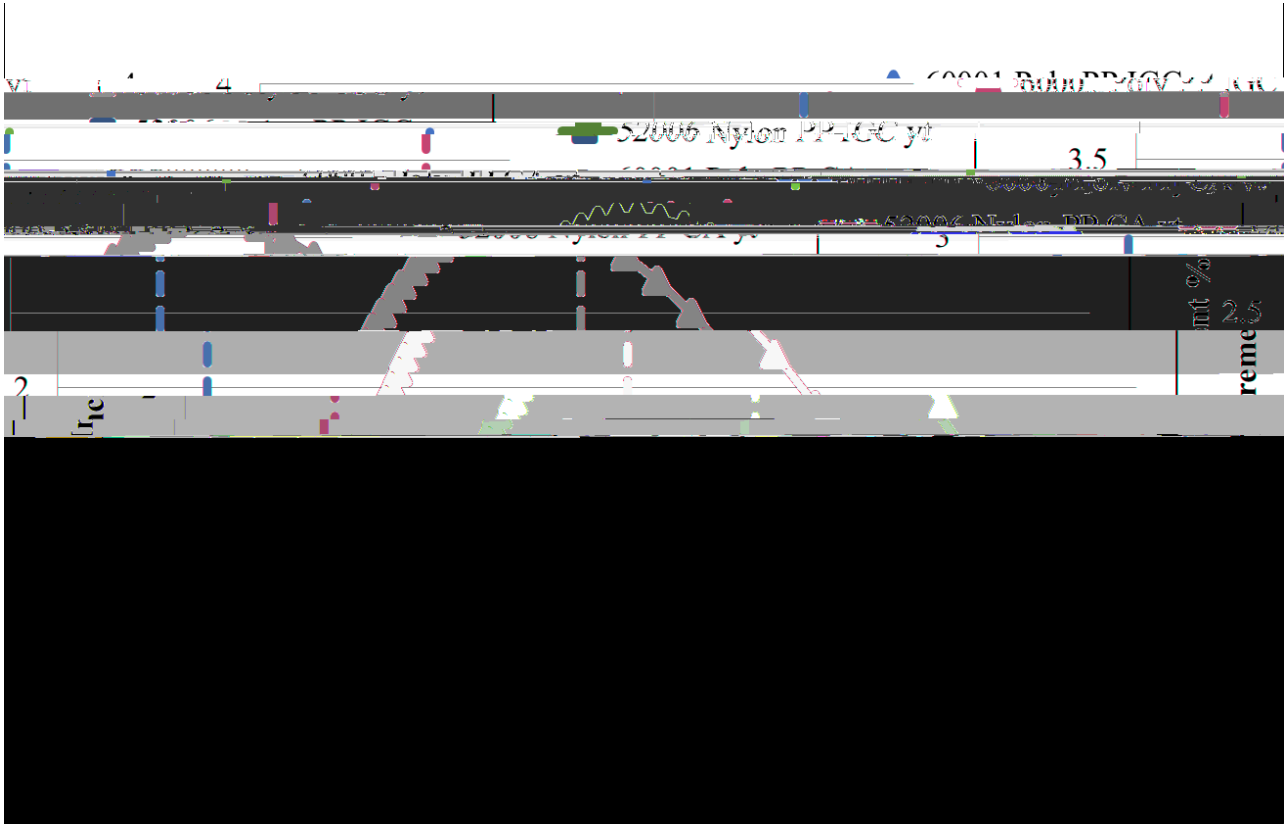
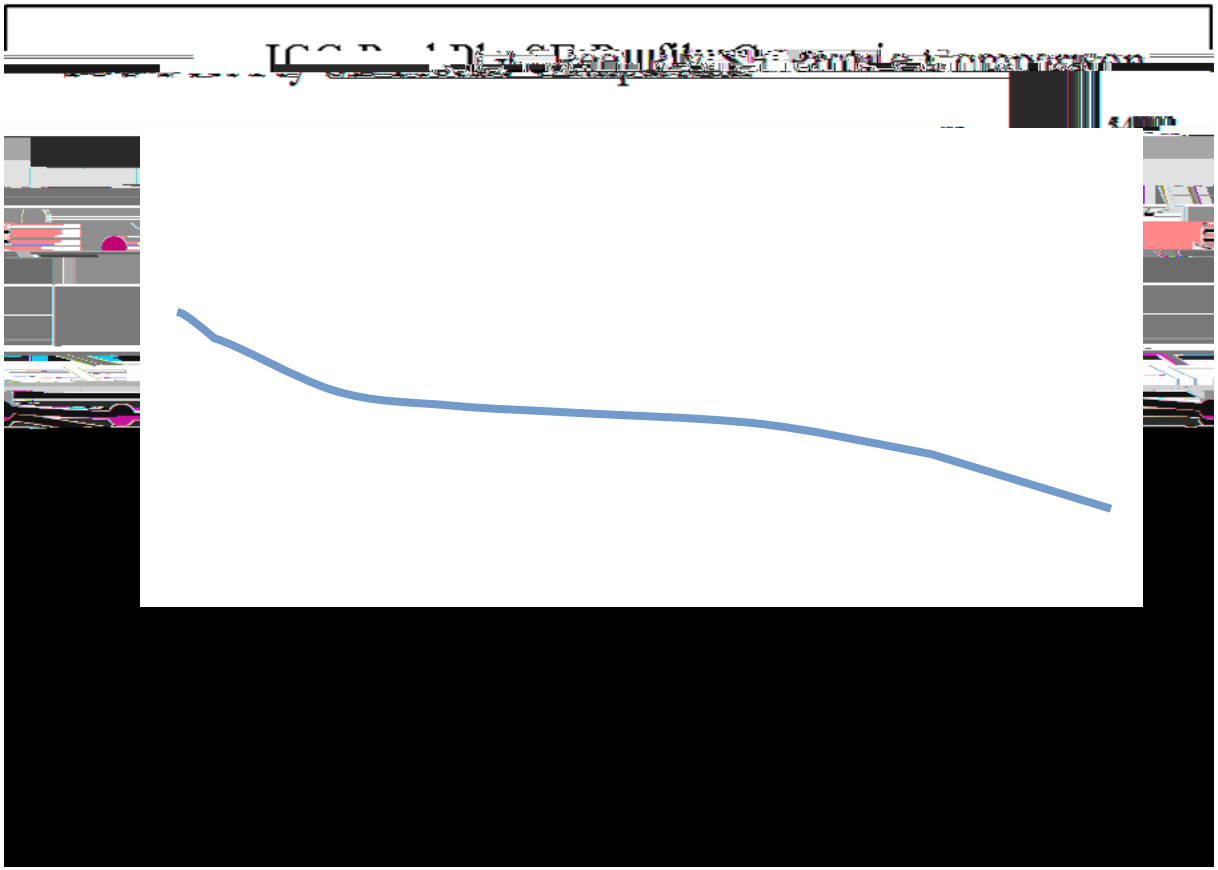
# IGC AND CA COMPARISON




1. Nylon and Polyester have significantly different distributions according to IGC
2. Contact angle is controlled by complex wetting phenomena
3. Contact angle correlation to the IGC data is different for each peel ply type



# Pilot Study Conclusions/Discussion

- Contact angle measurements result in a single value of surface energy (

# Current Work

- Understand the advance models of wetting versus gas interactions
- Characterize additional surface preparation methods with IGC

Priority Testing

- Relate surface preparation to bond quality types
- Additional statistical data and material coupon testing for a more complete representation of the bonding surface
  - X-ray photoelectron spectroscopy (XPS)
  - Scanning electron microscopy (SEM)
  - Double cantilever beam (DCB)

Although IGC is able to provide more information on surface energies related to various surface preparations techniques, other components contributing to the quality of the bonding surface need to be investigated.

# Current Work

- Surface Preparation Methods (in order of research priority)
  - Plasma Treated
  - Grit Blasted
  - Orbital Sanding
- Surface Energy Testing
  - IGC
  - Advanced Contact Angle Measurements
    - Static, advancing and receding

| 2016 | 2017 | | |
|---|---|---|---|
| Q4 | Q1 | Q2 | Q3 |
| Materials Selected | Panel Fabrication | | |

# Current Work

| Panel # | Substrate | Surface Prep | Cure Dwell | Comments |
|---|---|---|---|---|
| 1 | Toray's 3900/T800 UnidirectionalPrepreg | 60001 | 2 | Baseline for plasma treatment |
| 2 | Toray's 3900/T800 FabricPrepreg | 60001 | 2 | Baseline for plasma treatment |
| 3, 4, 5 | Toray's 3900/T800 UnidirectionalPrepreg | 60001 & Plasma Treated | 2 | 3 panels for 3 separate IGC runs |
| 6, 7, 8 | Toray's 3900/T800 FabricPrepreg | 60001 & Plasma Treated | 2 | 3 panels for 3 separate IGC runs |
| 9 | Toray's 3900/T800 UnidirectionalPrepreg | FEP | 2 | Baseline for sanding and grit blasting |
| 10 | Toray's 3900/T800 FabricPrepreg | FEP | 2 | Baseline for sanding and grit blasting |
| 11, 12, 13 | Toray's 3900/T800 UnidirectionalPrepreg | FEP & Grit Blasted | 2 | 3 panels for 3 separate IGC runs |
| 14, 15,16 | Toray's 3900/T800 FabricPrepreg | FEP & Grit Blasted | 2 | 3 panels for 3 separate IGC runs |
| 17, 18, 19 | Toray's 3900/T800 UnidirectionalPrepreg | FEP & Orbital Sanded | 2 | 3 panels for 3 separate IGC runs |
| 20, 21, 22 | Toray's 3900/T800 FabricPrepreg | FEP & Orbital Sanded | 2 | 3 panels for 3 separate IGC runs |
| 23, 24, 25 | Toray's 3900/T800 FabricPrepreg | 60001 & Plasma Treated | 6 | Contingency testing if time/funding permits |

Priority Testing

CECAM JAMS AMTAS

# Looking Forward

## Surface Characterization using iGC Methods

- Benefit to Aviation
  - Better understanding of surface prep.
  - Guide development of QA methods for surface prep.
  - Greater confidence in adhesive bonds
- Future needs
  - Surface energy (wetting) vs. bond quality
  - Surface energy at cure temperature
  - 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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# Questions?


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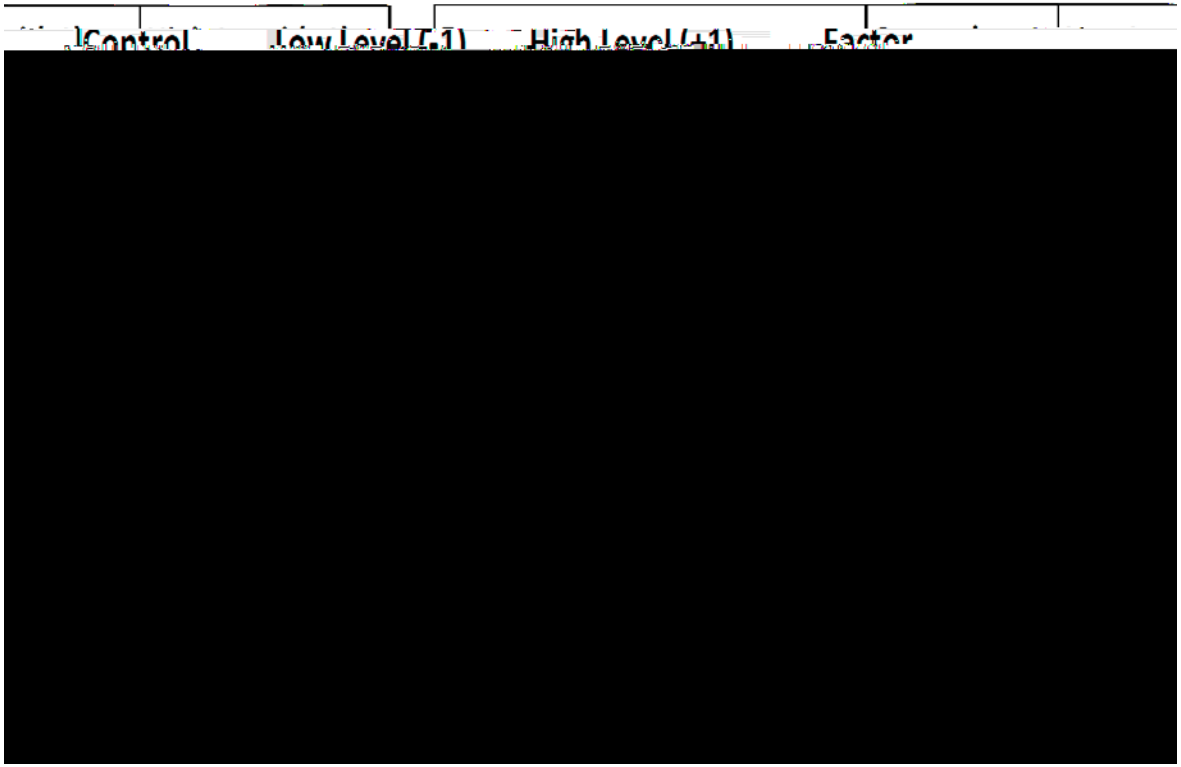
# DOE Test Matrix



# Potential Areas to Explore

1. Bonding procedure
   - Mound vs even spread
   - Using vacuum to reduce voids
2. Quantifying exposure of parts A and B
3. FTIR Measurement Procedures.
   - Direct measurement before cure
   - Direct measurement of fracture.
   - Use thinner surface samples to match bondline.
   - Apply chemometric analysis to analyze changes in spectra.

# IGC Overview

- Technique to characterize physicochemical properties of materials
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