Infrared Spectroscopy: A Potential Quality Assurance Method for Composite Bonding

2012 Technical Review

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Materials Science and Engineering

- Motivation and Key Issues
  - Most important step for bonding is SURFACE PREPARATION!!
  - Inspect surface prior to bonding to ensure proper surface preparation
- Objective
  - Develop QA technique for surface preparation
- Approach
  - Use FTIR to characterize different surface preparations and levels of contamination
    - CFRP prepared with peel ply for secondary bonding
    - Peel ply contamination
    - Manual abrasion to remove peel ply texture from CFRP

Quality Control Methodologies for Surface Preparation Processes for Composite Bonding

- Principal Investigators & Researchers
  - Brian D. Flinn (PI)
  - Ashley Tracey (PhD student, UW-MSE)
  - Elise Santa Maria (undergraduate, UW-MSE)
- FAA Technical Monitor
  - David Westlund
- Other FAA Personnel Involved
  - Larry Ilcewicz
  - Curtis Davies
- Industry Participation
  - Toray Composites
  - Precision Fabrics & Richmond Aerospace & Airtech International
  - The Boeing Company (Kay Blohowiak, Peter Van Voast, William Grace, Tony Belcher, Paul Vahey, Paul Shelley, Greg Werner and Marc Piehl)

Composite Bonding and Surface Preparation

- Peel ply is a desirable surface preparation because it produces repeatable and consistent surfaces and can prevent surface contamination
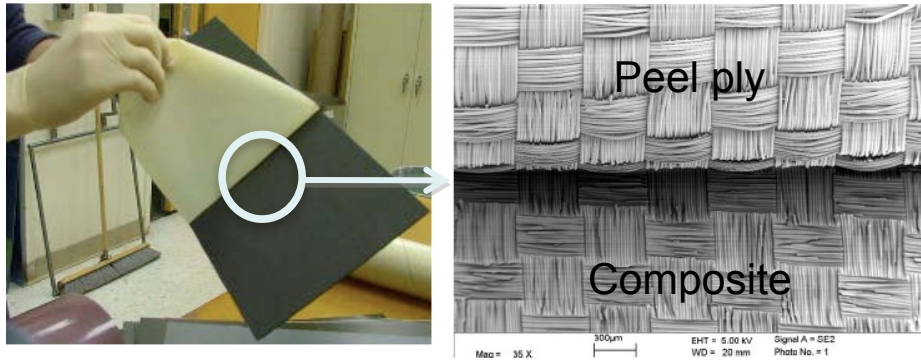
- Paste adhesives not compatible with peel ply surface → further surface treatment required

- Composite bonds are materials system specific (prepreg/ surface prep/adhesive) → potential problems that could degrade bond quality:
  - Incorrect peel ply?
  - Contamination?
  - Improper abrasion?

FTIR to Examine Surfaces

- Why might FTIR be used for QA?
  - Requirement of adhesion:
    - formation of primary chemical bonds between the adherend and adhesive
  - FTIR is used to measure surface chemistry
    - surface prep and contaminants influence surface chemistry
  - Portable units available
    - Suitable for factory and field applications

- Need to assess ability of FTIR as a QA technique

Assess ability of FTIR to identify improperly prepared surfaces

- Can FTIR detect different peel or release ply materials?

    – Nylon, polyester, SRB

- What level of siloxane detectable with FTIR?

    – Various levels of contamination (0.1%-2% and SRB)

- Can FTIR detect proper abrasion?

    – Different levels of manual abrasion to remove peel ply texture/residue

FTIR Methodology

Bruker Vertex 70 FTIR

- Bench-top instrument
- Diamond attenuated total reflectance (DATR)

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

Agilent Technologies Exoscan FTIR

- Handheld device
- Diffuse reflectance

**www.chem.agilent.com**

DATR Methodology

- MIR data region: 4000 cm^{-1} to 400 cm^{-1}

- Data collection: 16 scans with 4 cm^{-1} resolution for background and specimen

- Pressure clamp used for intimate contact between crystal and sample → max pressure: 30 ksi
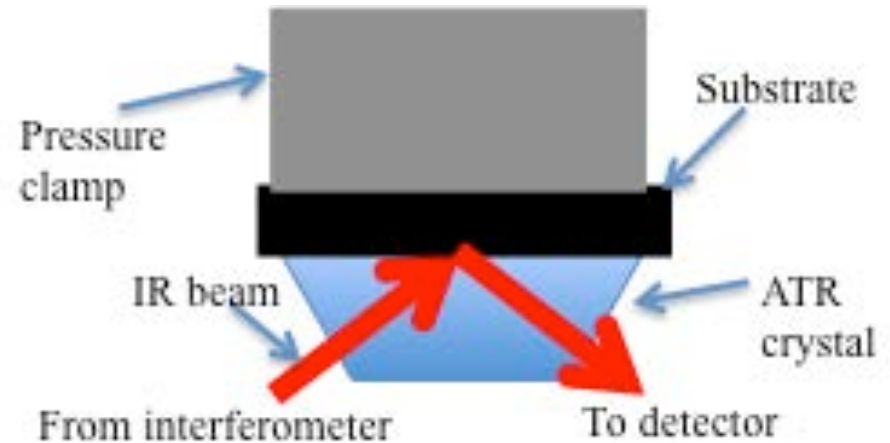
An infrared beam path for a single bounce DATR

Diffuse Reflectance Methodology

- MIR data region: 4000 cm^{-1} to 650 cm^{-1}

- Data collection: 90 scans with 16 cm^{-1} resolution for background and specimen
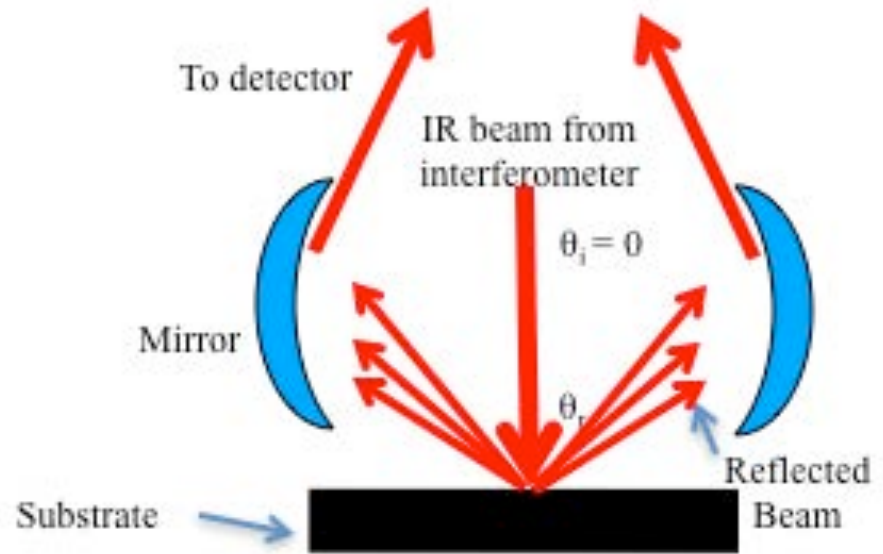
An infrared beam path for diffuse reflectance

Spectra Analysis

- FTIR spectra of CFRP surfaces complex
  - Multiple constituents → many spectral peaks
- How to analyze spectra with confidence?
  - Multivariate analysis!

- Multivariate analysis: Principal Component Analysis (PCA)
  - Identification of peak locations and intensities not obvious to the observer
  - Can be used to develop test interfaces to remove operator interpretation → go/no go output
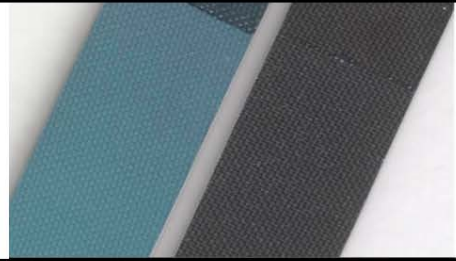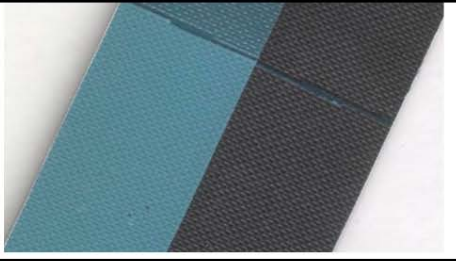
- Principal Component Analysis (PCA)
  - Exploratory data analysis – identify trends
  - 2 principal component analysis
  - Some spectra required preprocessing: Savitsky-Golay 1st derivative with 5 smoothing points fit to a 2nd order polynomial
    - Derivative to amplify changes in slope of spectral peaks while reducing effects of baseline offsets
    - Smoothing points to reduce influence of noise

Materials

- Toray 3900/T800 unidirectional laminates
  - Autoclave cure (177°C, 0.6MPa)
- Peel ply surface prep
  - Precision Fabric Group (PFG) 60001 polyester peel ply
  - PFG 60001 polyester peel ply with controlled siloxane amounts
    - 0.1% to 2% siloxane
  - PFG Super Release Blue (SRB) siloxane release ply
  - PFG 52006 nylon peel ply
- Manual abrasion surface prep
 - Random orbital sander with Diablo 120 grit ceramic abrasive sanding discs

- Peel ply surface preparation for adhesive bonding is materials system specific
  - Previous research showed CFRP prepared with different peel ply materials and bonded with MB 1515-3 film adhesive affect bond quality

	Polyester Prepared	Nylon Prepared	SRB Prepared
Failure Mode	Cohesive	Adhesion	Adhesion
G_{IC}	812 ± 35 J/m^2	$G_{IC} = 122 \pm 15$ J/m^2	$G_{IC} < 94$ J/m^2

➢ Need to ensure correct peel ply material used

DATR Sensitive to Peel Ply Material
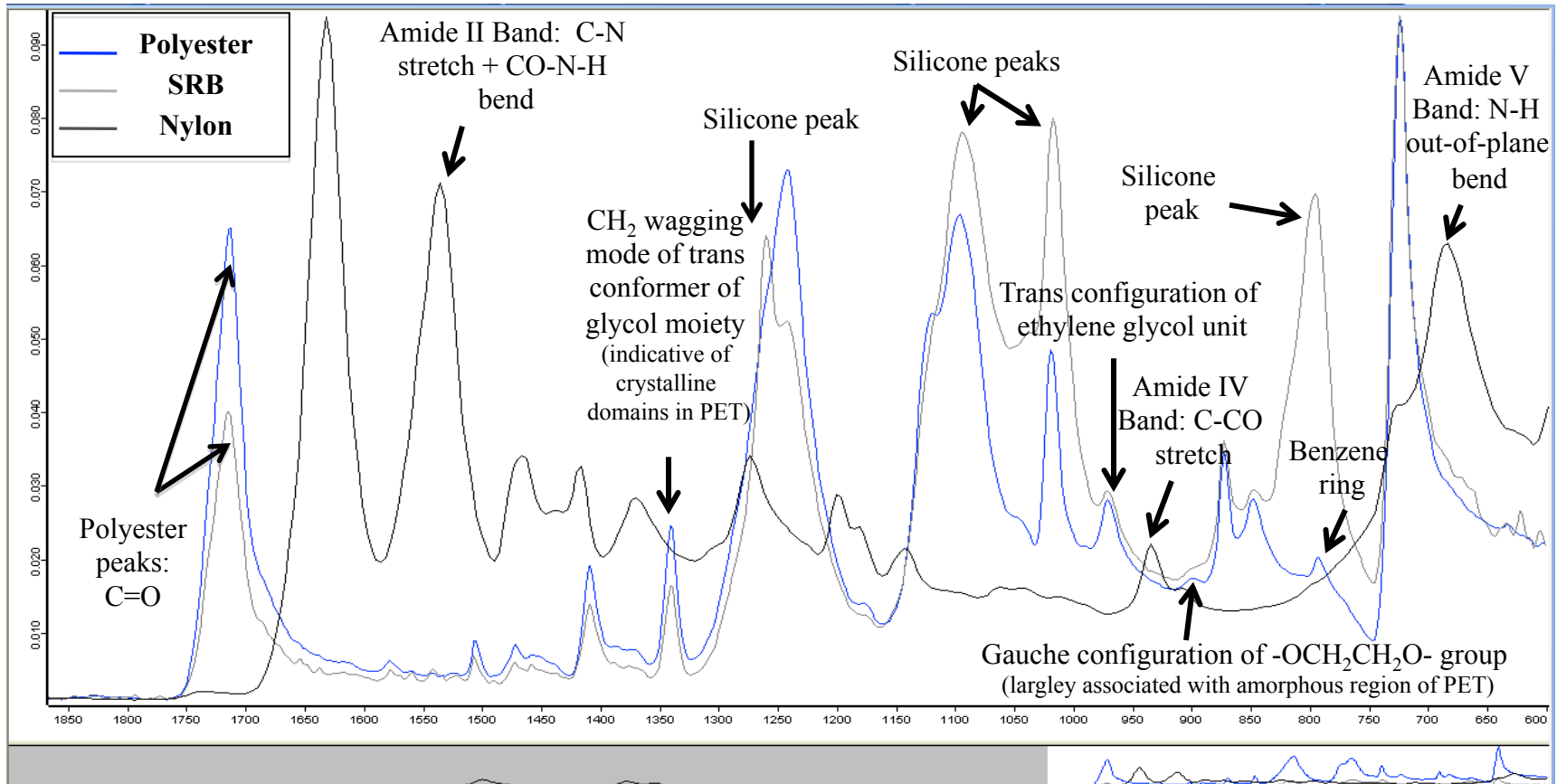
✓ DATR can differentiate between peel plies

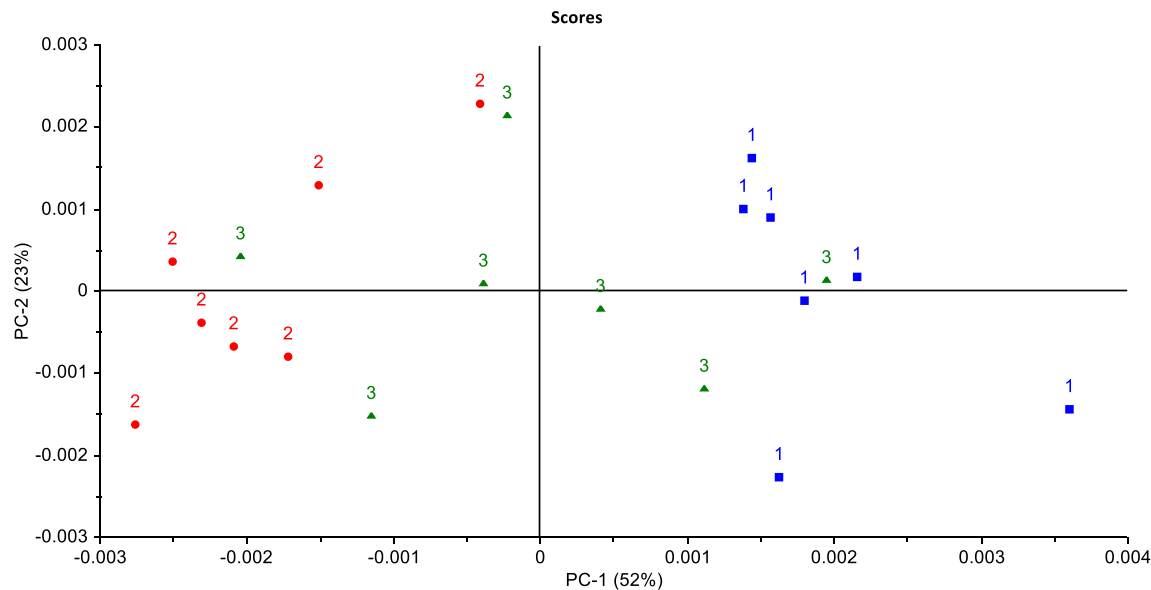➢ Peel ply prep detectable on CFRP surface?

DATR of CFRP Prepared with Different Peel Ply

Silicone band at 1260 cm^{-1} ?

Legend:
- Polyester prepared (green)
- SRB prepared (red)
- Nylon prepared (blue)
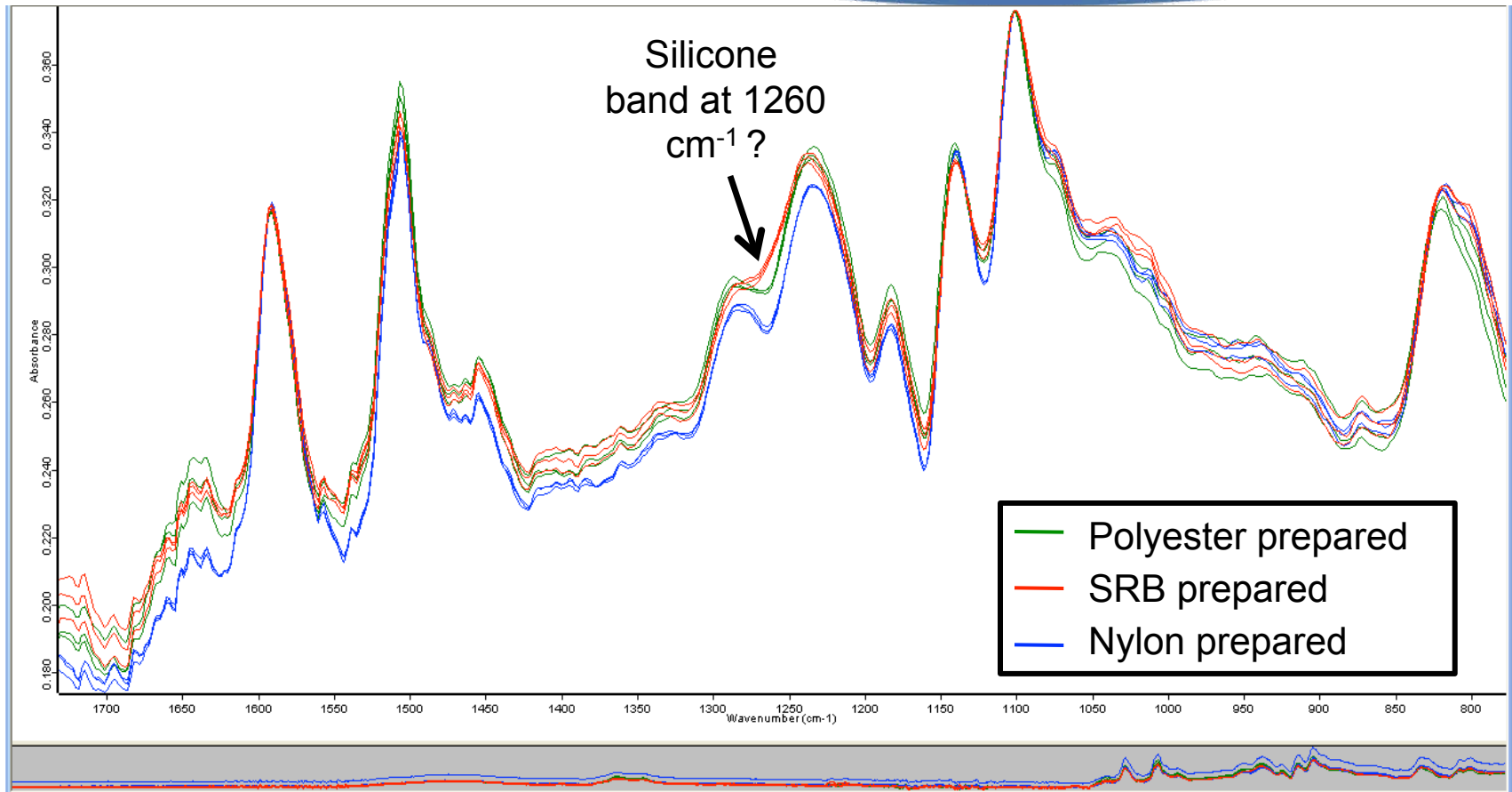
✓ Small differences between CFRP peel ply prep observed

➢ Multivariate analysis?

- ## Multivariate Analysis: PCA of two PCs
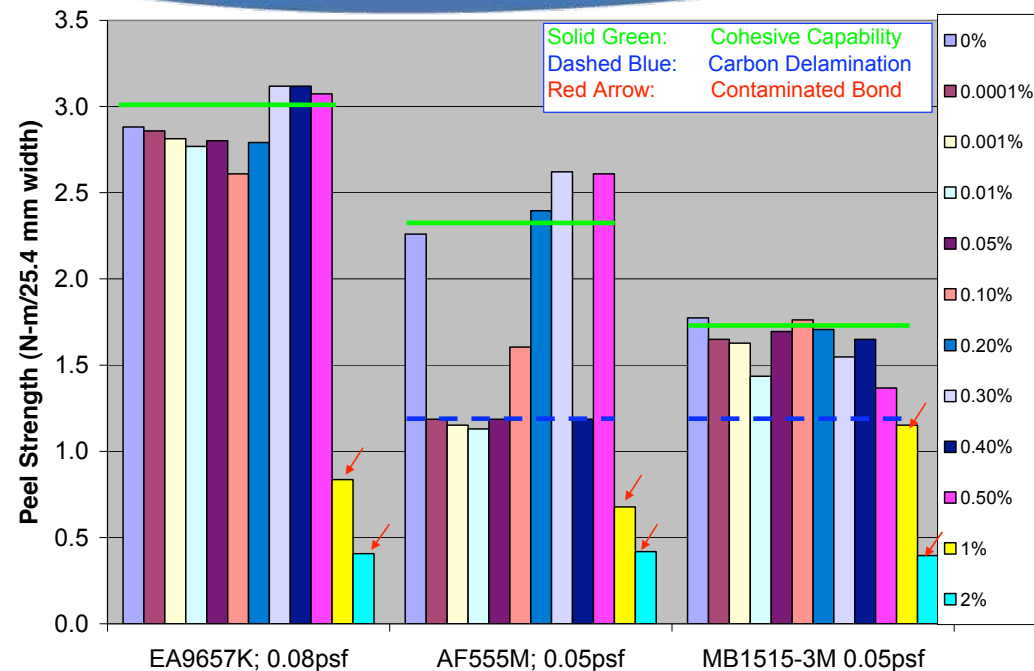 - Preprocessing: Savitsky-Golay 1st derivative with 5 smoothing points fit to a 2nd order polynomial

1=Nylon prep
2=SRB prep
3=Polyester prep

- PCA identifies no significant difference between CFRP peel ply prep
  - ➢ Peel ply prep detectable with diffuse reflectance?

- ## Multivariate Analysis: PCA of two PCs
  - Preprocessing: Savitsky-Golay 1st derivative with 5 smoothing points fit to a 2nd order polynomial

Scores

1=Nylon prep
2=SRB prep
3=Polyester prep
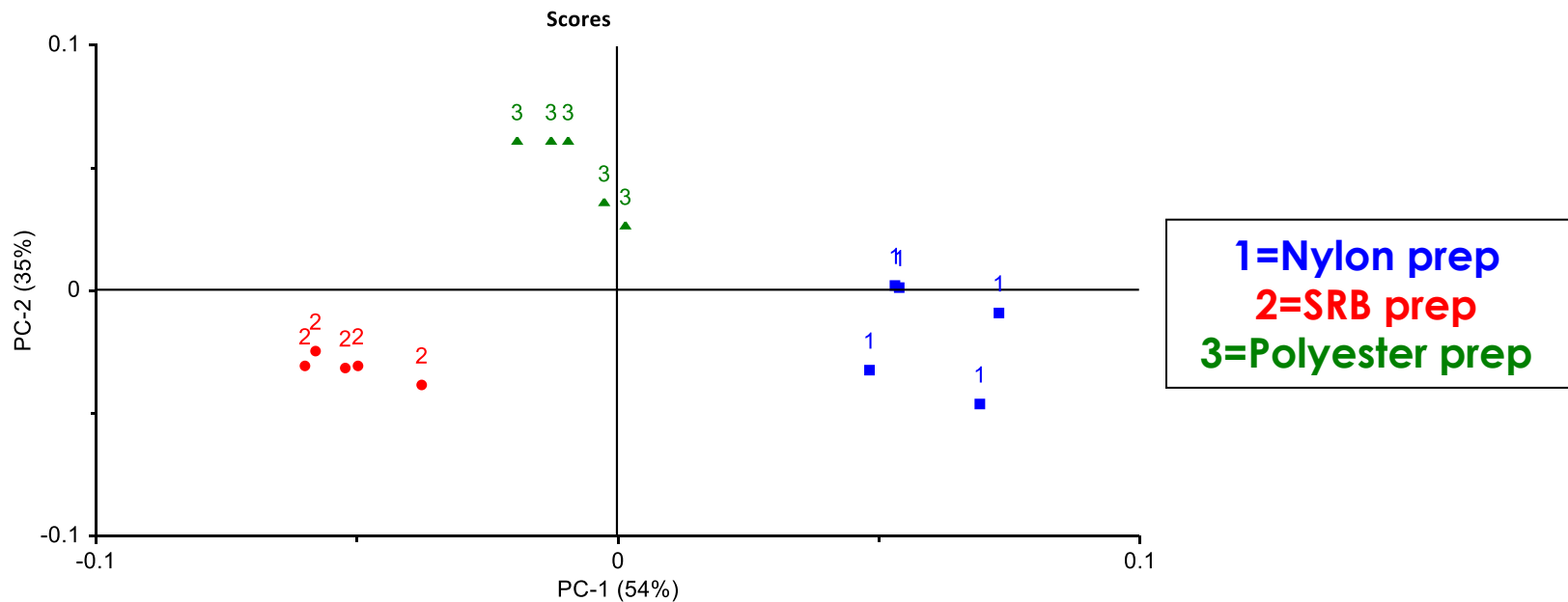
✓ PCA identifies CFRP peel ply surface prep

➢ What level of peel ply contamination is detectable?

Si Contamination of Peel Ply

- Surface contamination detrimental to bonding

  - contamination cannot be completely eliminated from all composite fabrication environments – siloxane

  ➢ Need to inspect CFRP surfaces for contaminates

Chart legend:
- Solid Green: Cohesive Capability
- Dashed Blue: Carbon Delamination
- Red Arrow: Contaminated Bond

Series: 0%, 0.0001%, 0.001%, 0.01%, 0.05%, 0.10%, 0.20%, 0.30%, 0.40%, 0.50%, 1%, 2%

Y-axis: Peel Strength (N-m/25.4 mm width)

X-axis: EA9657K; 0.08psf, AF555M; 0.05psf, MB1515-3M 0.05psf

B-RAT results showing bond quality is degraded of CFRP substrates with peel ply contamination levels at 1% siloxane and greater[1]

[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.

Si contamination on CFRP difficult to quantify with Diffuse Reflectance FTIR
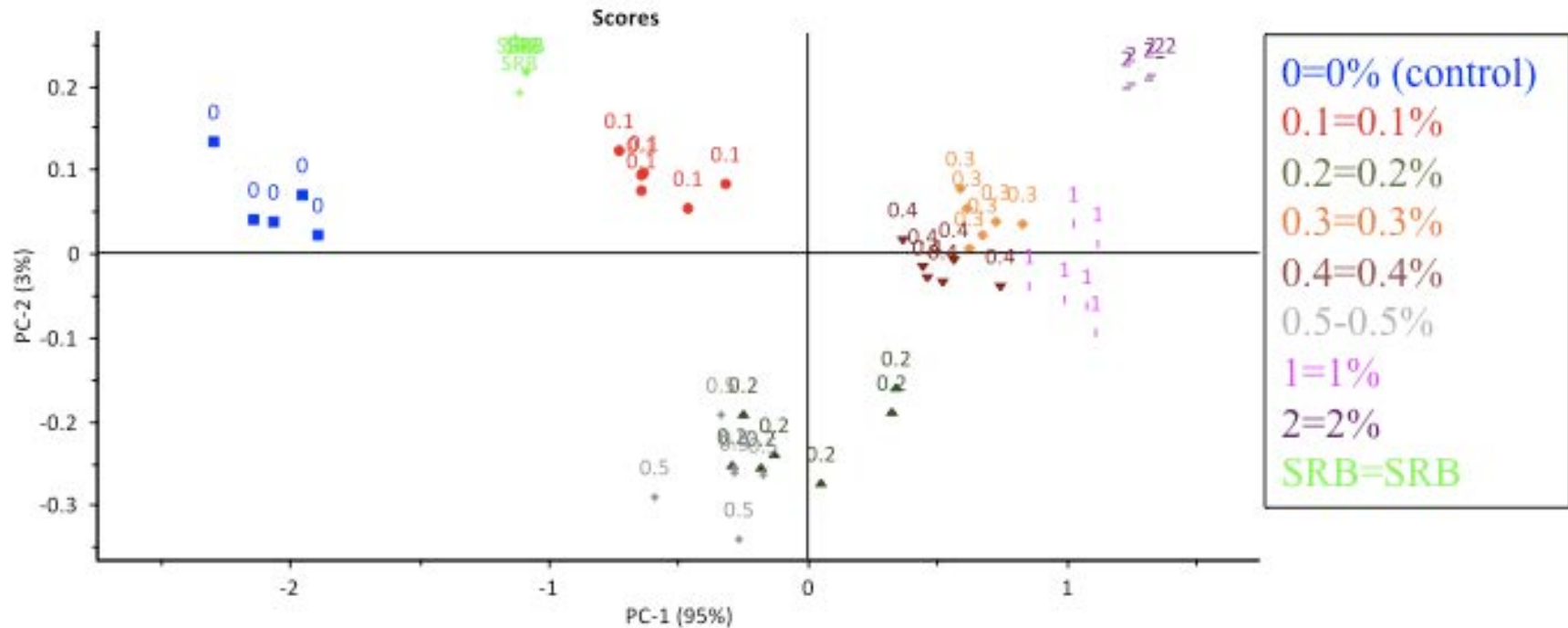
- PCA identifies some differences between siloxane contaminated samples
  - PCA does not show difference between acceptable and unacceptable levels of contamination
   - ➢ Different technique to identify level of contamination?

Hexane Extraction Results using FTIR with Vertical Integrating Sphere (sensitive to small amounts of material): 2" x 2" samples to quantify how much siloxane transfers to surface
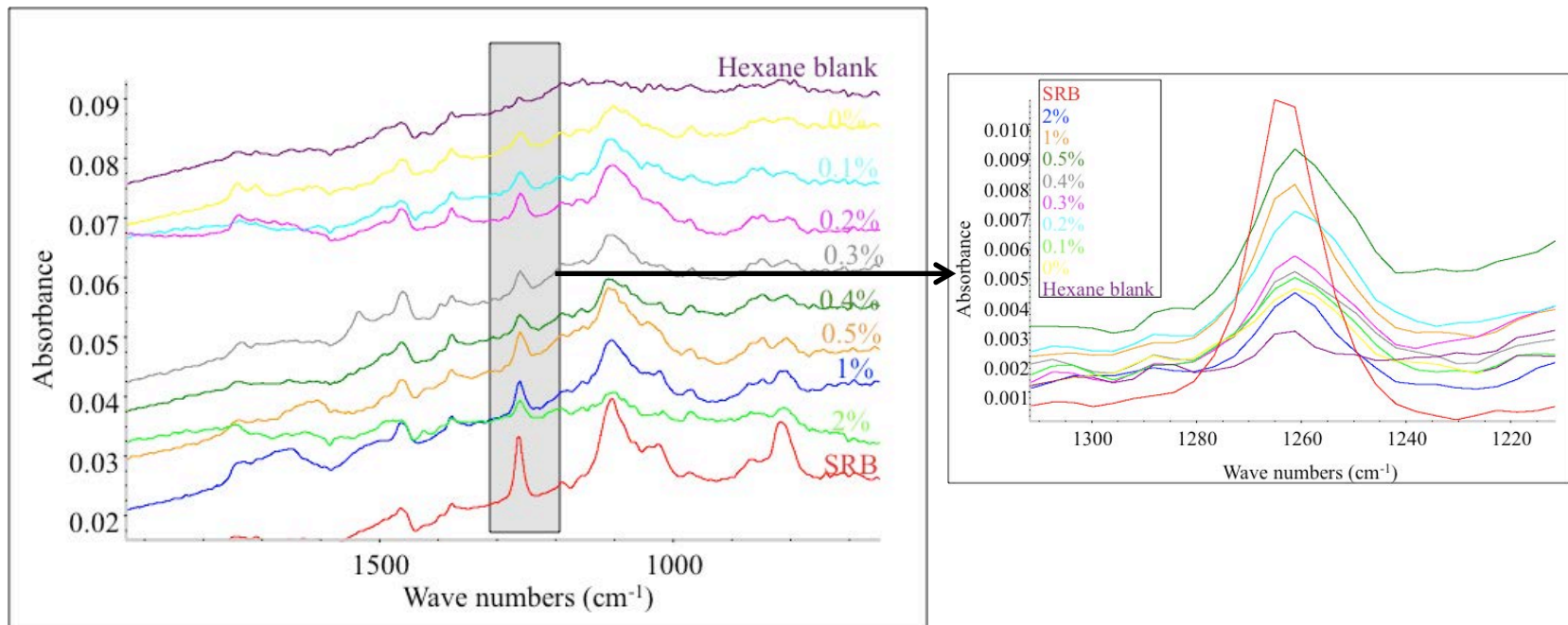
- All spectra show characteristic silicone peaks at 1260 cm^{-1}, 1100 cm^{-1}, 1022 cm^{-1} and 800 cm^{-1}
- Peak at 1260 cm^{-1} used for analysis of amount of siloxane transferred to surface
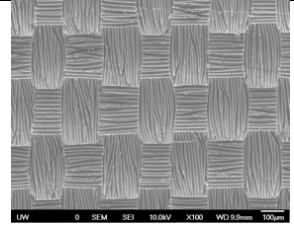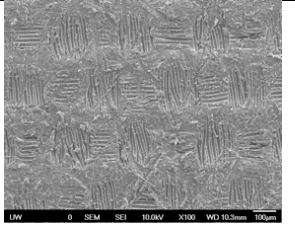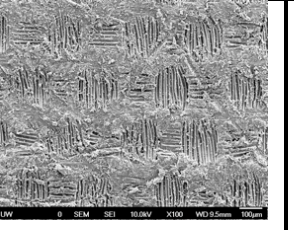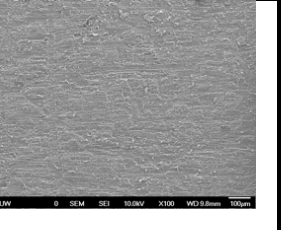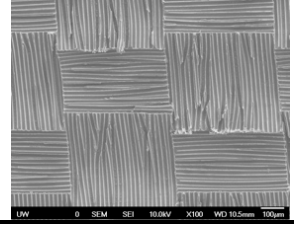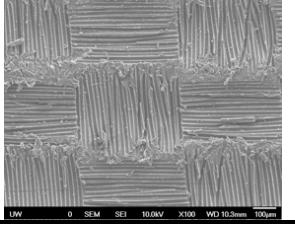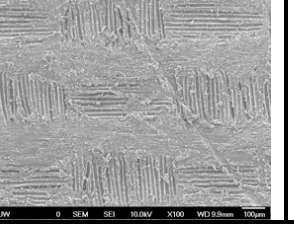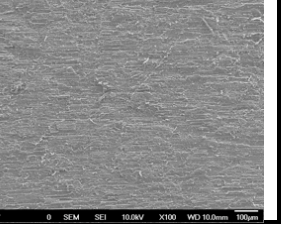
- CFRP surface contamination increases with increased peel ply contamination – maybe use a wipe and analyze residue to determine contamination level?
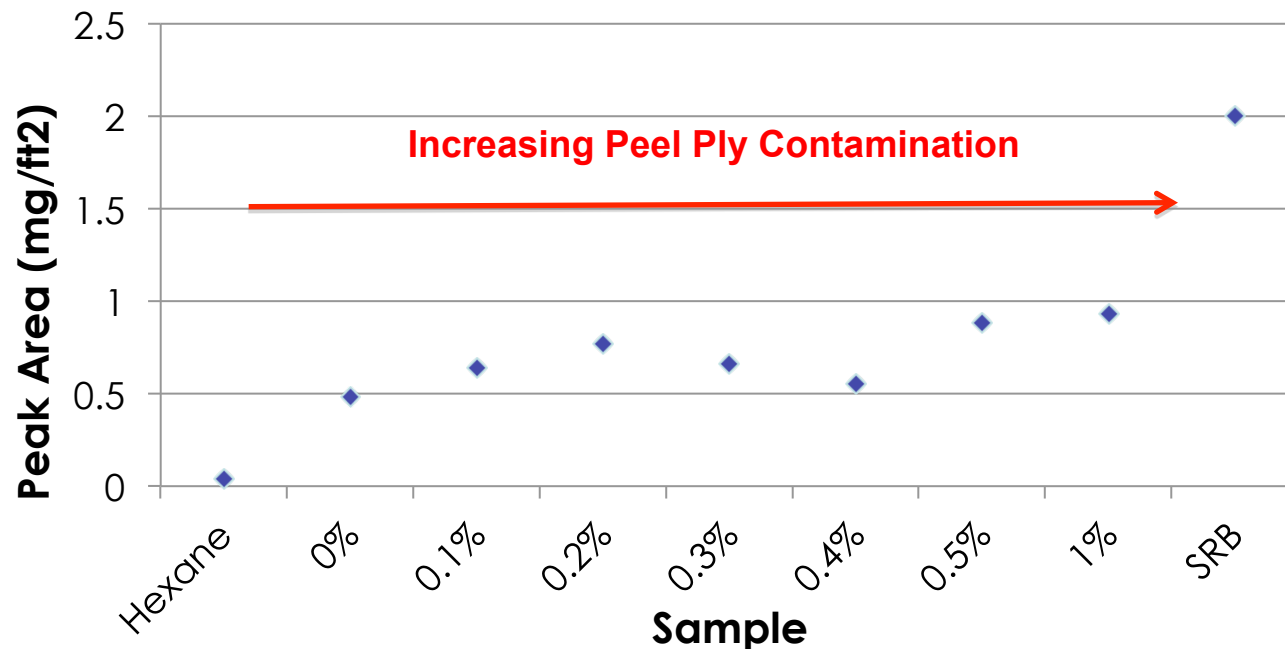
- Diffuse reflectance or DATR to analyze residue?
- ➢ Are different levels of abrasion detectable with FTIR?

Manual Abrasion to Remove Peel Ply Texture

- Previous research shows polyester peel ply prep of 177°C cure composites fail in adhesion when bonded with paste adhesives[2]
  - Surface abrasion can be used to remove peel ply texture/residue from composite → cohesive failure when bonded with paste adhesives[2]
 - Want to detect levels of abrasion to remove peel ply surface layer

	Peel Ply Texture, No Abrasion	> 75% Peel Ply Texture Evident	< 50% Peel Ply Texture Evident	No Peel Ply Texture
Nylon Prepared				
Polyester Prepared				

[2]Bossi, R., R. Carlsen, F.J. Boerio and G. Dillingham. "Composite Surface Preparation QA for Bonding." SAMPE 2005. Long Beach, CA, May 1-5, 2005.

CECAM
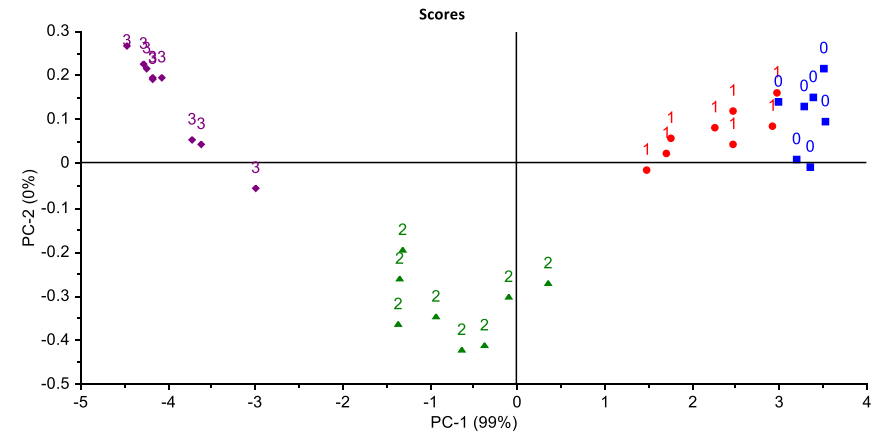
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Advanced Materials in Transport Aircraft Structures

0=polyester peel ply surface
1= >75% peel ply surface evident
2= <50% peel ply surface evident
3=no peel ply surface evident

Unacceptable abrasion

Acceptable abrasion
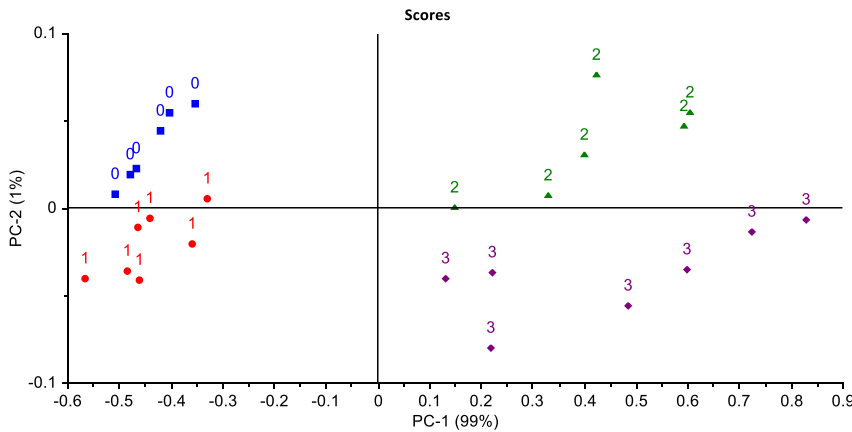
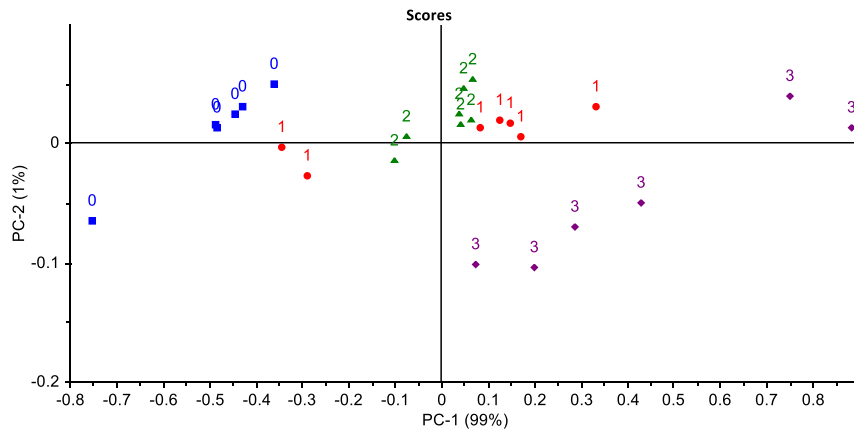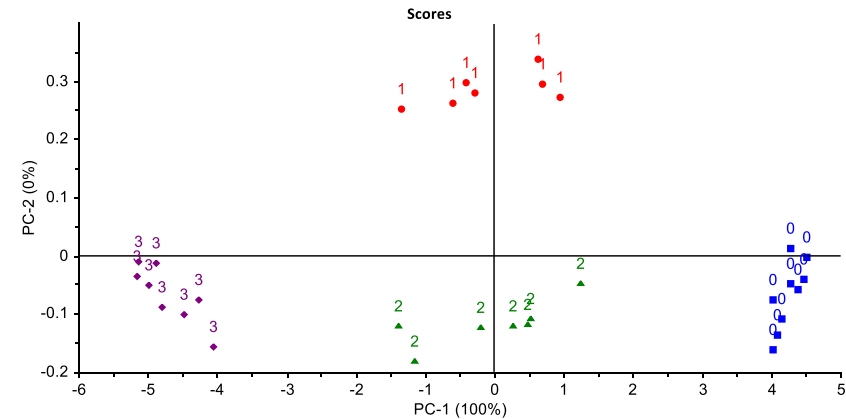**PCA of DATR Spectra**

PCA of Diffuse Reflectance Spectra

✓PCA of diffuse reflectance spectra shows difference between acceptable and unacceptable abrasion levels to remove polyester peel ply texture/residue → more sensitive than DATR

0=nylon peel ply surface
1= >75% peel ply surface evident
2= <50% peel ply surface evident } Unacceptable abrasion
3=no peel ply surface evident → Acceptable abrasion


PCA of DATR Spectra

PCA of Diffuse Reflectance Spectra

✓PCA of diffuse reflectance spectra shows difference between acceptable and unacceptable abrasion levels to remove nylon peel ply texture/residue → more sensitive than DATR

Summary of FTIR Results

	FTIR Sampling Interface	
	DATR	**Diffuse Reflectance**
Peel Ply Prep	?	✔
Si Contaminants	N/A	?
Abraded Surfaces to Remove Peel Ply Residue	✔-	✔
Scarfed Surfaces/Repair	TBD	TBD

- Diffuse reflectance shows greater sensitivity to surface preparation than DATR
  - Due to sampling volume?
    - DATR spectra from surface in contact with DATR crystal – ideal for smooth surfaces, less contact with rough surfaces
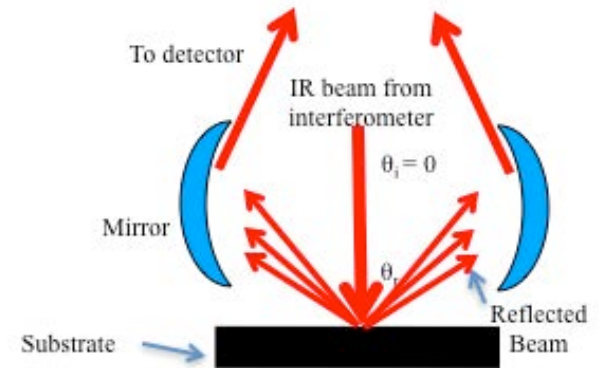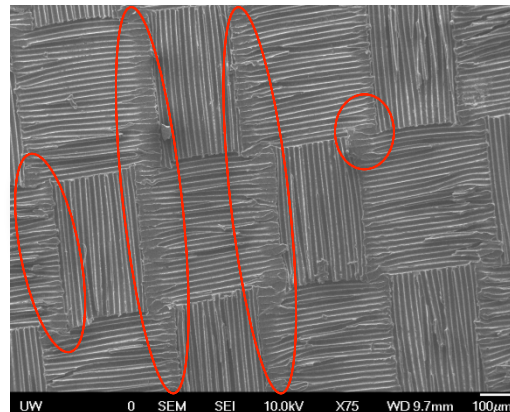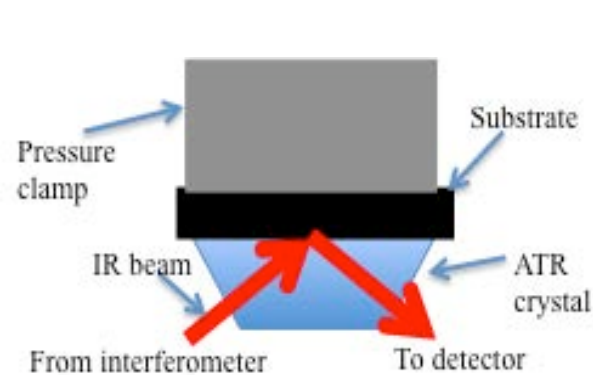
    - Diffuse reflectance spectra better for rough surfaces – can get information from peel ply channels

Conclusions

Diffuse reflectance more sensitive to rough surfaces than DATR

- Peel Ply Prep:
  - Diffuse reflectance can identify polyester vs. nylon vs. SRB prep
- Peel Ply Contamination:
  - Diffuse reflectance can identify some differences between siloxane contaminated samples
  - Hexane wipe alternative? Diffuse to analyze wipe?
- Abrasion to Remove Peel Ply Texture:
  - Diffuse reflectance can identify removal of nylon and polyester peel ply texture

FTIR has potential as a quality assurance technique for adhesive bonding process

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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
  - Surface characterization vs. bond quality model
    - Bond quality vs. amount of peel ply texture on abraded surface
  - QA methods 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 COE: JAMS, AMTAS

- The Boeing Company

  – Kay Blohowiak, Pete Van Voast, Will Grace, Marc Piehl, Paul Shelley, Paul Vahey, Greg Werner

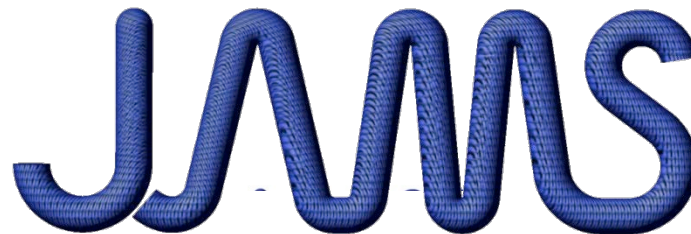- Precision Fabric Group

- Richmond Aircraft Products

- Airtech International

End of Presentation.

Thank you.