

JOINT ADVANCED MATERIALS & STRUCTURES  
CENTER OF EXCELLENCE

# Composite Thermal Damage Measurement With Handheld FTIR

Tucker Howie  
Materials Science and Engineering  
University of Washington

JAMS 2014 Technical Review  
March 25-26, 2014



# FAA Sponsored Project Information

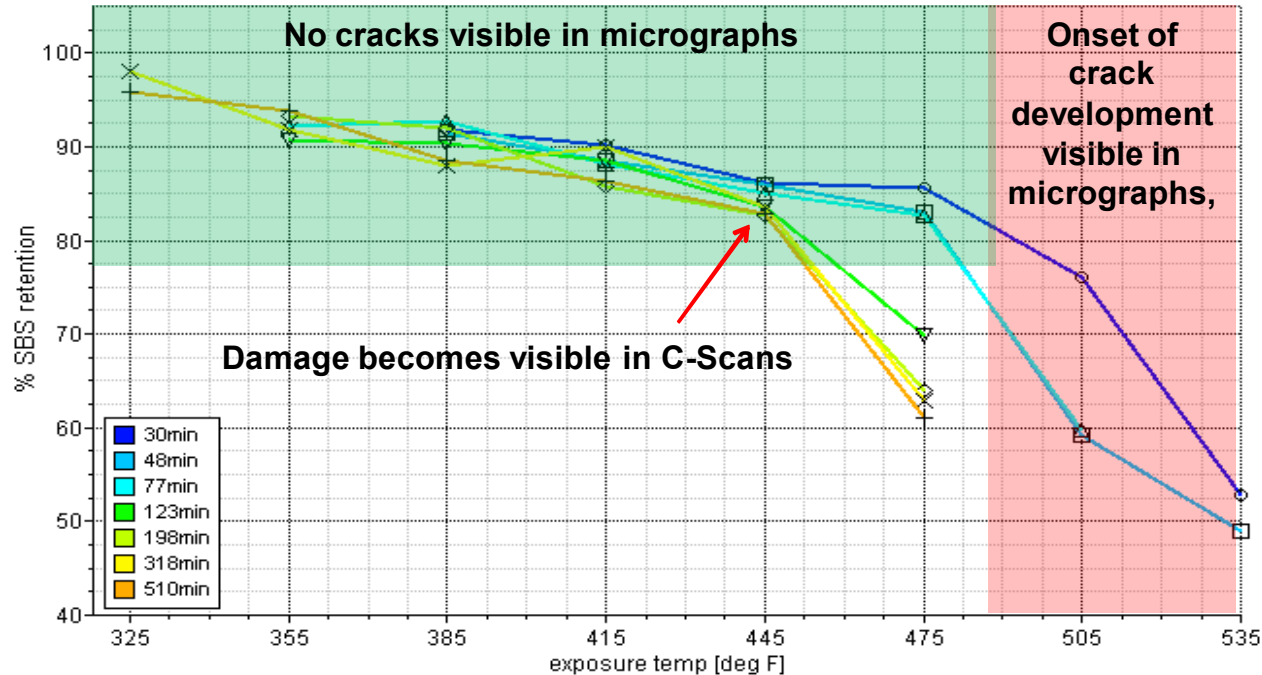
- Principal Investigators & Researchers
  - Brian Flinn (PI)
  - Tucker Howie (Post-doc, UW-MSE)
  - Ashley Tracey (Ph.D. candidate, UW-MSE)
  - Edward Roberts (undergraduate, UW-MSE)
- FAA Technical Monitor
  - David Galella and David Westland (year 3+)
  - Paul Swindell (year 1 & 2)
- Industry Participation
  - The Boeing Company (Paul Shelley, Paul Vahey)
  - Sandia National Lab (Dennis Roach)
  - Agilent (formerly A2 Technologies)

# Composite Thermal Damage Measurement with Handheld FTIR

- Motivation and Key Issues
  - Damage detection in composites requires different techniques than metals
  - Incipient thermal damage (ITD) occurs below traditional nondestructive evaluation (NDE) detection limits
- Objective
  - Determine if handheld Fourier transform infrared (FTIR) spectroscopy can detect ITD and guide repair
- Approach
  - Characterize panels with controlled thermal damage using FTIR and perform repair based on FTIR inspection

# Detection Methods for Thermal Damage

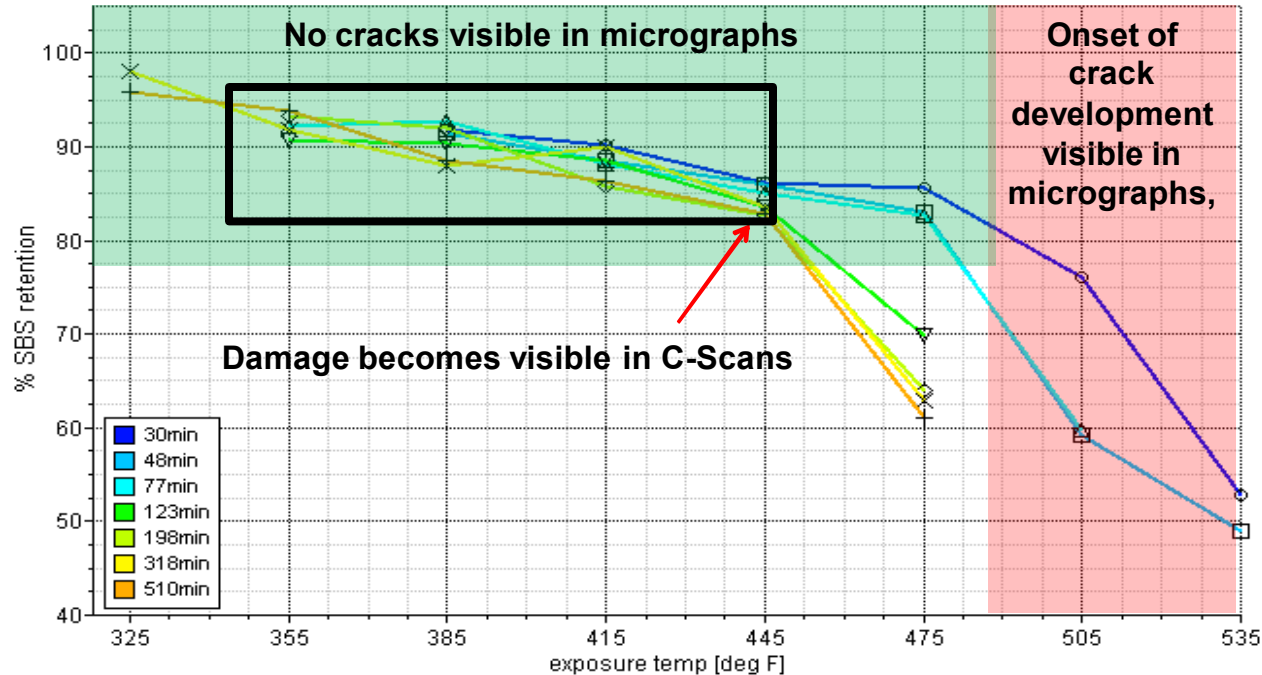
Short Beam Shear Strength Retention vs. Temp./Time – Epoxy 1



- Properties like short beam strength (SBS) degrade before detection possible with ultrasound or visual inspection
  - Damage termed ITD
- Need a method to detect ITD
  - FTIR?

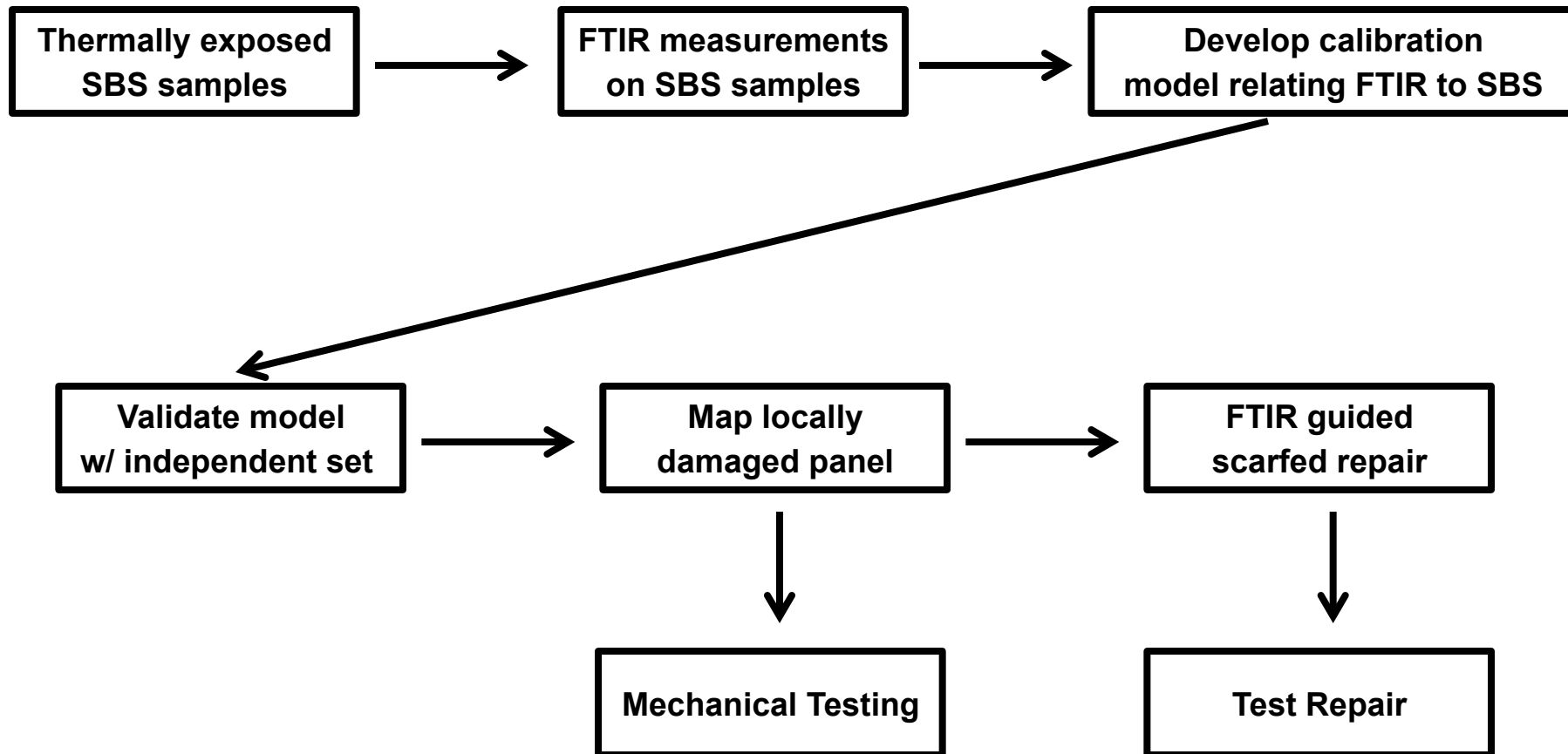
# Detection Methods for Thermal Damage

Short Beam Shear Strength Retention vs. Temp./Time – Epoxy 1



- Properties degrade before detection possible with ultrasound or visual inspection
  - Damage termed (ITD)
- Need a method to detect ITD
  - FTIR?

# Experimental Overview

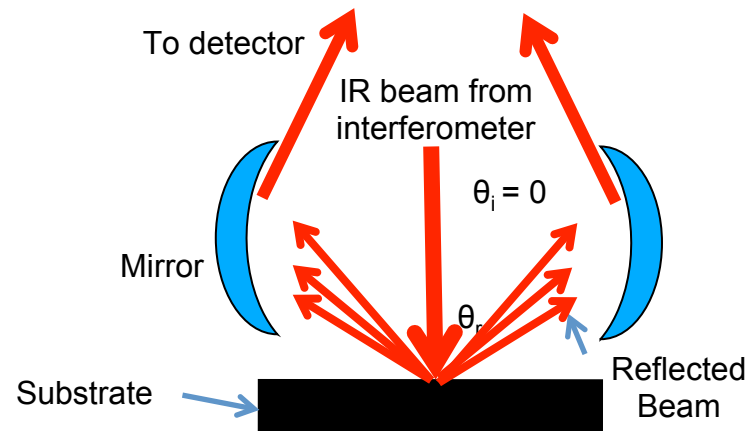


# Materials and Process – FTIR

- Detects chemical changes in the matrix due to thermal degradation
- Mid-IR data region:  $4000\text{ cm}^{-1}$  to  $650\text{ cm}^{-1}$
- Diffuse reflectance sampling interface
- Data collection: 90 coadded scans with  $16\text{ cm}^{-1}$  resolution for background and specimen



ExoScan FTIR



An infrared beam path for diffuse reflectance

# Materials and Process

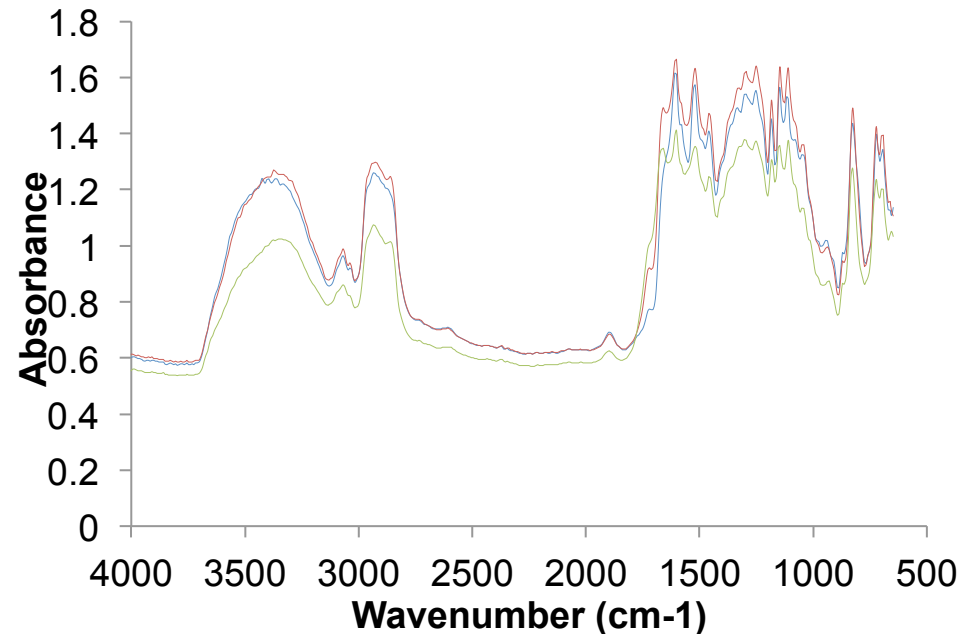
- Toray T800/3900 composites with various levels of thermal damage
  - SBS samples thermally exposed in convection oven
  - Locally damaged panels using heat blanket and insulation
- Sand SBS surfaces with 180 grit  $\text{Al}_2\text{O}_3$  sanding pads
- Measure sanded surface with diffuse reflectance FTIR
  - 3 samples per time/temp exposure
  - 3 measurements per sample

} 9 measurements per exposure level
- Use multivariate analysis to develop calibration model to relate FTIR spectra to SBS values
  - GRAMS IQ software



# Spectral Analysis

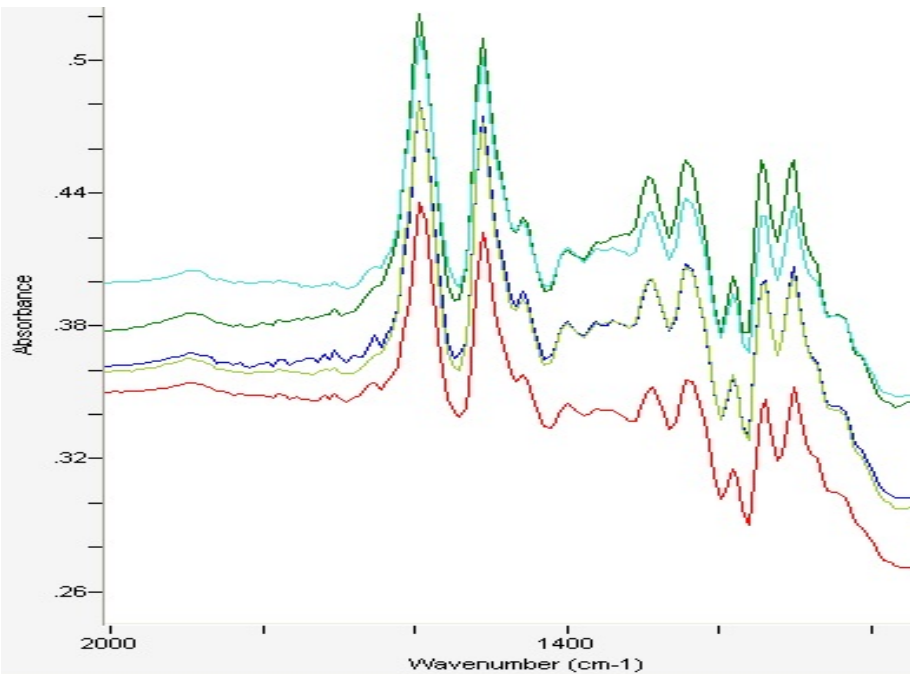
- FTIR spectra of CFRP surfaces complex
  - Multiple constituents → many spectral peaks
- How to analyze spectra with confidence?
  - Multivariate analysis!
- Principal Component Analysis (PCA)
  - Exploratory to identify trends
  - Useful for determining differences and similarities between measurements
  - Used to develop partial least squares (PLS) models



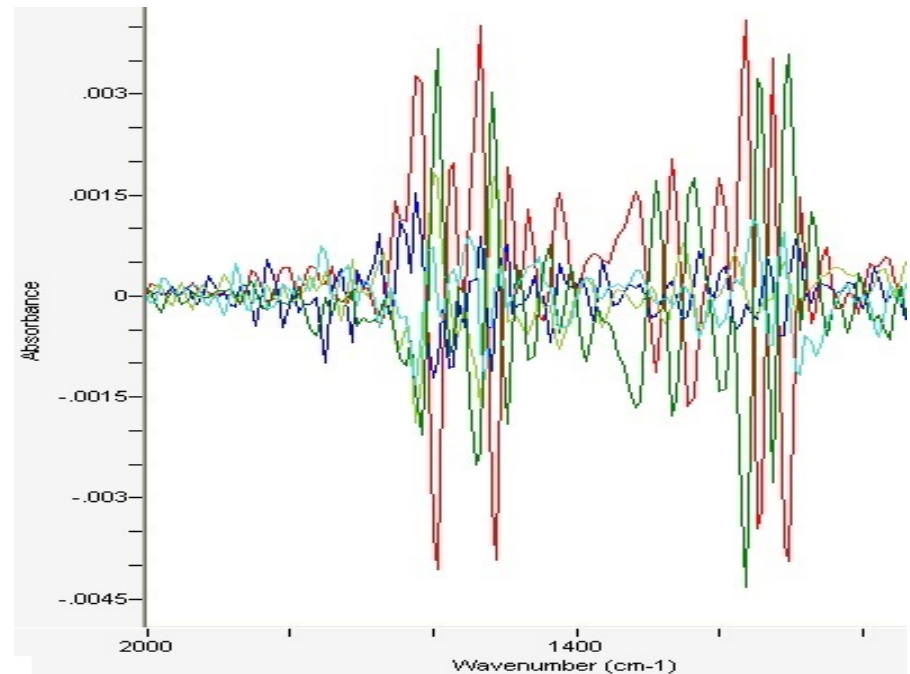
# Developing Model from FTIR Spectra

- FTIR spectra processed to remove baseline effects
  - Savitzky-Golay 1<sup>st</sup> derivative with mean centering and 7pt smoothing
- PLS model relating SBS to FTIR developed using PCA analysis on processed spectra

Raw Spectra



Processed Spectra

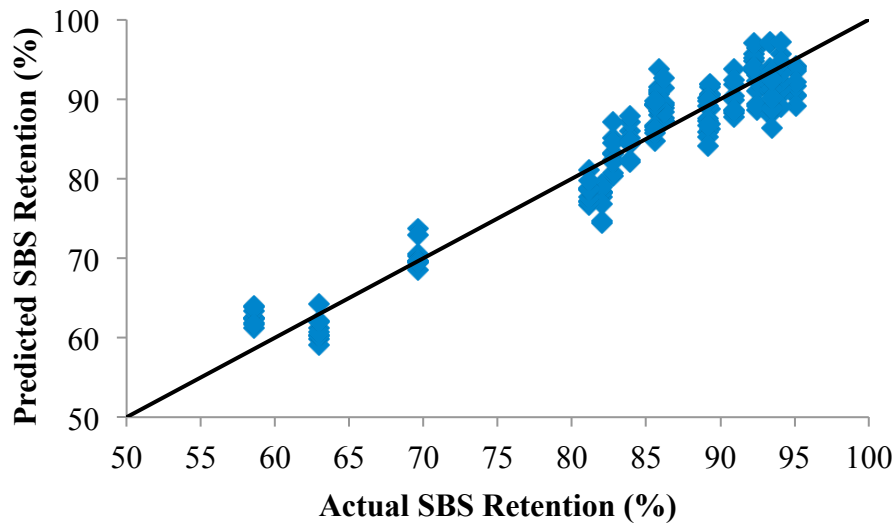


# Calibration Model and Model Validation

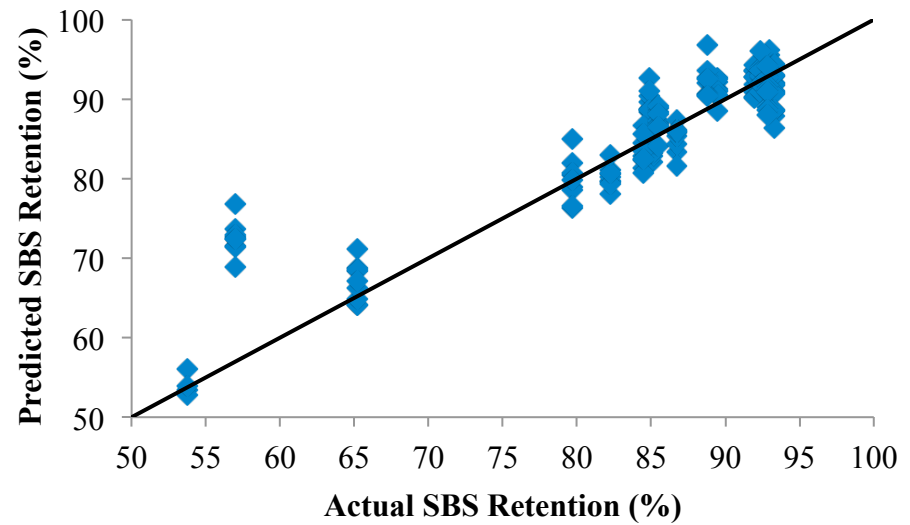
- A PLS model relating the SBS measurements to FTIR spectra was successfully generated for sanded surface
- Model was validated by predicting independent evaluation set
  - Model showed good predictive capabilities of evaluation set (~85% of samples had < 5% error)

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

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

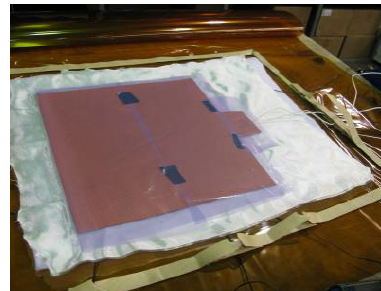
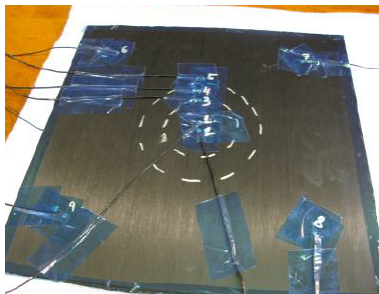


Prediction of independent evaluation set



# Locally Heated Panel Setup

- 12" x 12" panels (24-ply) subjected to localized hotspot
- Local hotspot generated by stacking insulation layers on top of heat blanket in center of panel
- Three peak temperatures (440 °F, 465 °F, 490 °F) exposed for 1 hr



## Insulation Stacking

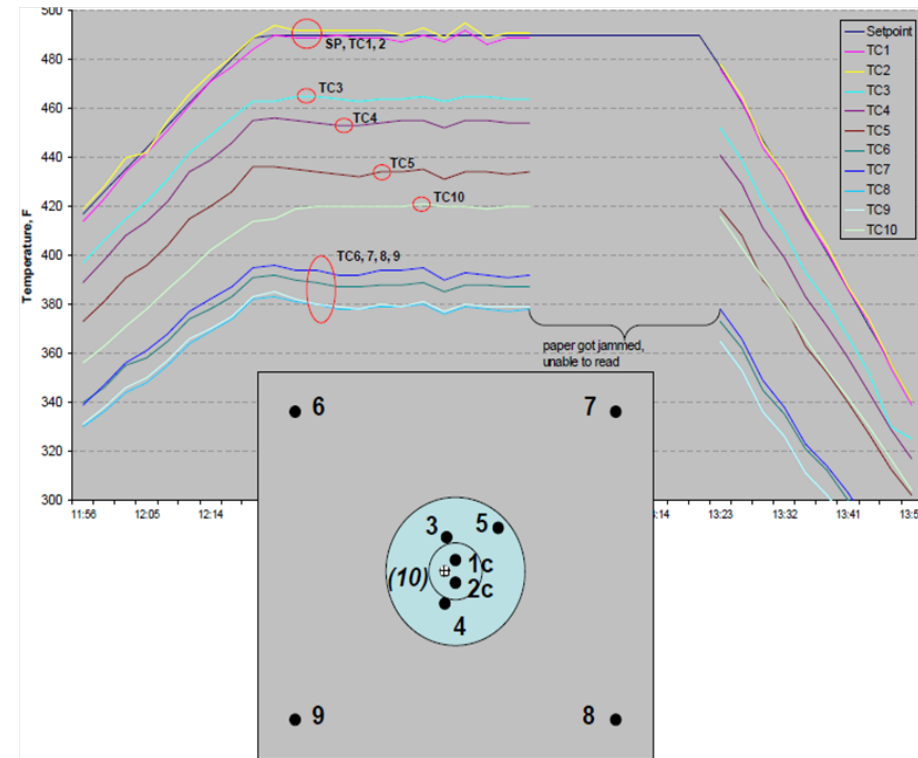
Small

Small

Small ~ 2.75"

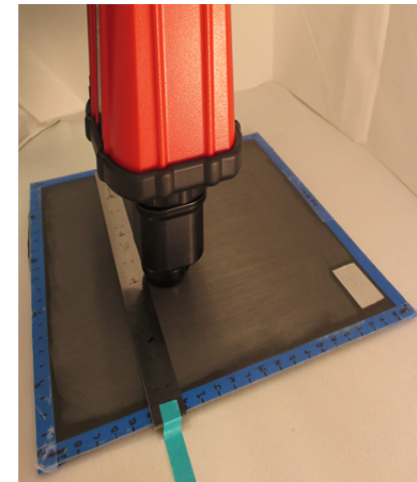
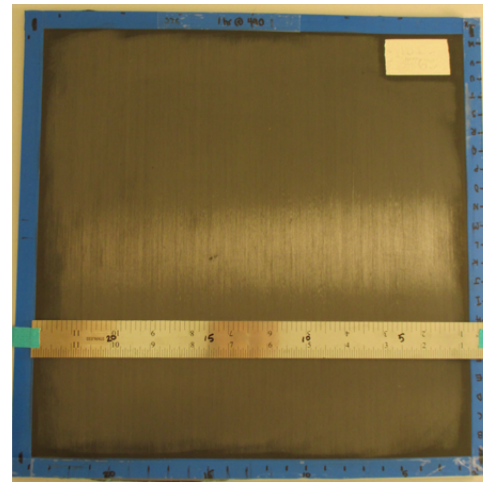
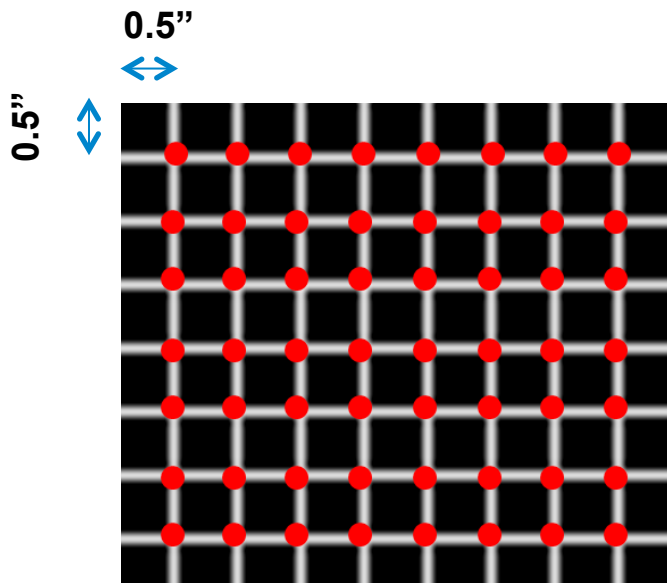
Intermediate ~ 3.75 "

Large ~ 4.75"



# Panel Mapping Procedures

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

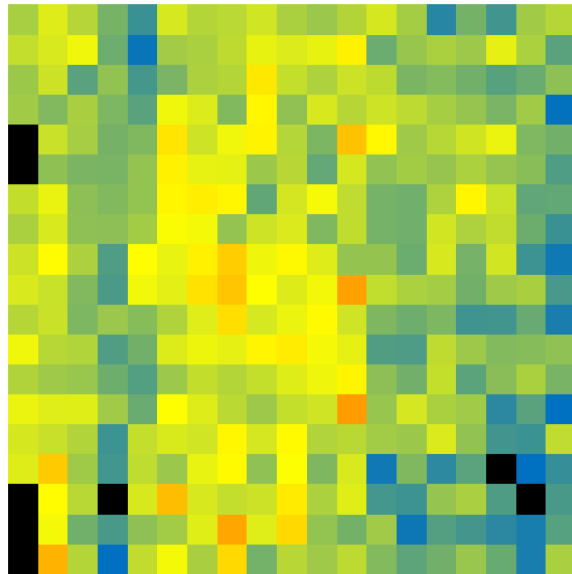


● Measurement location

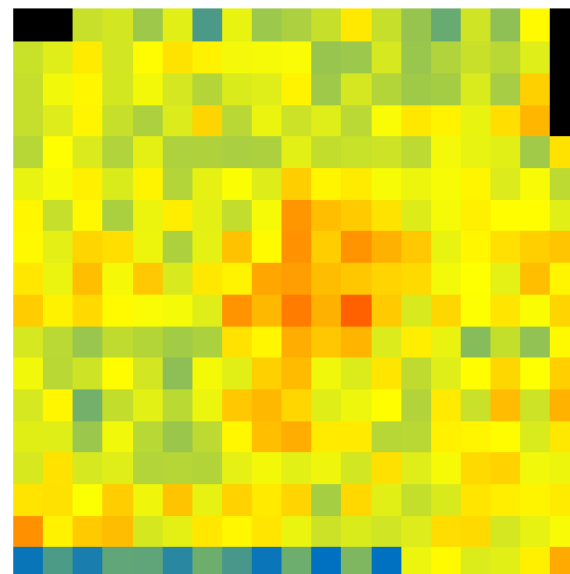
# Panel Mapping Results

- SBS retention predicted for each point using calibration model
- Each square represents SBS retention prediction from FTIR measurement taken at a grid point on the panel

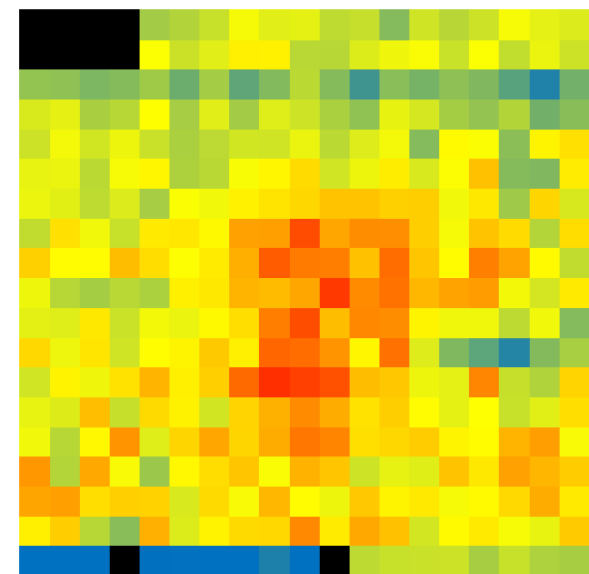
1 hr @ 440 °F



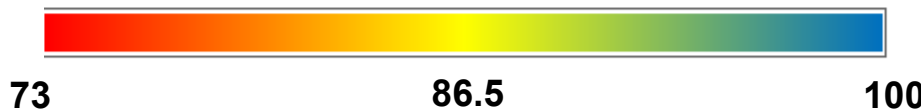
1 hr @ 465 °F



1 hr @ 490 °F



Predicted SBS Retention(%)



■ = bad or no measurement

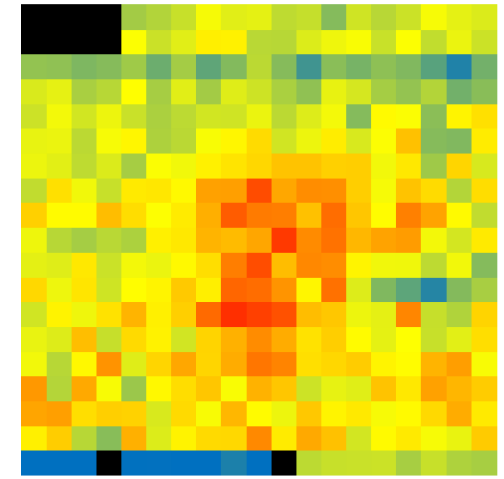
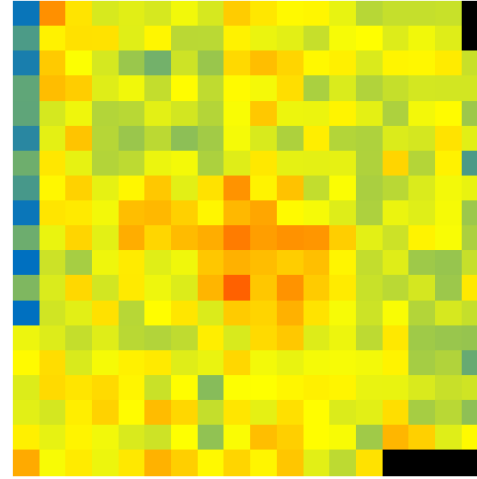
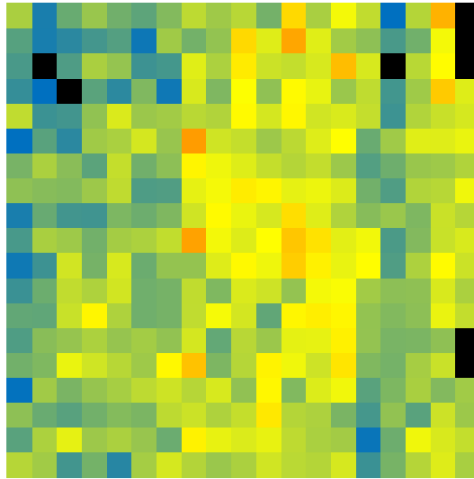
# Comparison of Panel Mapping

1 hr @ 440 °F

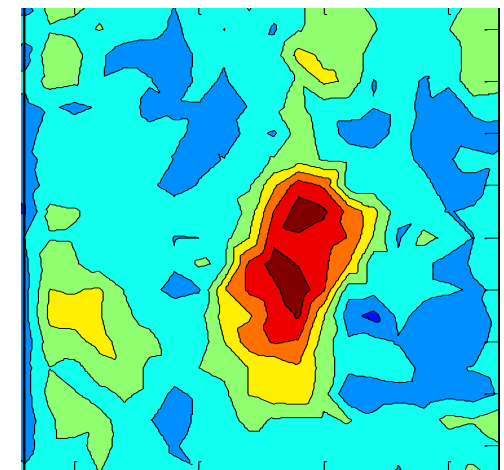
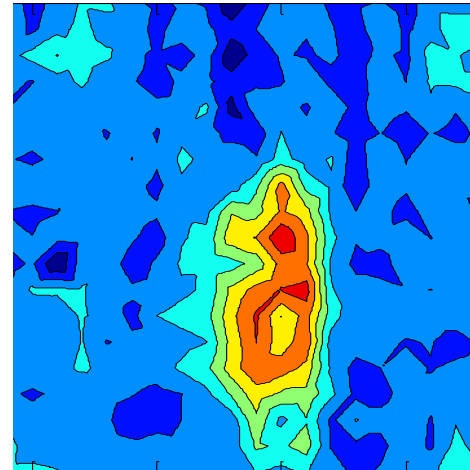
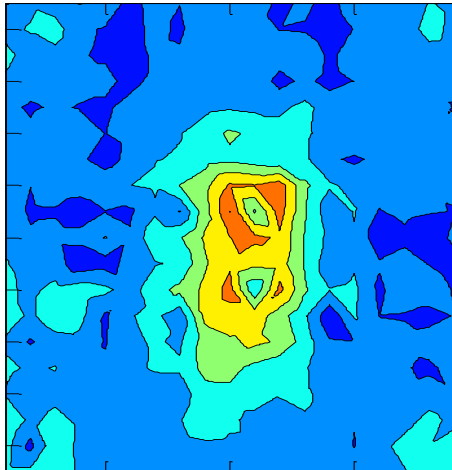
1 hr @ 465 °F

1 hr @ 490 °F

Sanded  
Surface



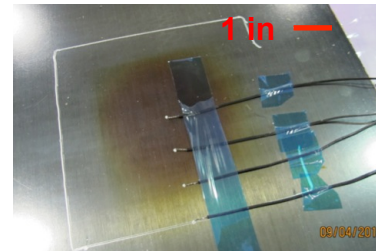
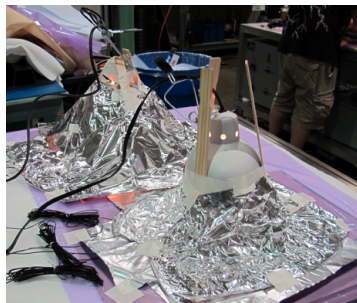
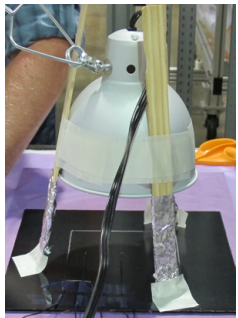
Resin Rich  
Surface  
(1<sup>st</sup> year)



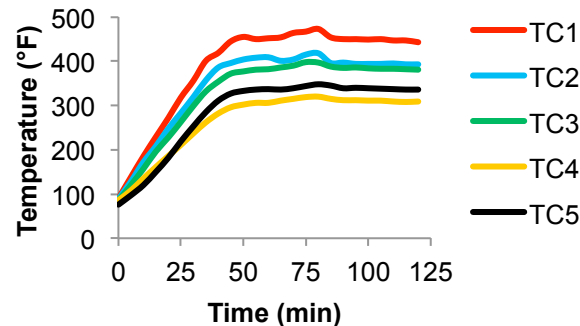
Note: Scaling, panel orientation, and color scheme are different

# Mechanical Testing

- 12" x 12" quasi-isotropic panels (24-ply) locally heated using a heat lamp
  - Max exposure ~ 450 °F for 1 hr
- Panel cut up into alternating strips for SBS and dynamic mechanical analysis (DMA) samples
  - Panel too thick for DMA samples → specimens cut from surface exposed to heat



Side view

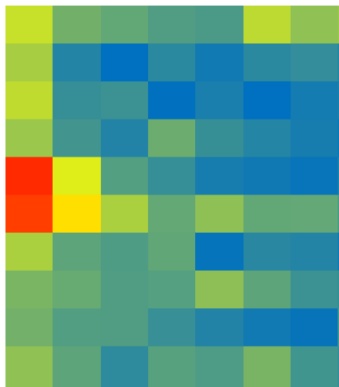




# SBS Measurements

- SBS measurements did not exhibit strong sensitivity to localized thermal exposure
  - Surface damage vs. max shear in center of the part
- Predictions of SBS from FTIR spectra were reasonable
  - ~95 % of predictions had < 10 % error

Map (half of panel)

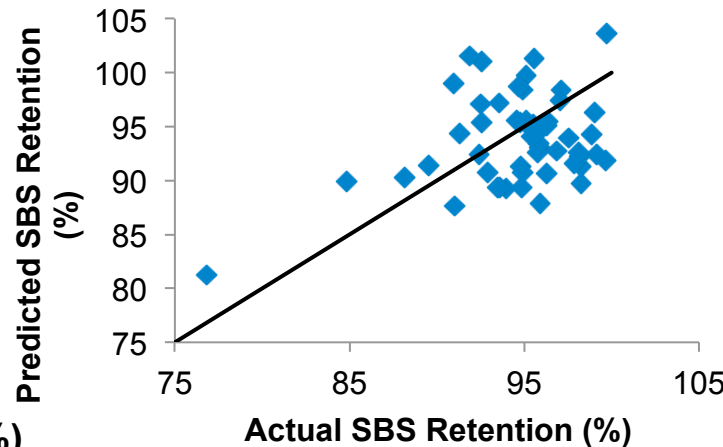


Measured SBS Retention(%)

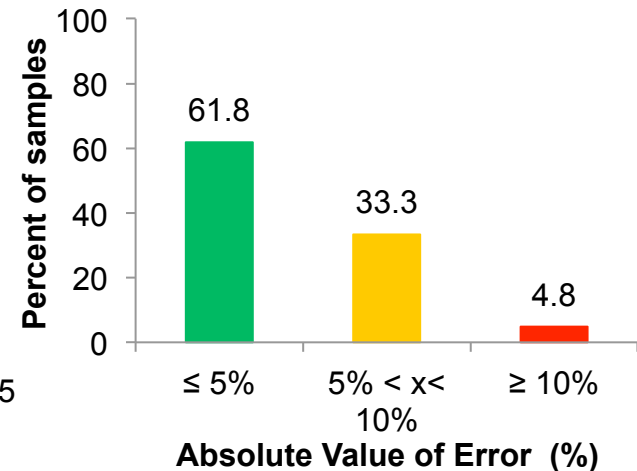


73                      86.5                      100

SBS Predicted vs. Actual



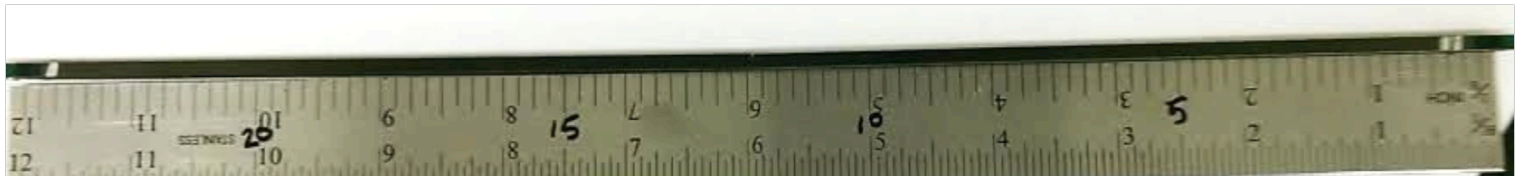
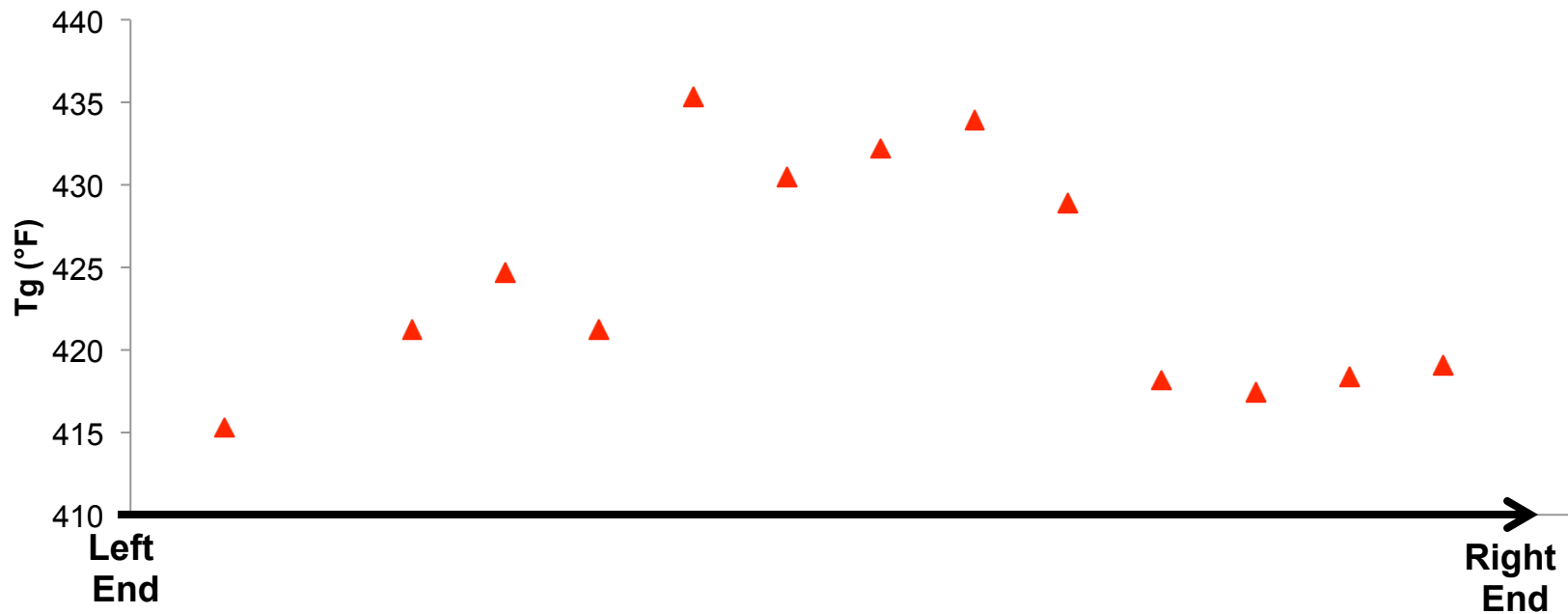
Prediction Error



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

# DMA Measurements

- Tg measurements sensitive to localized thermal exposure
  - Maximum increase of ~ 17-18 °F in thermally exposed region

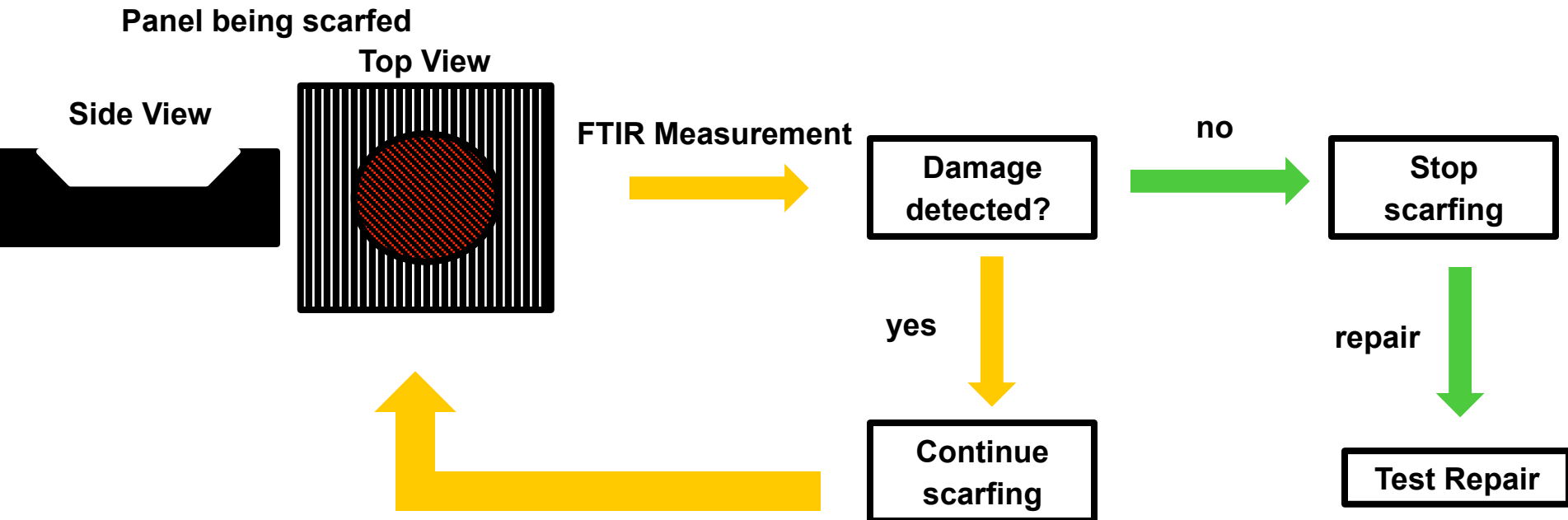


# Summary

- PLS model to relate FTIR spectra to SBS was successfully developed and validated
- Surface mapping of panels with localized heat damage completed
- Preliminary mechanical testing performed

# Future Work



- Perform scarfed repair guided by FTIR
- Test scarfed repair
- Mechanical testing on samples from locally heated panel from 1<sup>st</sup> and 2<sup>nd</sup> year of project



# Looking Forward

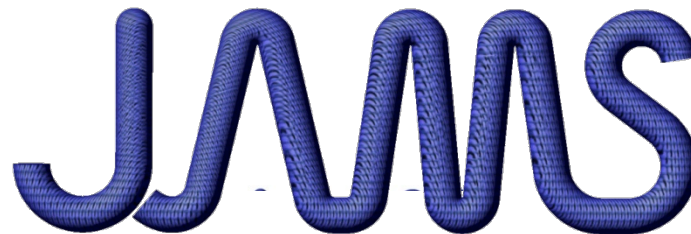
- Benefit to Aviation
  - Improved thermal damage detection
  - Greater confidence in repairs
- Future needs
  - Application to other composite systems
  - Other applications of handheld FTIR
    - Chemical damage
    - Surface prep for bonding

# Acknowledgements

- **FAA, JAMS, AMTAS** 
- **Boeing Company** 
  - Paul Vahey, Paul Shelley, Megan Watson, John Spalding
- **Sandia National Labs** 
- **Agilent Technologies**  **Agilent Technologies**
- **UW MSE** 
- **Flinn Group:**
  - Jonathon Morasch, David Pate

Questions and comments are  
strongly encouraged

Thank you



JOINT ADVANCED MATERIALS & STRUCTURES  
CENTER OF EXCELLENCE

