

# Composite Thermal Damage Measurement with Handheld FT-IR

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and Brian Flinn<sup>1</sup>

1. Materials Science and Engineering, University  
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2. The Boeing Company, Seattle, WA



# FAA Sponsored Project Information

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## Principal Investigators & Researchers

- Brian Flinn (PI)
- Tucker Howie (Post Doc, UW-MSE)
- Ashley Tracey (PhD student, UW-MSE)

## FAA Technical Monitor

- David Westland (year 4)
- David Galella (year 3)
- Paul Swindell (year 1 & 2)

## Industry Participation

- The Boeing Company (Paul Shelley, Paul Vahey)
- Sandia National Lab (Dennis Roach)
- Agilent (formerly A2 Technologies)

## Motivation and Key Issues

- Damage detection in composites requires different techniques than metals
- Incipient thermal damage (ITD) occurs below traditional non-destructive evaluation (NDE) detection limits
  - ITD is chemical damage. NDE detects physical damage such as delaminations and microcracking

## Objective

- Determine if handheld Fourier transform infrared (FTIR) spectroscopy can detect ITD and guide repair

## Approach

- Characterize panels with controlled thermal damage and perform repair based on FTIR inspection

# Project Background

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Continuation of existing project (year 4 of 4)

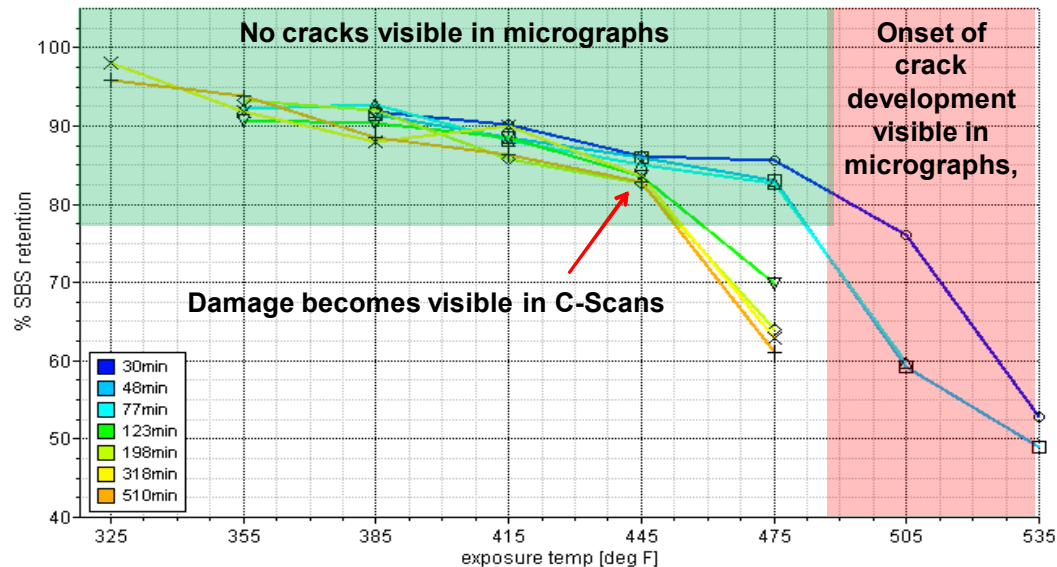
Years 1 and 2 (A2 Technologies, Boeing and U of DE)

- Characterization of homogeneous thermal damage
  - Ultrasound
  - Short beam strength (SBS)
  - Microscopy
  - Handheld Fourier transform infrared (FTIR) spectroscopy (ExoScan)
- Calibration curve for FTIR detection of thermal damage (SBS data)
- Mapped surface of localized thermal damage on resin rich surface

Year 3 & 4 (UW and Boeing)

- Contact angle and fluorescence spectroscopy
- 3-D characterization of localized thermal damage
- FTIR guided scarf repair
- Mechanical testing of locally damaged and repaired panels

# Thermal Damage vs. Detection Method



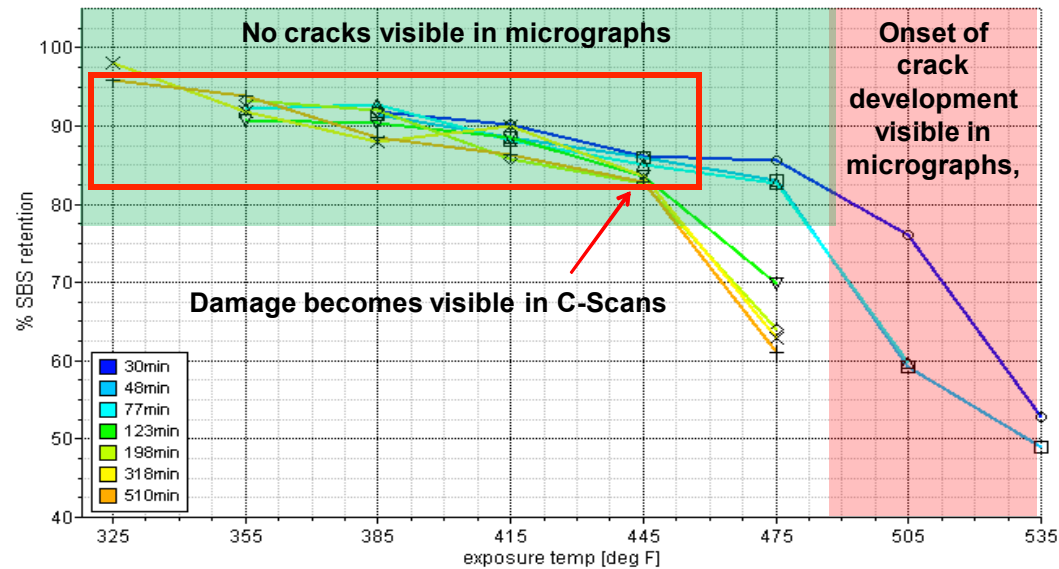
Short beam strength (SBS) degrades before detection possible with ultrasound or visual inspection

- Damage termed ITD

Need a method to detect ITD

- FTIR?

# Thermal Damage vs. Detection Method



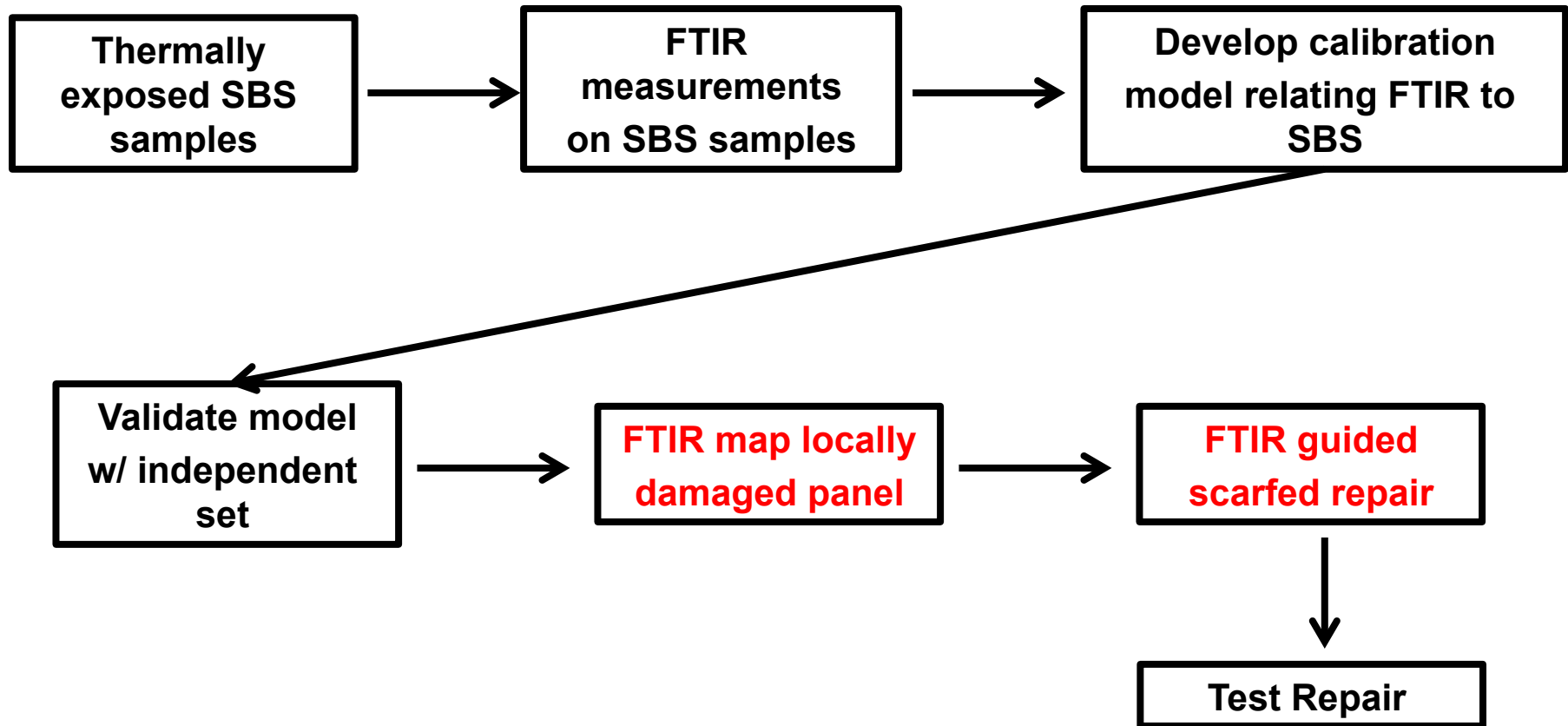
Short beam strength (SBS) degrades before detection possible with ultrasound or visual inspection

- Damage termed ITD

Need a method to detect ITD

- FTIR?

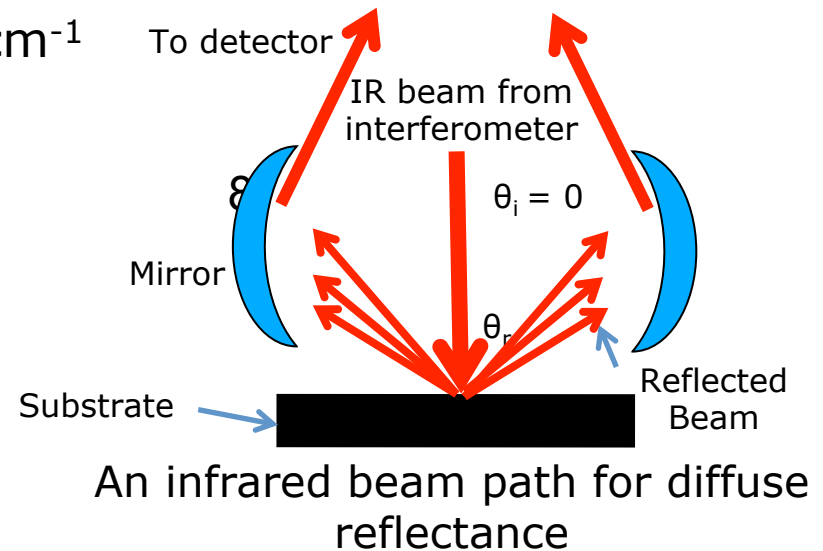
# Experimental Overview



- Toray T800/3900 composites with various levels of thermal damage
  - SBS calibration samples thermally exposed in convection oven
  - Panels with localized damage from heat blanket and insulation
- Sand surfaces with 180 grit  $\text{Al}_2\text{O}_3$  sanding pads using pneumatic orbital sander to simulate repair
- FTIR spectra taken with Exoscan FTIR using diffuse reflectance
  - Mid-IR range:  $4000\text{ cm}^{-1}$  to  $650\text{ cm}^{-1}$
  - 90 scans,  $16\text{ cm}^{-1}$  resolution

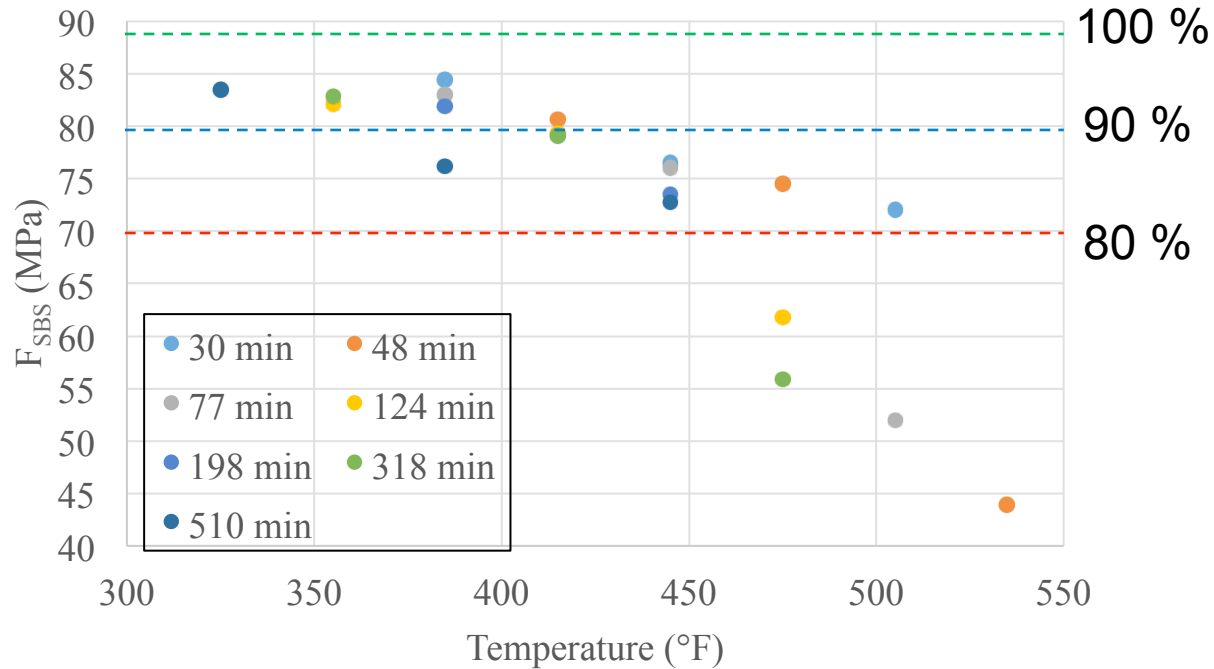


ExoScan FTIR





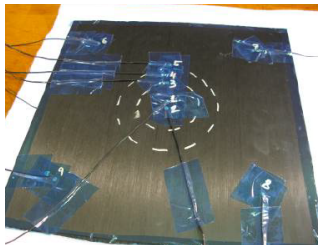
# SBS Calibration Samples



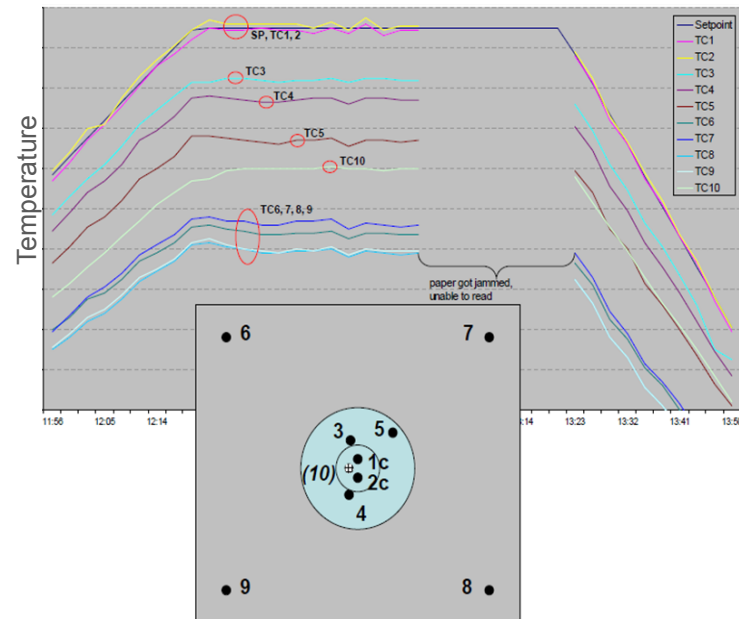
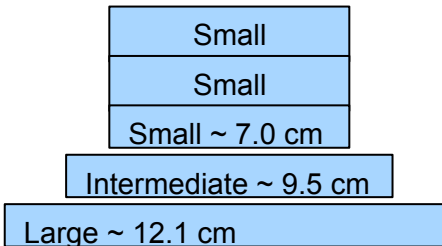
- Range of thermal exposure chosen using Design of Experiments (DOE) in ITD region
- $F_{SBS}$  values ranged from 43.9 MPa to 84.4 MPa (undamaged  $F_{SBS} = 88.8$  MPa)

# Locally Heated Panel Setup

- 24-ply unidirectional 12 in x 12 in panels subjected to localized hotspot
- Local hotspot from heat blanket + extra insulation layers in center of panel
- Panels exposed for 1 hour at one of three peak temperatures (440 °F, 465 °F, 490 °F)
  - Panels referred to as low, medium, and high exposure respectively

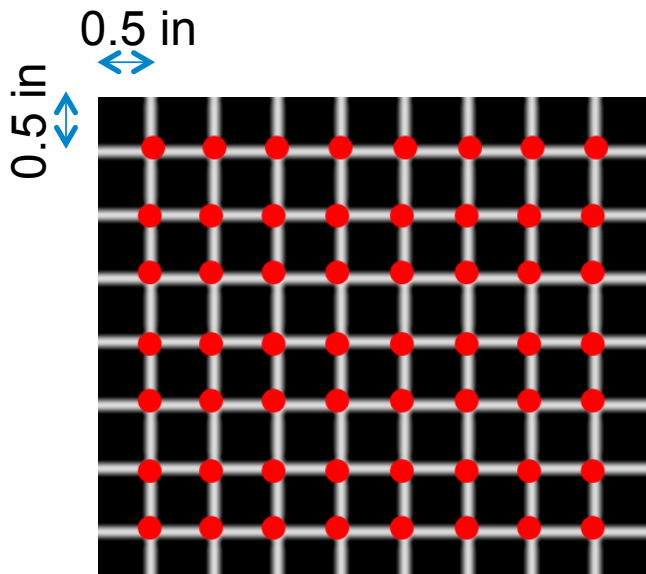


## Insulation Stacking

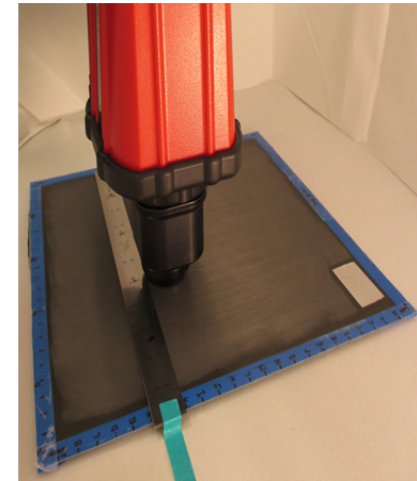
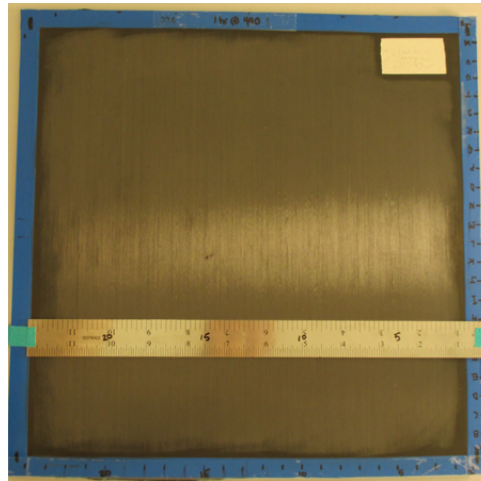


# Panel Mapping Procedures

- Grid with 0.5 in between points marked on edges of panel
- FTIR positioned using rulers to align with grid
- Measurements taken at every point on grid
  - 3 measurements taken at every point

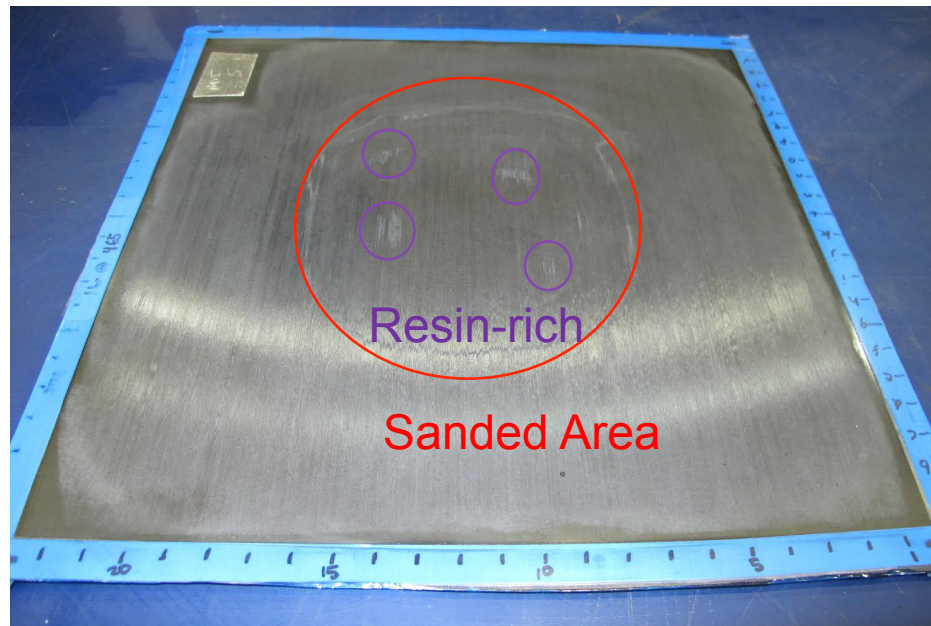


- Measurement location

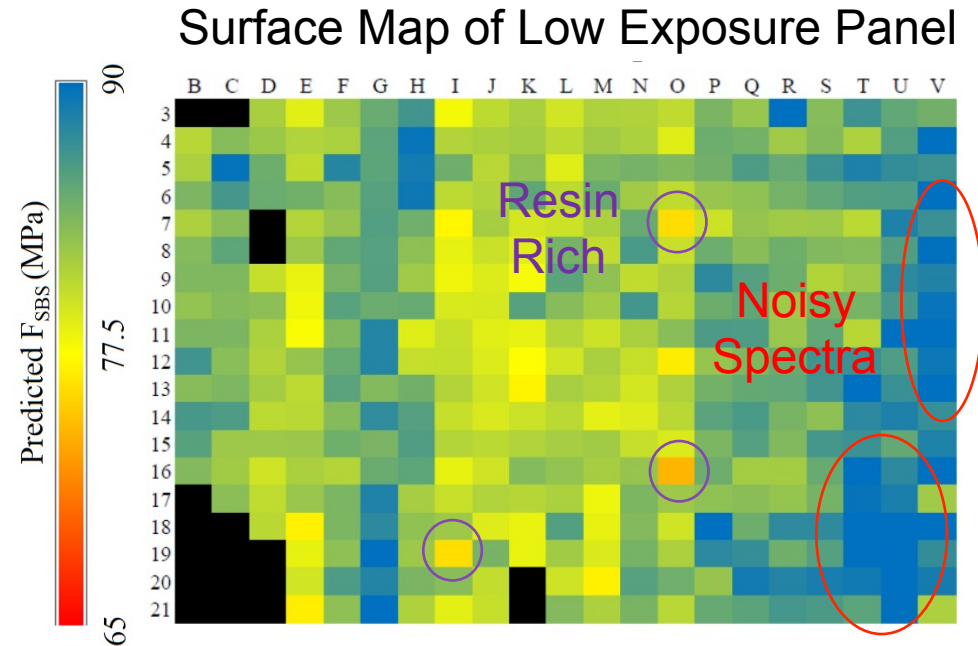
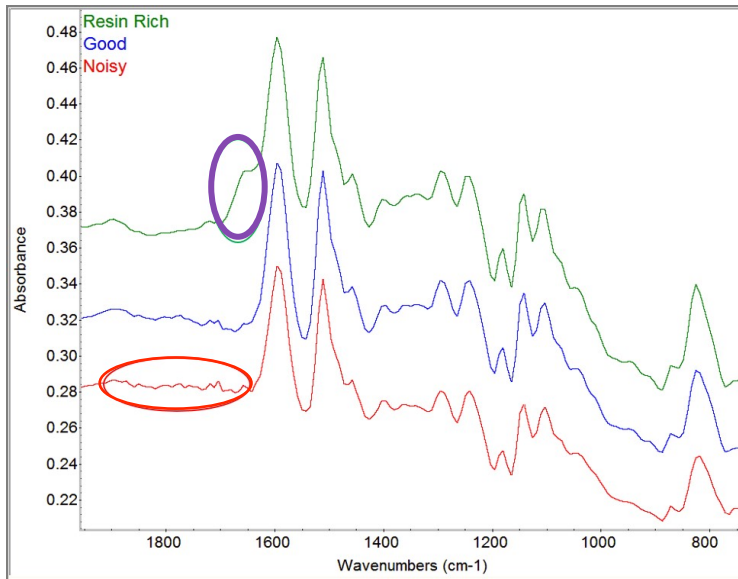


# Panel Sanding

- Located damage based on FTIR inspection and sanded down to next ply
  - Small areas of over-sanding leading to resin rich spots



# Effect of Spectral Features on Model Predictions

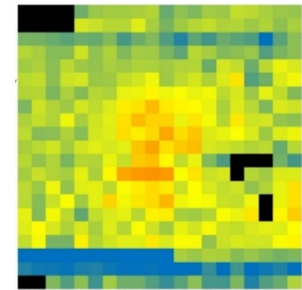
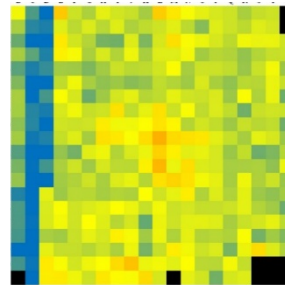
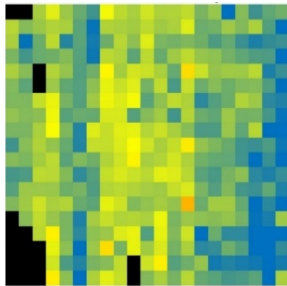


- Resin rich spectra exhibit broad carbonyl peak between 1600-1690  $\text{cm}^{-1}$ 
  - Predicted lower  $F_{\text{SBS}}$  values
- Noisy spectra were observed by an increase in noise in the baseline
  - Predicted higher  $F_{\text{SBS}}$  values
  - Can be mitigated by taking a new background reference

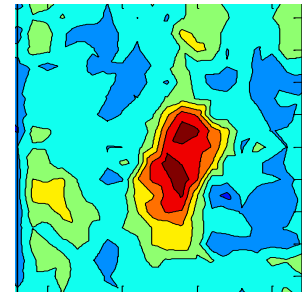
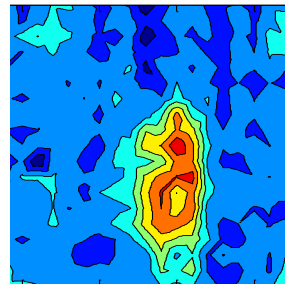
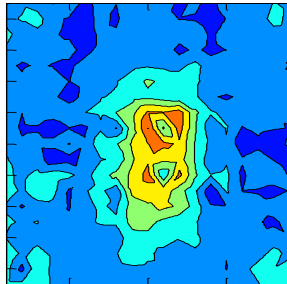
# Comparison of Surface Mappings Predictions

Low Exposure    Med. Exposure    High Exposure

**Sanded Surface  
 (Model 4)**



**Resin Rich  
 Surface  
 (1<sup>st</sup> year)\***

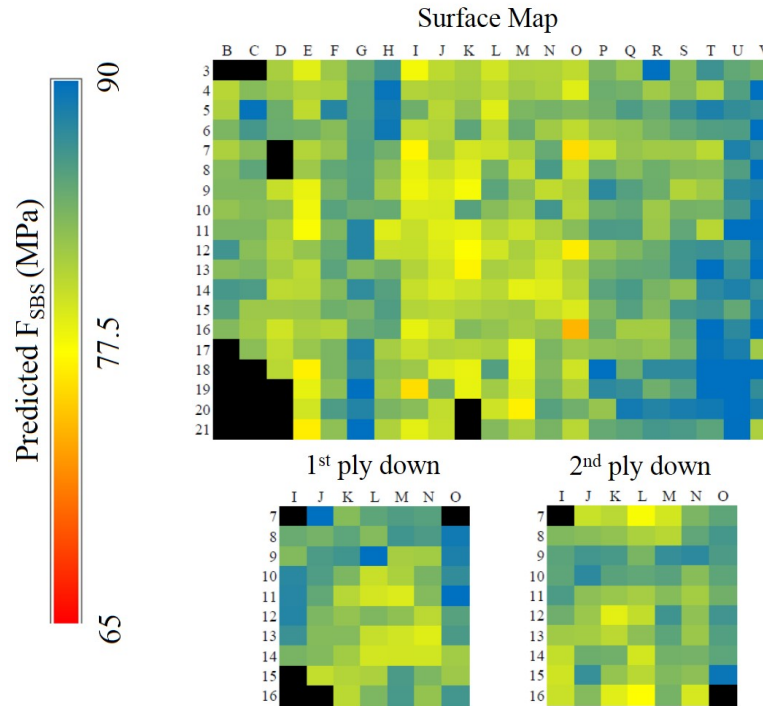


Note: Scaling, panel orientation, and color scheme are different

- Reasonable agreement between predictions results on the two surfaces



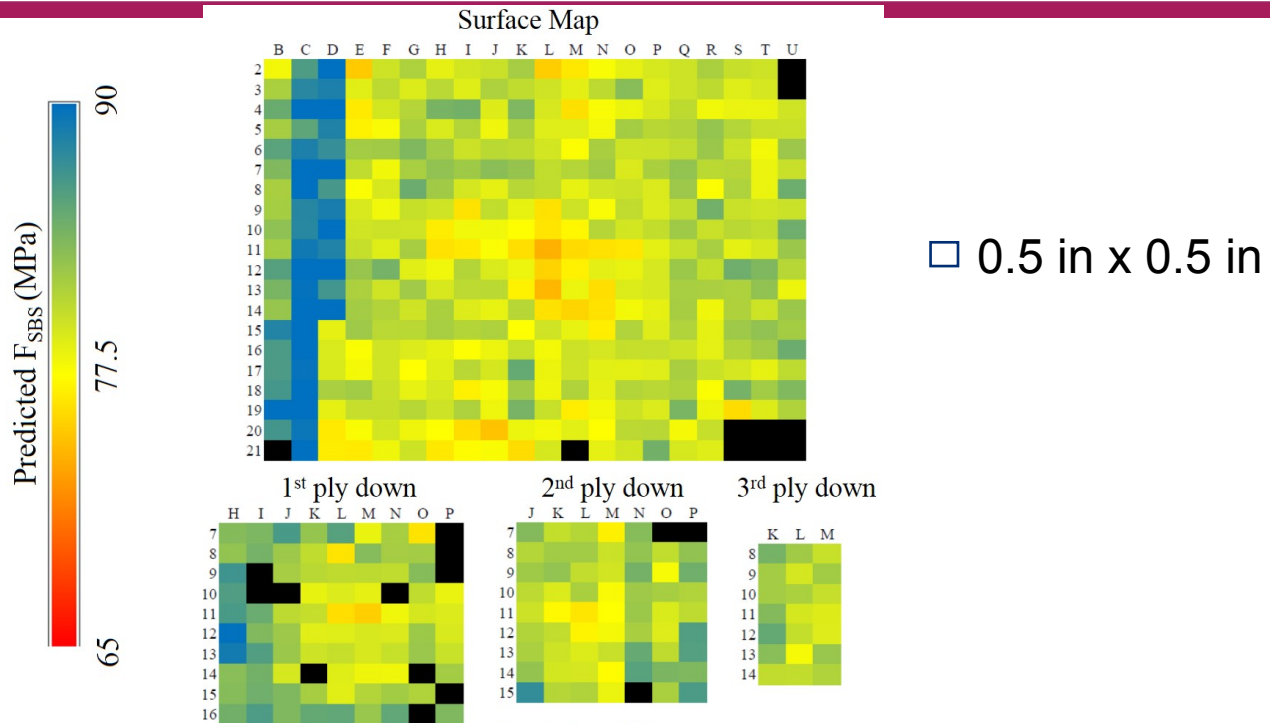
# Inspection of Low Exposure Panel



□ 0.5 in x 0.5 in

- Low exposure panel used to establish a Go/No Go threshold to damage removal
- Sanded down 2 plies to evaluate prediction variance
  - 79.5 MPa (90% retention of undamaged  $F_{SBS}$ ) chosen as the threshold (Green or blue color on the map)

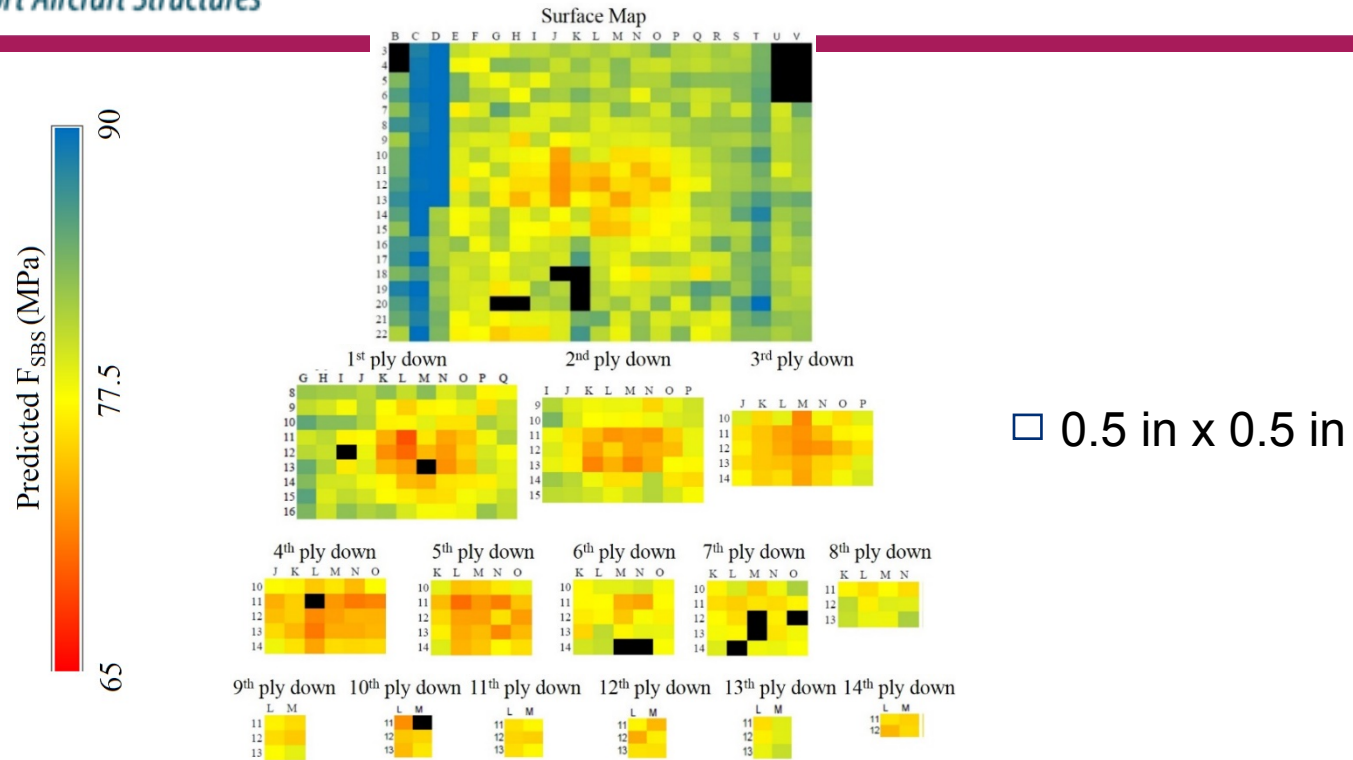
# Inspection of Medium Exposure Panel



- Medium exposure panel exhibited moderate damage in 3" x 3"
  - Low  $F_{SBS}$  values around 74-75 MPa ( $\sim 82\%$  of undamaged  $F_{SBS}$ )
- Most of the damage was removed after sanding to the first ply down
- Panel passed Go/No Go threshold after sanding 3 plies down



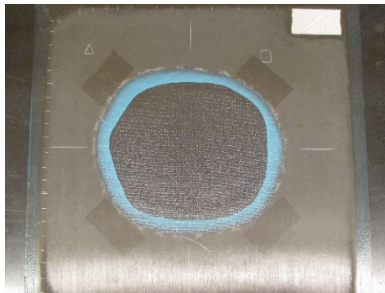
# Inspection of High Exposure Panel



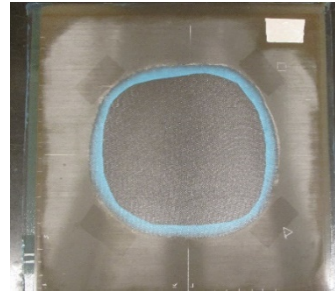
- High exposure panel had large damage region  $\sim 5'' \times 5''$ 
  - Low  $F_{SBS}$  values around 72-73 MPa ( $\sim 80\%$  of undamaged  $F_{SBS}$ )
- Go/No Go threshold not passed in the center of the panel after sanding down 14 plies
- Inspection stopped due to repair size restriction

# Repair Process

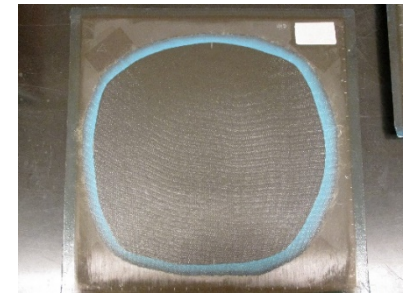
- After inspection was completed panels were sanded to a 30:1 scarf angle
- Patch plies were cut from Toray T800/3900 unidirectional prepreg
  - The high exposure patch was double vacuum debulked (DVD) to help removal volatiles that could be trapped in a thick patch
- MetlBond 1515-3M adhesive
- Patches were cured under a vacuum using a heat blanket controlled by a hot bonder at 350 °F for 2.5 hours



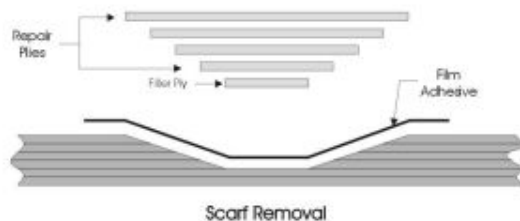
Low Exposure (2-ply)



Medium Exposure (3-ply)

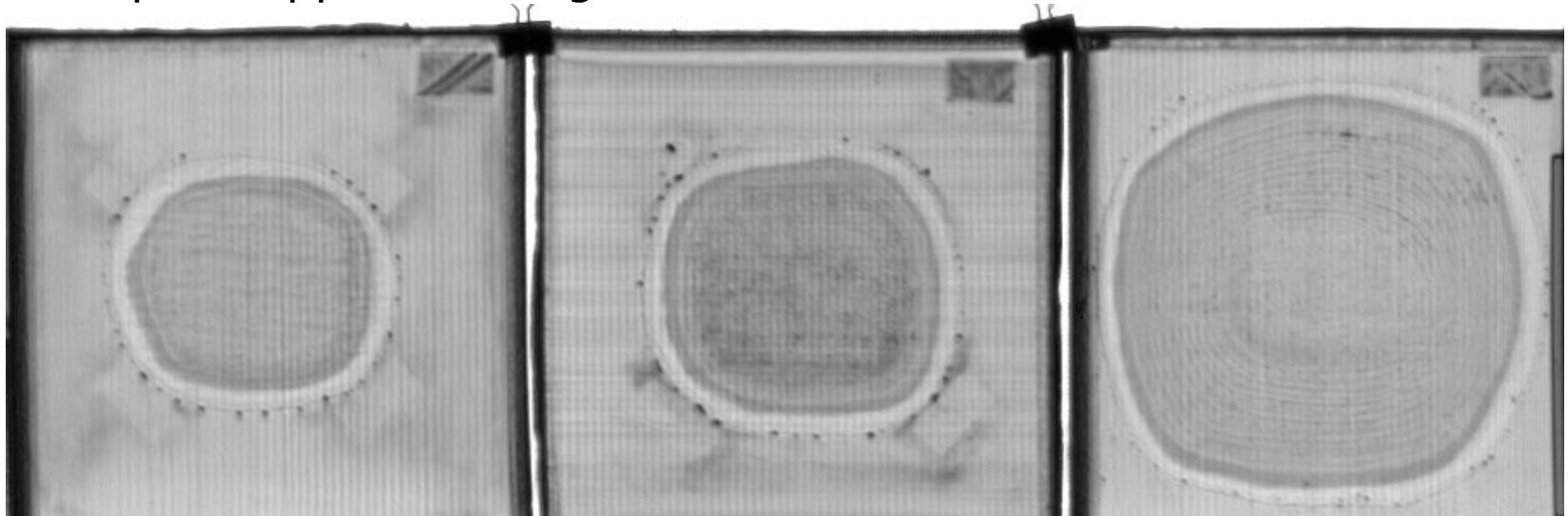


High Exposure (14 plies)



# Inspection of Repairs

- Repaired panels inspected using ultrasound
  - 5Mhz 2.5" Focused TTU
  - 6" Water Path
  - .04 Resolution
- Repairs appear to be good



Low Exposure (2-ply)

Medium Exposure (4-ply)

High Exposure (14 plies)

- Mechanical testing of repaired and duplicate damage panels to evaluate removal of ITD
  - Short beam strength
    - Gives interlaminar shear stress and is known to be sensitive to incipient thermal damage
    - Max shear stress occurs at center of the sample, but most damage is on the plies near surface
  - Tension test with 45° samples
    - Sensitive to matrix dominated properties
    - May not be sensitive to damage in ITD range
  - Compression after impact
    - Sensitive to ITD
    - Potentially sensitive to surface damage
    - Number of test samples

# Ongoing Work

- Mechanical testing of repaired and duplicate damage panels to evaluate removal of ITD
  - Short beam strength
    - Gives interlaminar shear stress and is known to be sensitive to incipient thermal damage
    - Max shear stress occurs at center of the sample, but most damage is on the plies near surface
  - Tension test with 45° samples
    - Sensitive to matrix dominated properties
    - May not be sensitive to damage in ITD range
  - Compression after impact
    - Sensitive to ITD
    - Potentially sensitive to surface damage
    - Number of test samples

# Ongoing Work

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- Making three additional panels to determine effectiveness of testing method prior to testing panels
  - Undamaged Panel
  - Undamaged Panel with large repair
  - Extra damage panel exposed at  $\sim 480$  °F for 1 hr
- If tensile test can detect ITD the remaining six panels (3 repaired and 3 damaged) panels will be tested
- This work should be completed by December 31, 2014

- Improved consistency of model predictions
- Go/No Go Threshold set at prediction of 90 %  $F_{SBS}$  retention
- Used FTIR to map and guide repair of thermally damaged panels
  - Low exposure panel exhibited almost no damage
  - Medium exposure panel had moderate damage which was removed after sanding down 3 plies
  - Part of high exposure panel did not reach Go/No Go threshold after removing 14 plies
    - Stopped to repair size restrictions
- Panels repaired using scarf repair process
- Repaired panels inspected with ultrasound and repaired panels look good
- Mechanical testing of panels is currently ongoing

FAA, JAMS, AMTAS 

Boeing Company 

- John Spalding, Mary Vargas, Mark Bradley

Sandia National Labs 

Agilent Technologies  Agilent Technologies

- John Seelenbinder

UW MSE 

- Edward Roberts, Jake Plummer, David Pate,  
Jonathan Morasch

University of Delaware 



# Looking Forward

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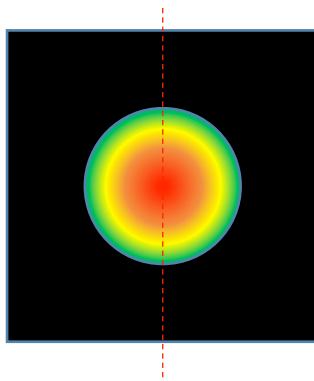
- Benefit to Aviation
  - Improved damage detection
  - Greater confidence in repairs
- Future needs
  - Application to other composite systems
  - Other applications of handheld FTIR
    - Chemical damage
    - Surface prep for bonding

QUESTIONS?

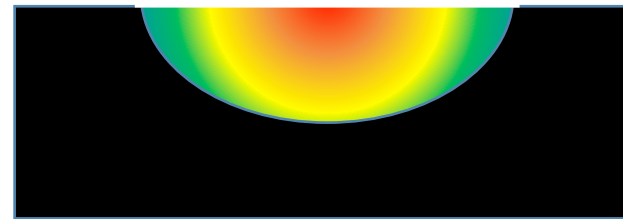
# Considerations for Mechanical Testing

- Properties desired for mechanical testing
  - Need to test both repaired and damaged panels to compare results
  - Tests matrix dominated properties
  - Preferably large area such that it contains a significant portion of damaged region
  - Prefer failure of composite rather than bondline in repaired panels
- Damage distribution in the panels
  - Highest damage on surface in the center of the panel
  - Damage decreases radially outward from the center on the panel
  - Damage into the depth of panel has ellipsoidal form

Top View of Damage Panel



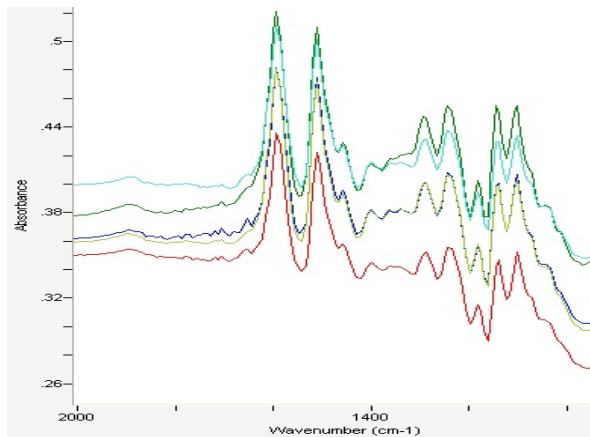
Representative cross-section of damage area  
taken along red dashed line



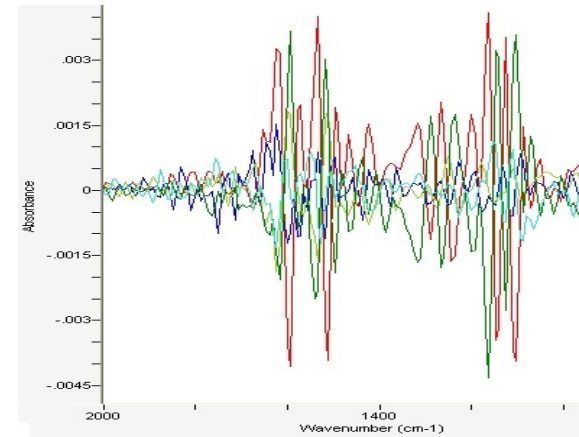
Red = high damage  
Blue/Green = light damage  
Black = no damage

# Generating Calibration Model

- Model needed to correlate FTIR spectra to  $F_{SBS}$  data
- Spectra preprocessed using Savitzky-Golay 1<sup>st</sup> derivative and 7-pt smoothing
  - Removes baseline effects
  - Accentuates differences in spectra
- Partial Least Squares (PLS) model generated using Principal Components Analysis (PCA) in GRAMS IQ software



Raw Spectra



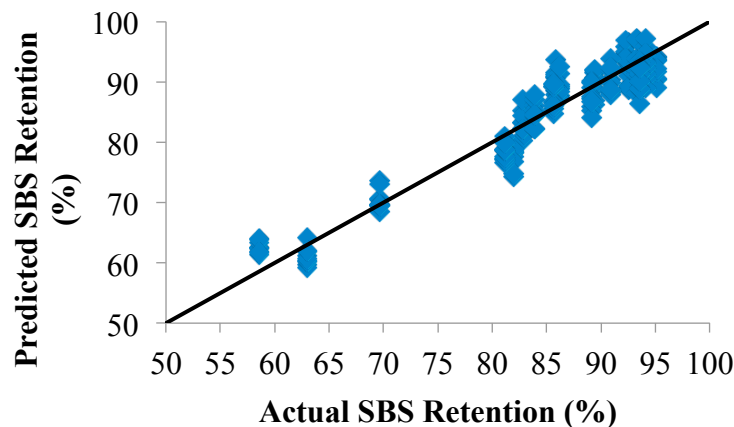
Processed Spectra

Model validated by predicting independent evaluation set

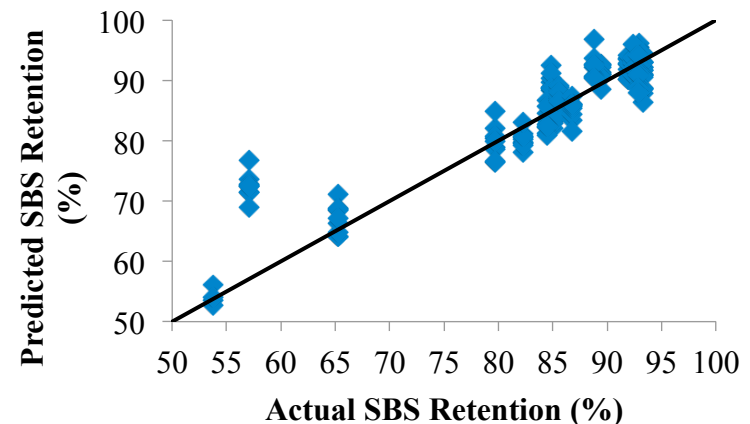
- Model showed good predictive capabilities of evaluation set (~85% of samples had < 5% error)

$$\%Error = \frac{predicted - actual}{actual} * 100\%$$

**Cross-validation of calibration set using leave-one-out method**



**Prediction of independent evaluation set**



- ✓ PLS model relating the SBS measurements to FTIR spectra successfully generated

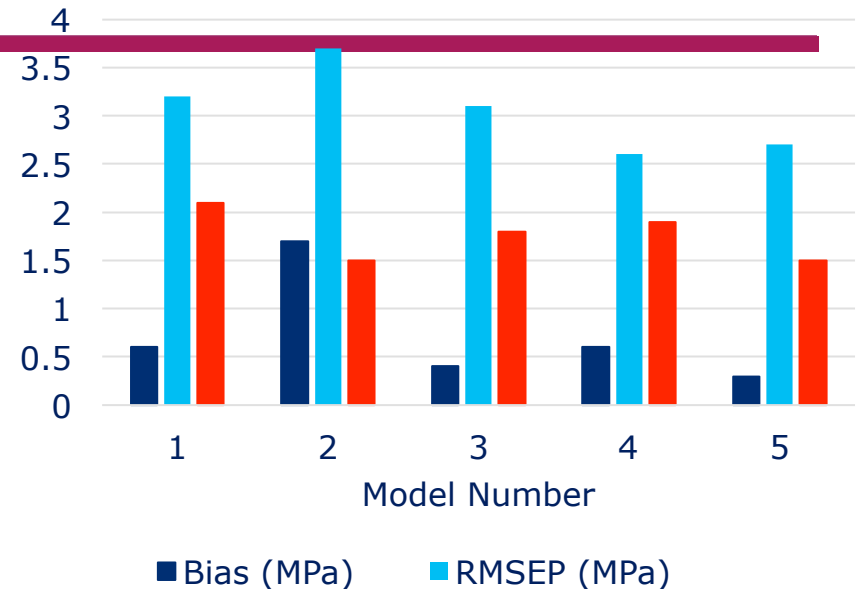
Model	F <sub>SBS</sub> Values in Calibration Set	# of Principal Components	Frequency Range (cm <sup>-1</sup> )
1	All	5	1700-950
2	All	3	1700-950
3	All	4	1700-950
4	F <sub>SBS</sub> > 60.0 MPa	4	1700-950
5	F <sub>SBS</sub> > 60.0 MPa	4	1600-950

Models generated by altering 3 variables

- Sample Set → lower F<sub>SBS</sub> values may skew model (physical damage not chemical)
- Principal components → overfitting vs. underfitting
- Restrict frequency range → reduce influence of certain peaks

# Model Analysis

Model	F <sub>SBS</sub> Values in Calibration Set	# of Principal Components	Frequency Range (cm <sup>-1</sup> )
1	All	5	1700-950
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3	All	4	1700-950
4	F <sub>SBS</sub> > 60.0 MPa	4	1700-950
5	F <sub>SBS</sub> > 60.0 MPa	4	1600-950



Model predictions of independent sample set evaluated on three criteria:

- Bias:  $\sum_{i=1}^n (x_{i,pred} - x_{i,actual}) / n$
- Root mean square error of prediction:  $\sqrt{\sum_{i=1}^n (x_{i,pred} - x_{i,actual})^2 / n}$
- Average Mahalanobis Distance (M-distance): Measure of the spread of the data