Summary of Marine and Hydrokinetic (MHK) Composites Testing at Montana State University

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What’s Presented today?

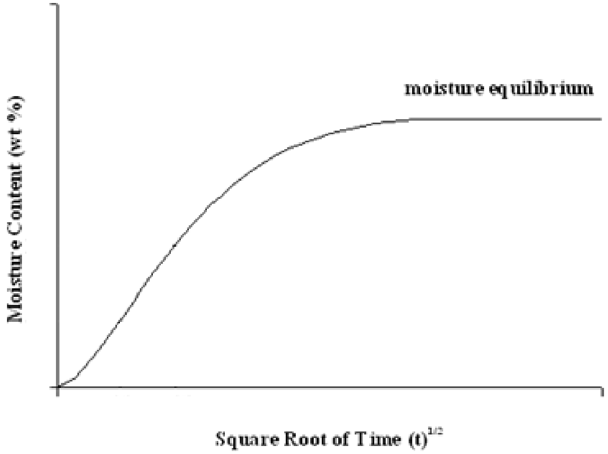
- This collection of work details four areas of investigation within the DOE/SNL/MSU marine hydrokinetic (MHK) energy materials effort.
    - first section investigates the effect of moisture uptake into a continuous fiber composite, considering the effect of an applied uniaxial tensile stress on diffusion rate and maximum mass uptake.
    - second section investigates damage development and propagation in composite materials due to moisture uptake. Included in these experimental results are mechanical strength and in-situ acoustic emission results.
    - third section investigates the effect of moisture uptake on glass composites with differing fiber angle and layup sequences. Both mechanical strength and in-situ acoustic emission results are presented for unidirectional and symmetric cross-ply coupons.
    - fourth section investigates the strength reduction and in-situ acoustic emission results for a wide breadth of fiber reinforced composite materials before and after moisture update. The evaluated coupons were provided from industrial suppliers and tested as potential materials for MHK applications

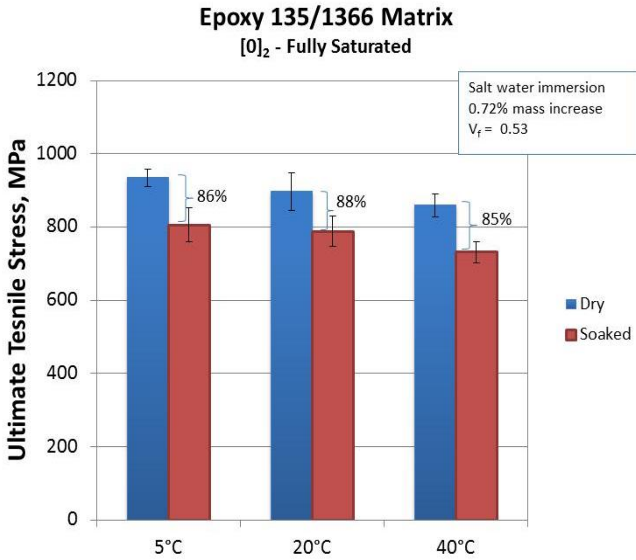
Problem Definition

- To cultivate a successful industry it becomes pertinent to develop a comprehensive understanding of immersed MHK structures

- Well documented that composite materials absorb moisture
  - Significant mechanical and physical degradation
  - Primarily unstressed systems investigated

- Structure will be subjected to stresses
  - Becomes vital to understand what effects these stresses have on the moisture absorption process in composite material systems

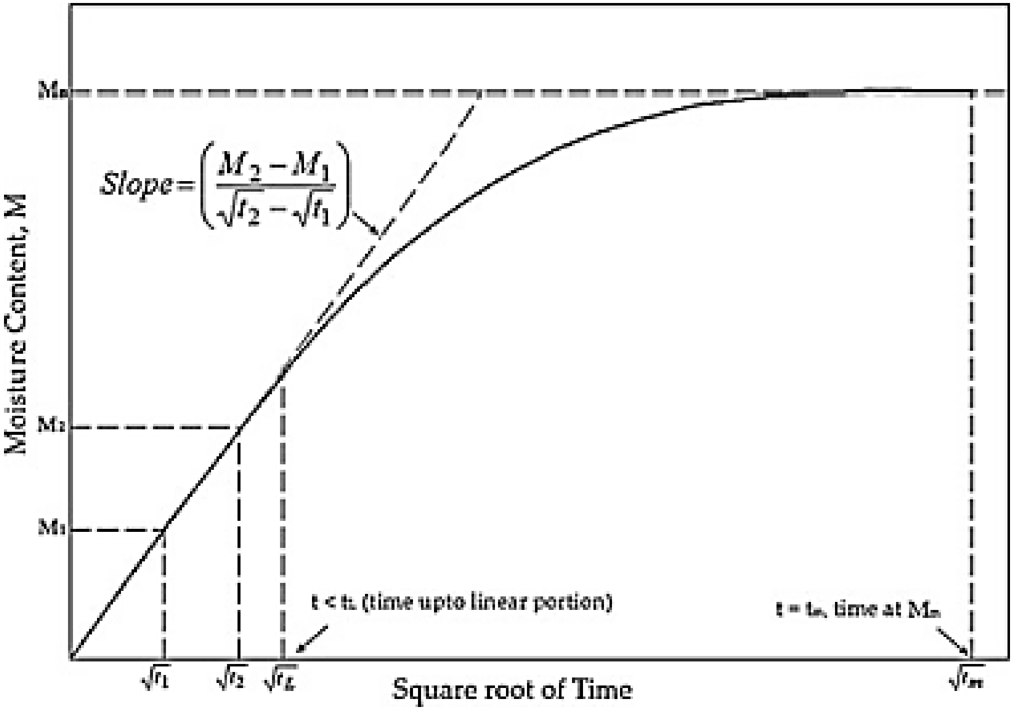




Problem Definition

- Seek to fully characterize the effects of tensile stresses on the moisture diffusion characteristics of Epoxy Glass composites
  - To gain a clear understanding of the mechanisms at work the effects of varying both fiber angle and magnitude of applied stress will be investigated

Fickian Uptake Curve



- Initially linear uptake region, transitions to non-linear
- Asymptotically approaches Maximum Percent Moisture Content, M_∞
  - Pure Epoxy resin systems $M_\infty = 2.5 - 3.0\%$
- All Fickian materials will demonstrate a curve of this shape

Diffusivity

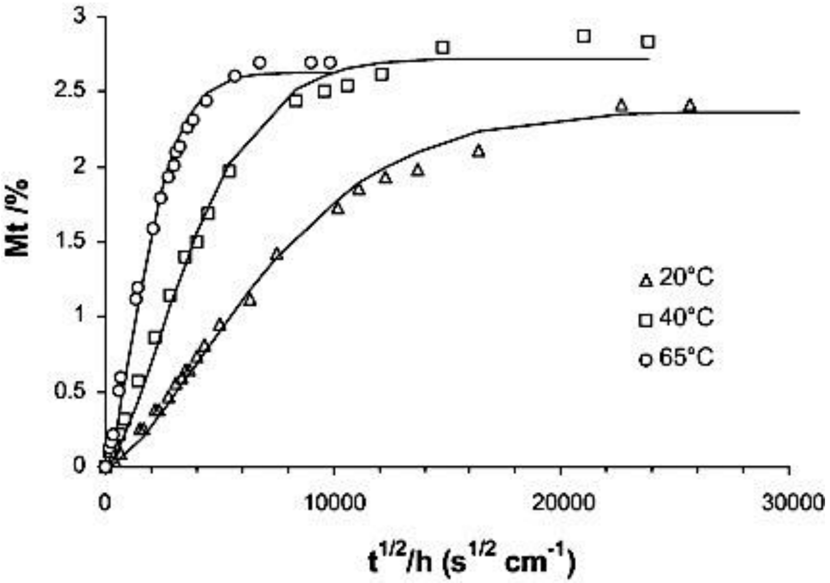
- Diffusion coefficient *D* is a rate constant which relates mass flux to the concentration gradient
  - Units (length^2/time)
  - Defines the rate at which mass diffuses into a concentration gradient
  - Directly proportional to initial slope of the uptake curve

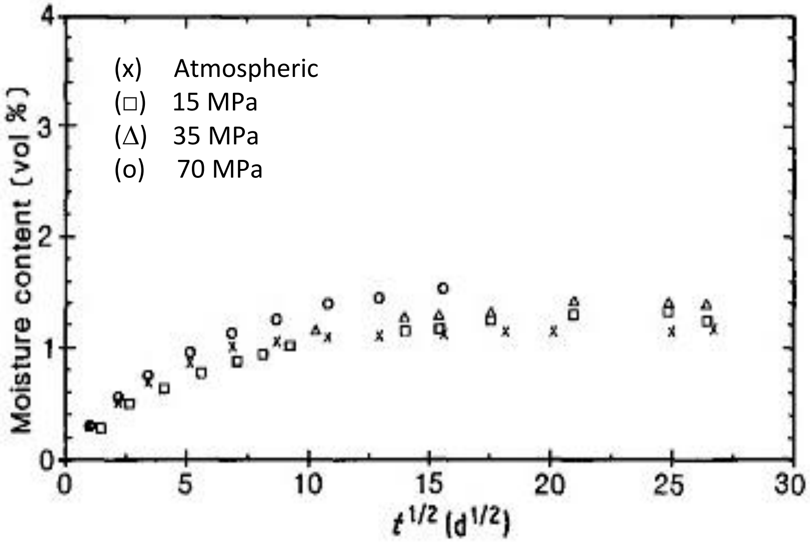
- For a homogenous thin plate,

$$D = \pi \left(\frac{h}{4M_\infty}\right)^2 \left(\frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}}\right)^2$$

Temperature and Pressure Effects

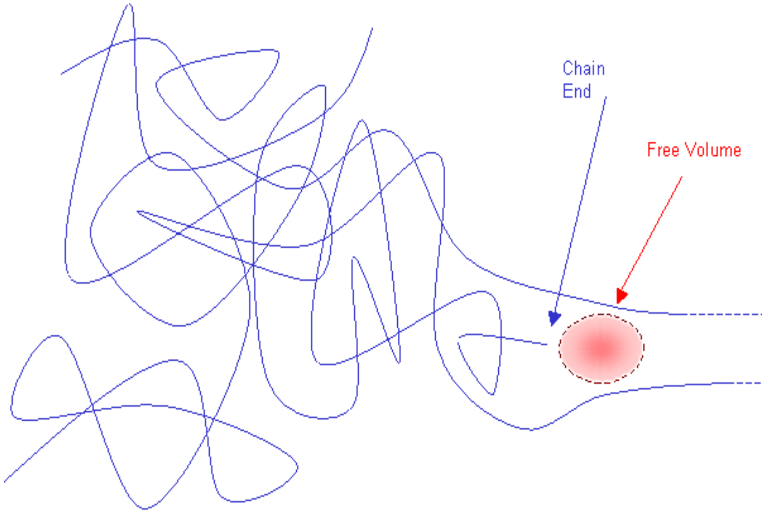
- Ambient Temperature
  - Diffusivity changes
  - Maximum content unaltered
  - Important to compare at same ambient temperature
- Hydrostatic Pressure (where 10 MPa roughly equates to 1000m of sea depth)
  - Diffusivity unaltered
  - Maximum content unaltered





Free Volume (v_f)

- The free volume is a fundamental quantity in polymeric systems
  - Small amount of unfilled volume at the end of a polymer chain
  - Mathematically, the free volume is defined as the difference between the measured volume and occupied volume



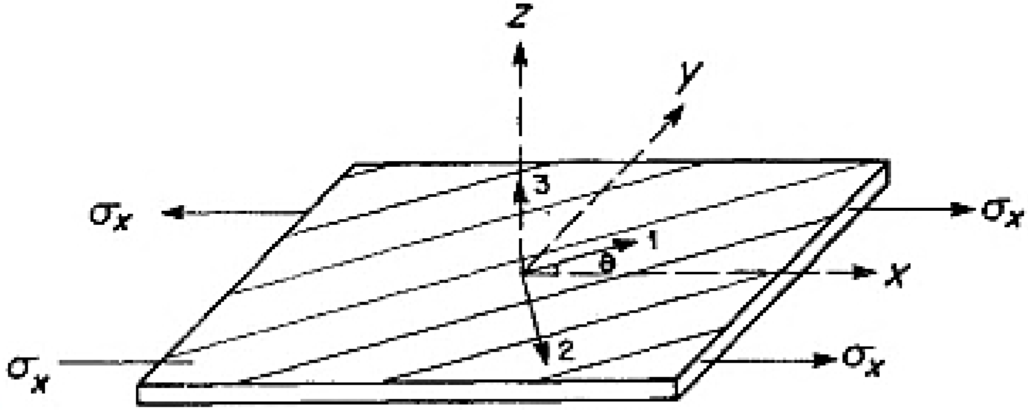
- Free volume theories are used as a basis in describing molecular movement (moisture diffusion e.g.) in polymer systems.

Volume Strain of the Matrix

- Recall, only the matrix absorbs moisture
  - Therefore, only changes in the free volume of the matrix will cause changes in moisture diffusion parameters.

$$\upsilon_{f\sigma} = \upsilon_{f0} + (\Delta V/V_0)_m$$

$$(\Delta V/V_0)_c = (\Delta V/V_0)_m \phi_m + (\Delta V/V_0)_f \phi_f$$



Volume Strain of the Matrix

- Through laminate plate theory the value for the volumetric strain of the matrix is found…
  - Function of applied tensile stress (σ_x), fiber angle (θ), fiber volume fraction (ϕ), and elastic properties of the constituents (E and ν for composite and fibers).

$$(\Delta V/V_0)_m \phi_m = \sigma_x \left\{ cos^2\theta \left[\left(\frac{1-2\nu_{12c}}{E_{1c}} \right) - \phi_f \left(\frac{1-2\nu_{12f}}{E_{1c}} \right) \right] \right.$$

$$\left. + sin^2\theta \left[\left(\frac{1}{E_{2c}} - \frac{\nu_{12c}}{E_{1c}} - \frac{\nu_{23c}}{E_{2c}} \right) - \phi_f \left(\frac{1}{E_{2f}} - \frac{\nu_{12f}}{E_{1f}} - \frac{\nu_{23f}}{E_{2f}} \right) \right] \right\}$$

Changes in Diffusion Parameters

Maximum Moisture Content

$$v_{f\sigma} = v_{f0} + (\Delta V/V_0)_m \quad \text{and} \quad v_{f0} = \mathrm{M}_{\infty 0}\frac{\rho_m}{\rho_w}$$

$$\mathrm{M}_{\infty\sigma} = v_{f\sigma}\frac{\rho_w}{\rho_m}$$

$$\mathrm{M}_{\infty\sigma} = \left[v_{f0} + (\Delta V/V_0)_m\right]\frac{\rho_w}{\rho_m}$$

$$\boxed{\mathrm{M}_{\infty\sigma} = \mathrm{M}_{\infty 0} + (\Delta V/V_0)_m\frac{\rho_w}{\rho_m}}$$

Changes in Diffusion Parameters

Diffusivity

$$\upsilon_{f\sigma} = \upsilon_{f0} + (\Delta V/V_0)_m \quad \text{and} \quad \upsilon_{f0} = \mathrm{M}_{\infty 0}\frac{\rho_m}{\rho_w}$$

$$\ln\frac{D_\sigma}{D_0} = \frac{a}{\phi_m}\left(\frac{1}{\upsilon_{f0}} - \frac{1}{\upsilon_{f\sigma}}\right)$$

$$\boxed{\ln\frac{D_\sigma}{D_0} = \frac{a}{\phi_m}\frac{(\Delta V/V_0)_m}{\upsilon_{f0}\left[\upsilon_{f0} + (\Delta V/V_0)_m\right]}}$$

Recap

- Began with moisture absorption of composite materials, Springer (1976).
  - $D_{1,2,3}$, $D_{x,y,z}$, and D for unstressed composite plate
- Free volume theories to describe diffusion in polymers
  - Free volume changes → Changes in diffusion parameters
  - Neumann (1986): $\mathrm{M}_{\infty} = v_f \frac{\rho_w}{\rho_m}$
  - Hurt (1980): $\ln \frac{D_\sigma}{D_0} = a\left(\frac{1}{v_{f0}} - \frac{1}{v_{f\sigma}}\right)$

Continued…

- Laminate Plate Theory to calculate volume change of the only the polymer matrix
  - $\upsilon_{f\sigma} = \upsilon_{f0} + (\Delta V/V_0)_m$
  - $\mathrm{M}_{\infty\sigma} = \mathrm{M}_{\infty 0} + (\Delta V/V_0)_m \frac{\rho_w}{\rho_m}$
  - $\ln \frac{D_\sigma}{D_0} = \frac{a}{\phi_m} \frac{(\Delta V/V_0)_m}{\upsilon_{f0}\left[\upsilon_{f0} + (\Delta V/V_0)_m\right]}$
- All input parameters are know quantities:
  - Stress (σ_x), fiber angle (θ), volume fraction (ϕ), densities of fluid and matrix (ρ), and elastic properties of the constituents (E and ν for composite and fibers).

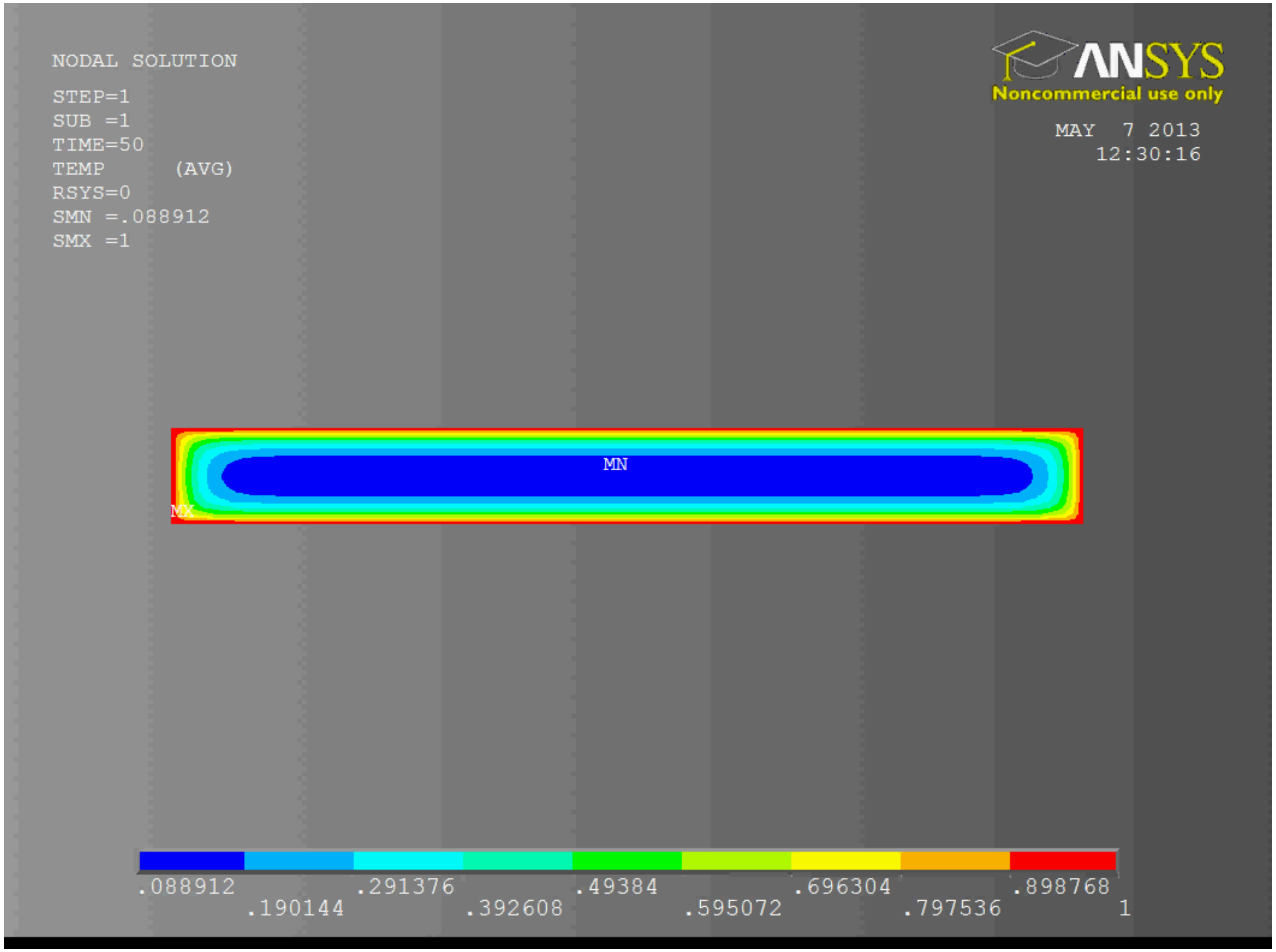
Finite Element Analysis

- ANSYS 13.0 – strong time dependent analysis tools
- Thermal-Moisture Diffusion Analogy as presented by Wong and Koh (2002)
  - Fourier Heat diffusion ←→ Fickian Mass Diffusion

$$\frac{\partial \mathrm{C}}{\partial \mathrm{t}} = \mathrm{D}\left(\frac{\partial^2 C}{\partial x^2} + \frac{\partial^2 C}{\partial y^2} + \frac{\partial^2 C}{\partial z^2}\right)$$

$$\frac{\partial \mathrm{T}}{\partial \mathrm{t}} = \frac{k}{\rho c}\left(\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2}\right)$$

Property	**Thermal**	**Moisture**
Field Variable	Temperature, T	Saturation Ratio, w
Density	ρ (kg/m^3)	1
Conductivity	k (W/m °C)	$D \times M_\infty$ (mm^2/hr)
Specific Capacity	c (J/kg °C)	M_∞



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SMX =1
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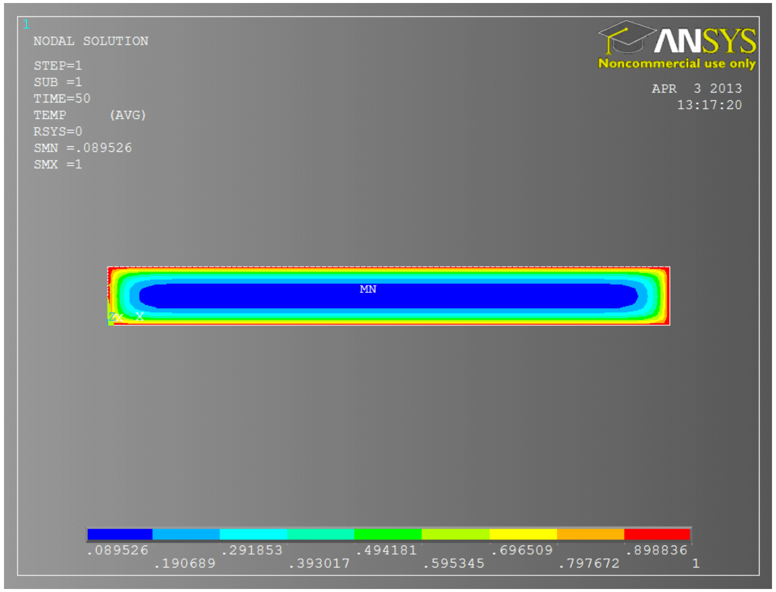

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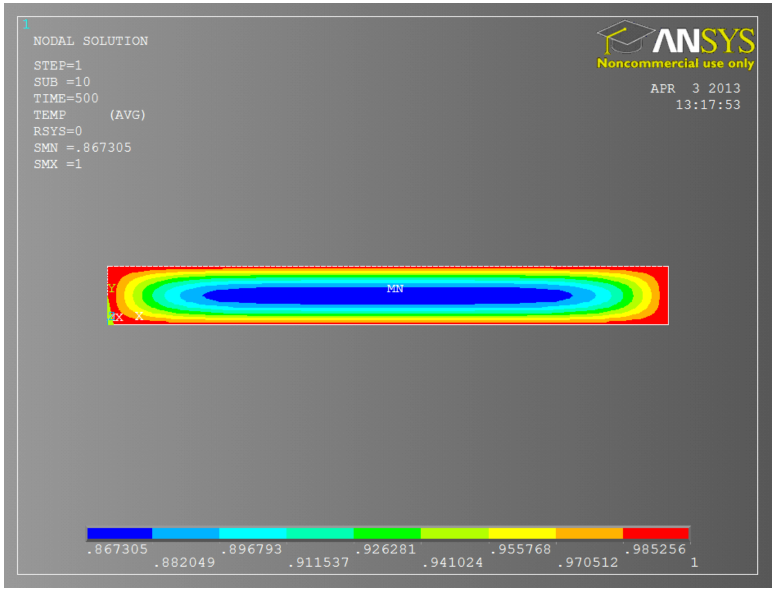

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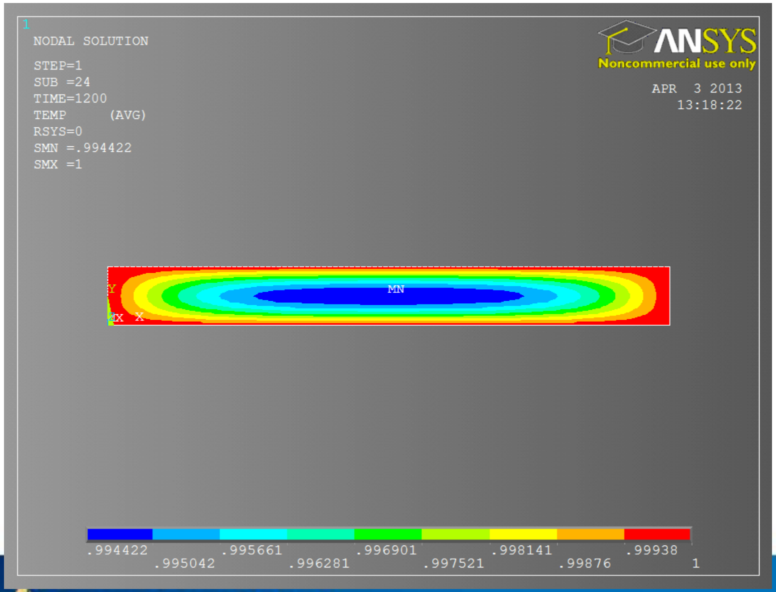

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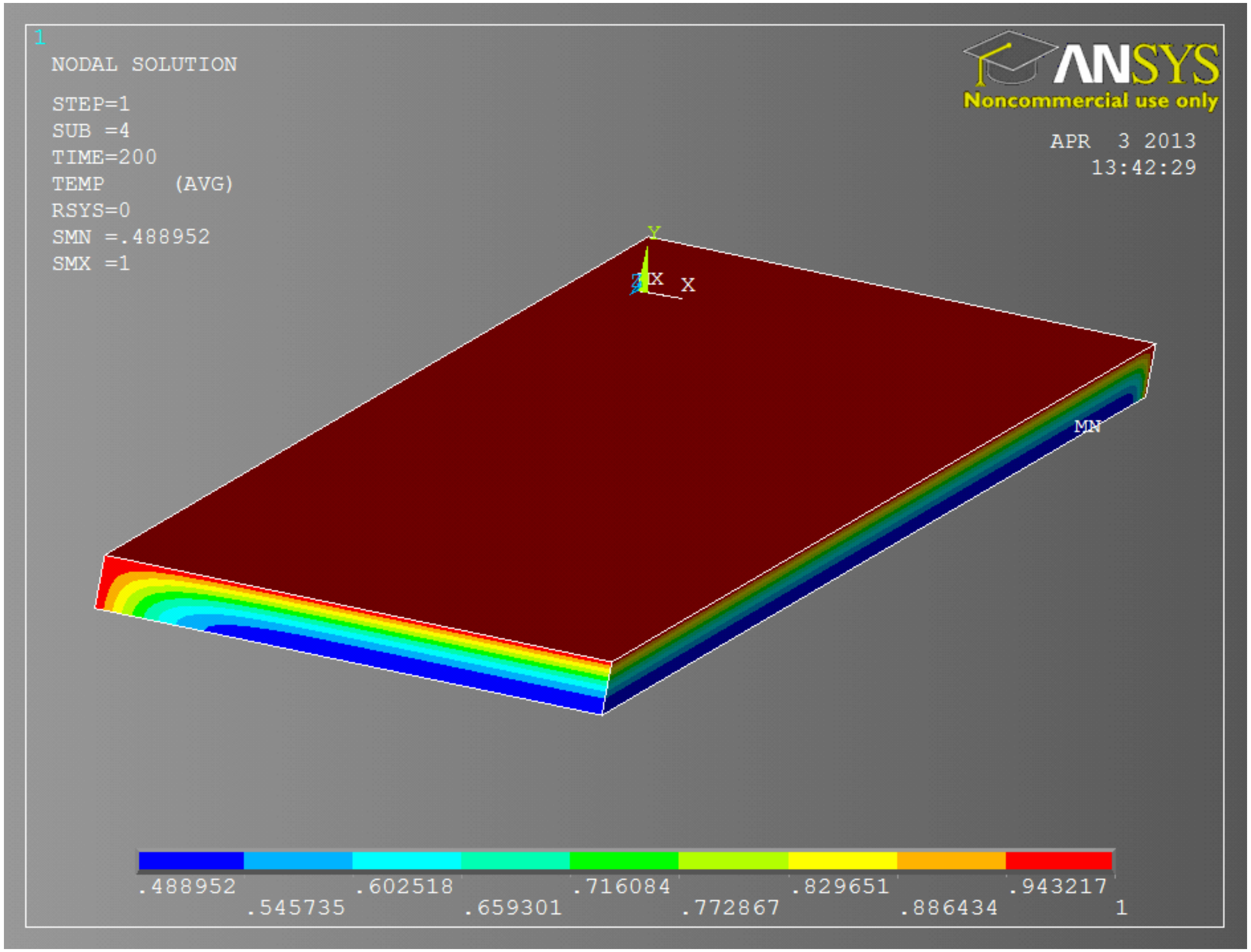

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FEA continued

- Diffusivity defined separately in each axes direction (D_x, D_y, and D_z)
  - In order to verify FE code and thermal-moisture analogy the effective system diffusivity D calculated was through reproduced uptake curves

$$M(t) = \left(\frac{\Sigma\, Temperature\ at\ each\ node}{Total\ number\ of\ nodes}\right) M_\infty$$

$$\mathrm{D} = \pi \left(\frac{h}{4M_\infty}\right)^2 \left(\frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}}\right)^2$$

Experimental Procedures

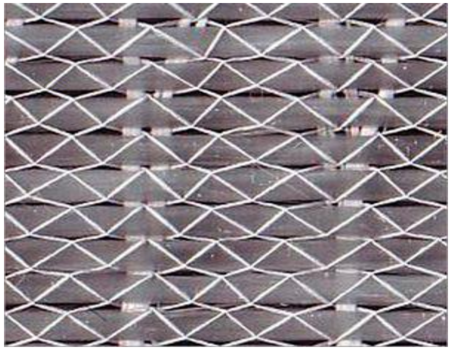
- Overview
- Manufacturing
- Sample Preparation
- Weight Gain Measurements

Experiment Overview

- Goal is to experimentally validate proposed model and finite element simulation by immersing stressed unidirectional FRP composite samples
    - Varying both magnitude of applied tensile stress and the fiber angle

Manufacturing

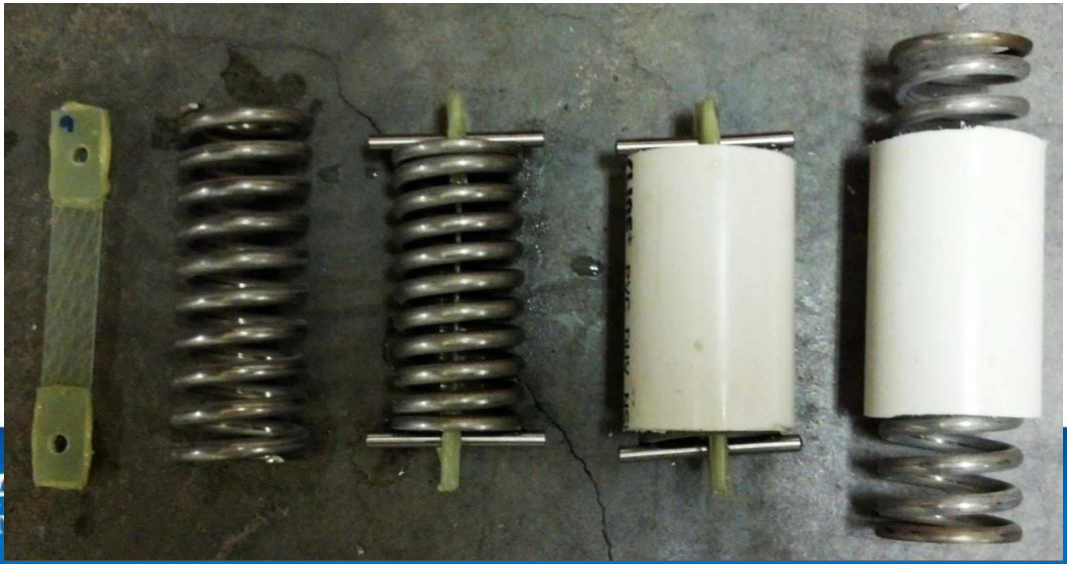
- Momentive's Epikote epoxy resin system
- Saertex U14EU920 series glass fiber stitched fabric. By weight:
  - 91% at 0-degree orientation
  - 8% at 90-degree orientation
  - 1% comprised of fabric stitching

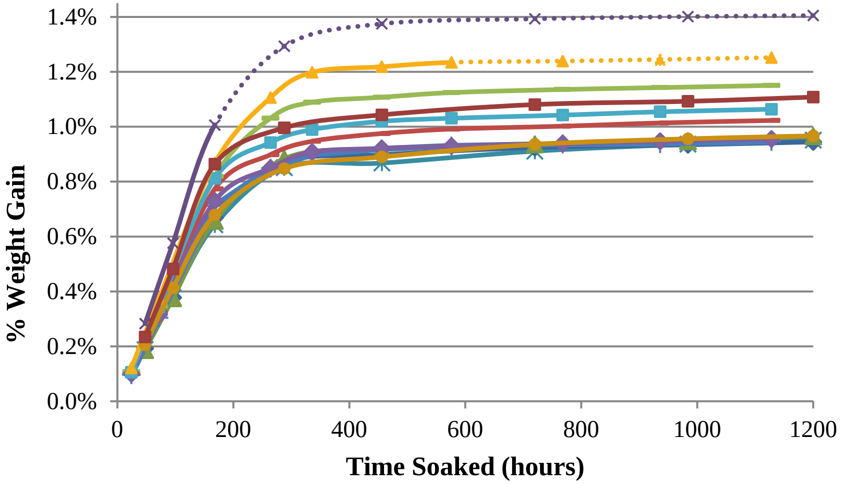


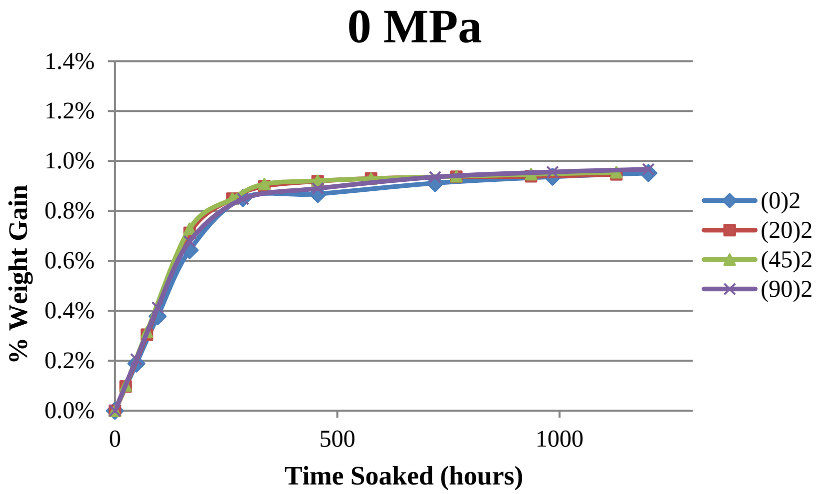
- All samples were cut from a single unidirectional fiber composite plate manufactured using Vacuum Assisted Resin Transfer Molding (VARTM) Process
  - 30 x 20 inch, two-ply thick, 0-degree

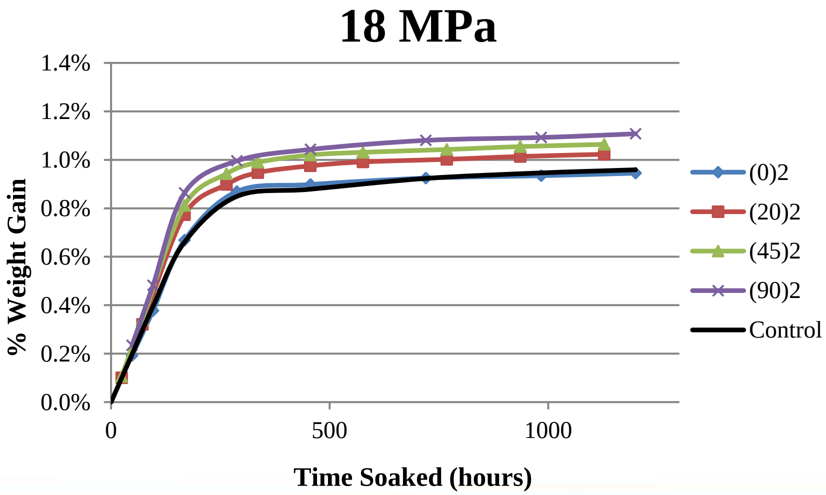
Sample Preparation

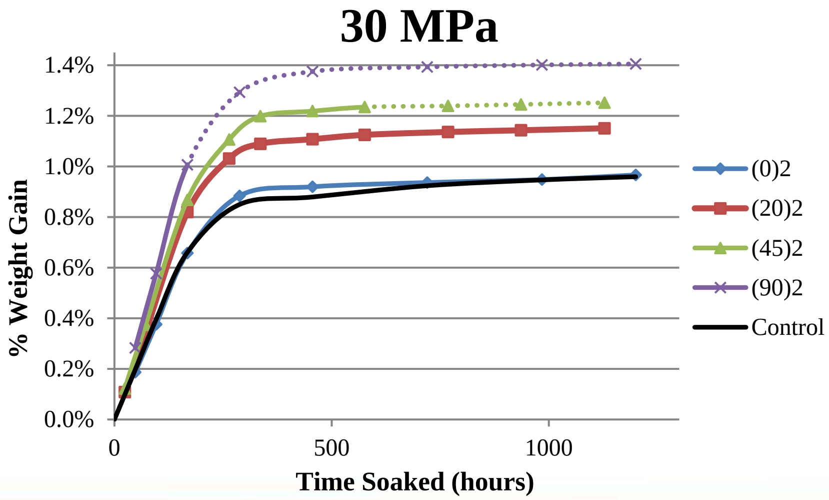
- Sample size 4.5 x 0.6 inch
  - Samples cut at desired fiber orientation
  - One-inch tabs adhered at ends of samples
  - Holes drilled for Stainless Steel restraining pins
- Stainless Steel compression springs used to apply tensile stress

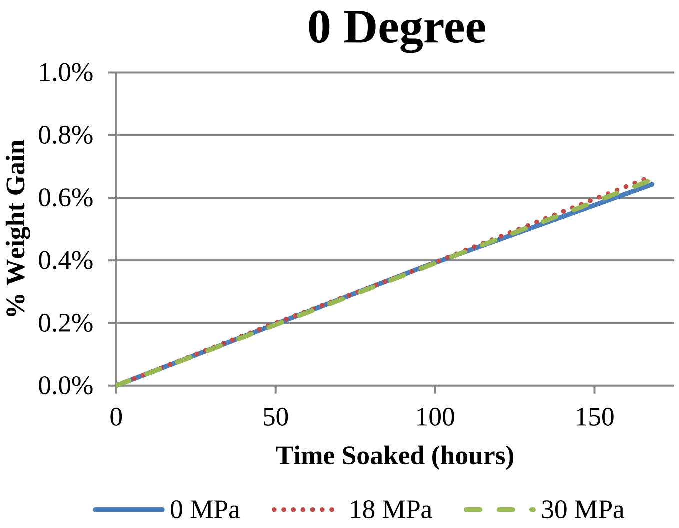


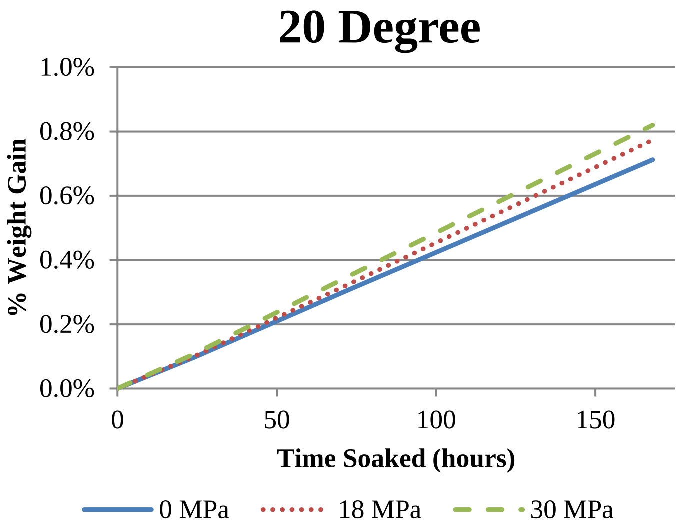


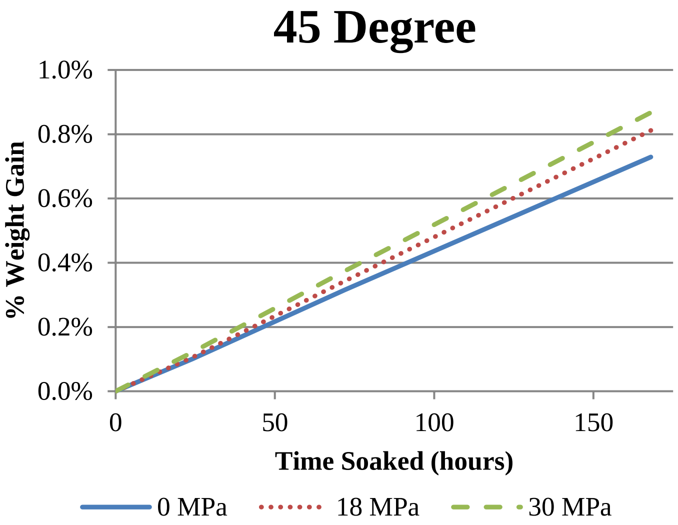

0 MPa

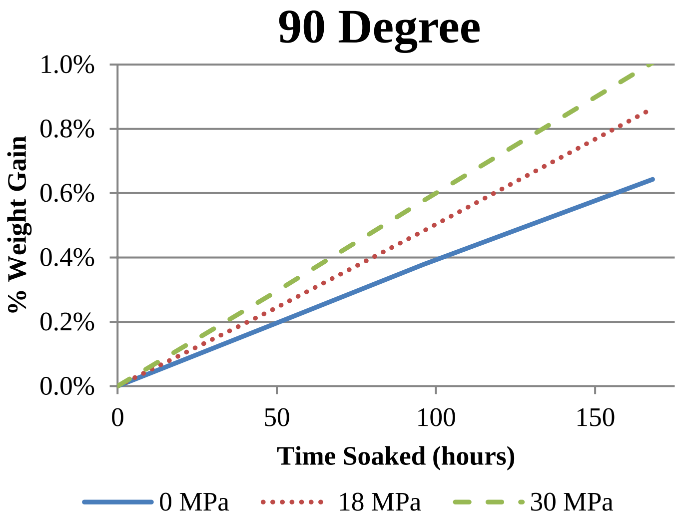

18 MPa


30 MPa


0 Degree


20 Degree


45 Degree


90 Degree

- All 45 and 90 degree samples loaded at 30 MPa fractured prior to achieving full saturation
  - Significant mechanical degradation
  - Apparent crack propagation along fiber-matrix interface

Maximum Moisture Content

		σ_x	M_∞ (%)			Percent Error (%)	
θ(deg)	**ϕ_f**	**(MPa)**	**Experimental**	**ANSYS**	**Model**	**ANSYS**	**Model**
0	0.52	0	0.9692	1.0652	1.0652	9.91	9.91
		18	0.9453	1.0703	1.0676	13.22	12.94
		30	0.9758	1.072	1.0718	9.86	9.84
20	0.52	0	0.9466	1.0651	1.0652	12.52	12.53
		18	1.0235	1.0773	1.0776	5.26	5.29
		30	1.151	1.085	1.0852	-5.73	-5.72
45	0.52	0	0.9559	1.0652	1.0652	11.43	11.43
		18	1.0644	1.1031	1.1027	3.64	3.60
		30	1.2523**	1.1354	1.1349	-9.33	-9.37
90	0.52	0	1.0102	1.0652	1.0652	5.44	5.44
		18	1.1246	1.1363	1.1358	1.04	1.00
		30	1.4057**	1.1836	1.1829	-15.80	-15.85

** Sample fracture prior to achieving full saturation

ANSYS and Model: $\mathrm{M}_{\infty\sigma} = \mathrm{M}_{\infty 0} + (\Delta V/V_0)_m \dfrac{\rho_w}{\rho_m}$

Diffusivity Values

- Experimental: Extracted directly from weight gain curves

$$D = \pi\left(\frac{h}{4M_\infty}\right)^2\left(\frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}}\right)^2$$

- ANSYS: Defined *D* separately in coordinate direction (xyz), using Springer formulations. The weight gain curves were then reconstructed using…

$$M(t) = \left(\frac{\Sigma\, Temperature\ at\ each\ node}{Total\ number\ of\ nodes}\right)M_\infty$$

This served to verify that the code was running properly

Continued…

- Model: Volume Strain Formulations
  - Composite properties known from layup (σ_x, θ, ϕ, ρ, E, ν)
- Allows calculation of unstressed D_0 of composite

$$D_0 = D_{z0}\left(\frac{h}{l}\sqrt{\frac{D_{x0}}{D_{z0}}} + \frac{h}{w}\sqrt{\frac{D_{y0}}{D_{z0}}} + 1\right)^2$$

- Stressed diffusivity D_σ is then found…

$$\ln\frac{D_\sigma}{D_0} = \frac{a}{\phi_m}\frac{(\Delta V/V_0)_m}{v_{f0}\left[v_{f0} + (\Delta V/V_0)_m\right]}$$

Diffusivity

		σ_x	D (mm^2/hour) * 10^{-2}			Percent Error (%)	
θ(deg)	ϕ_f	(MPa)	Experimental	ANSYS	Model	ANSYS	Model
0	0.52	0	0.1073	0.1046	0.1076	-2.52	0.28
		18	0.1156	0.1118	0.1075	-3.29	-7.01
		30	0.112	0.1132	0.1074	1.07	-4.11
20	0.52	0	0.125	0.1197	0.1134	-4.24	-9.28
		18	0.1374	0.1296	0.1366	-5.68	-0.58
		30	0.1813	0.1619	0.1559	-10.70	-14.01
45	0.52	0	0.1237	0.1187	0.1211	-4.04	-2.10
		18	0.1444	0.1429	0.1482	-1.04	2.63
		30	0.1911	0.1691	0.1743	-11.51	-8.79
90	0.52	0	0.1195	0.1151	0.1177	-3.68	-1.51
		18	0.1705	0.1631	0.1699	-4.34	-0.35
		30	0.2132	0.1977	0.1987	-7.27	-6.80

Observations

- All 0-degree samples, regardless of tensile loading, exhibit similar M_∞ and D values
- Magnitude at which the diffusion parameters change increases with fiber angle ($\theta = 0° \rightarrow \theta = 90°$)
  - This is due to larger volume strain in the matrix at $\theta = 90°$
- In general, the model over-estimates M_∞ values and under-estimates D

Conclusions

- The model successfully predicts maximum moisture content and diffusivity values for stressed unidirectional composite samples.
- The model uses commonly known composite input parameters (σ_x, θ, ϕ, ρ, E, ν) in addition to neat resin properties D and M_∞
- ANSYS FEA code has shown very good agreement with experimental data, validates thermal-moisture diffusion analogy

CHARACTERIZATION OF THE EFFECTS OF HYGROTHERMAL-AGING ON MECHANICAL PERFORMANCE AND DAMAGE PROGRESSION OF FIBERGLASS EPOXY COMPOSITE

Hygrothermal Aging: Degradation Mechanisms

Physical degradation:

- Moisture induced swelling alters the internal stress state of the composite causing damage or altering the micromechanical damage behavior

Chemical degradation

- Water alters the microstructure of the polymer or interface
  - Plasticization
  - Hydrolysis
  - Secondary crosslinking (epoxy)

Acoustic Emission (AE)

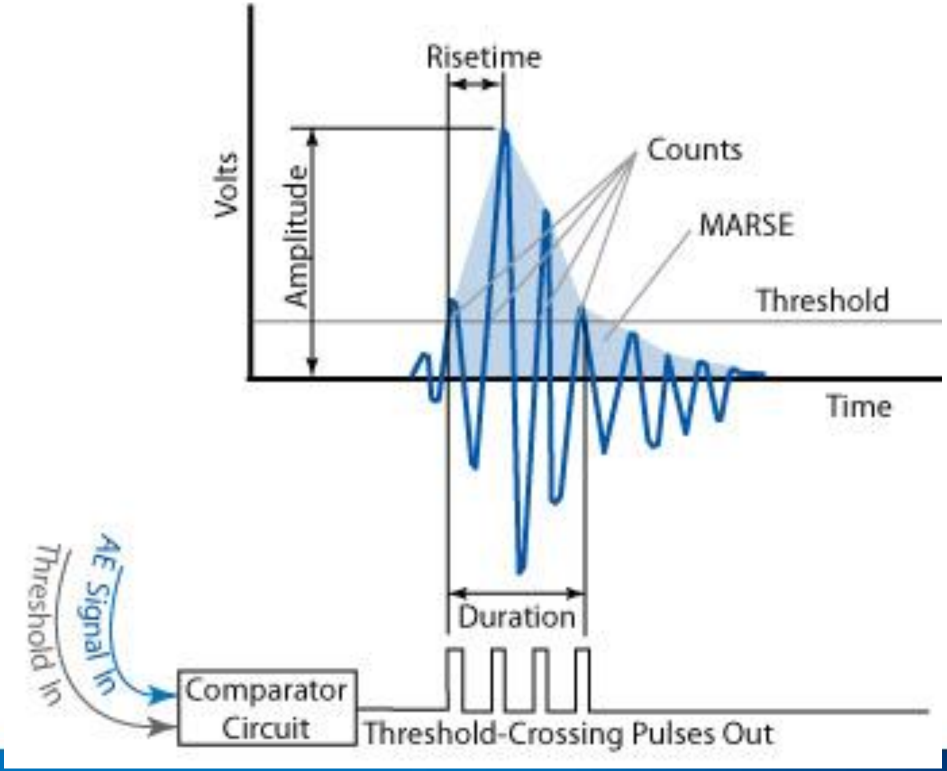
AE monitoring

- As composite materials are loaded, damage occurs within the material.
- Each damage event causes a release of strain energy resulting in a stress wave
- Piezoelectric transducers mounted in various locations on the surface of the test specimen record time-amplitude for these stress waves
- The AE DAQ records a waveform for ***every*** measurable damage event that occurs (can be thousands).

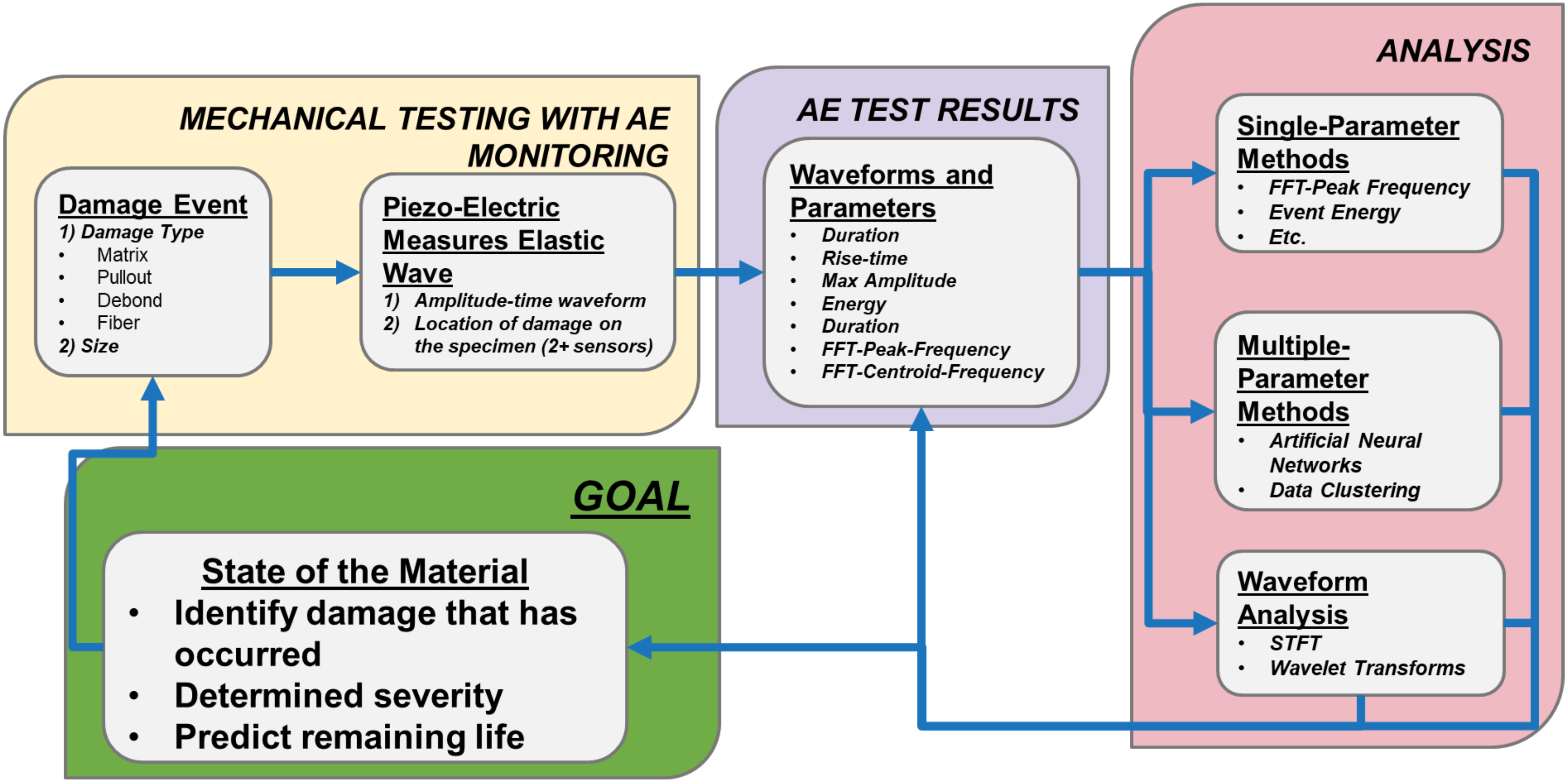
Waveform Parameters

Basic parameters are extracted from an AE event waveforms and serve as descriptors used in AE analysis

- Energy
- FFT-Peak-Frequency
- Max Amplitude
- FFT-Centroid-Frequency
- Duration
- Rise-time
- etc.



AE Analysis Techniques



MECHANICAL TESTING WITH AE MONITORING

Damage Event

1) Damage Type
- Matrix
- Pullout
- Debond
- Fiber

2) Size

Piezo-Electric Measures Elastic Wave

1) *Amplitude-time waveform*
2) *Location of damage on the specimen (2+ sensors)*

AE TEST RESULTS

Waveforms and Parameters

- *Duration*
- *Rise-time*
- *Max Amplitude*
- *Energy*
- *Duration*
- *FFT-Peak-Frequency*
- *FFT-Centroid-Frequency*

ANALYSIS

Single-Parameter Methods

- *FFT-Peak Frequency*
- *Event Energy*
- *Etc.*

Multiple-Parameter Methods

- *Artificial Neural Networks*
- *Data Clustering*

Waveform Analysis

- *STFT*
- *Wavelet Transforms*

GOAL

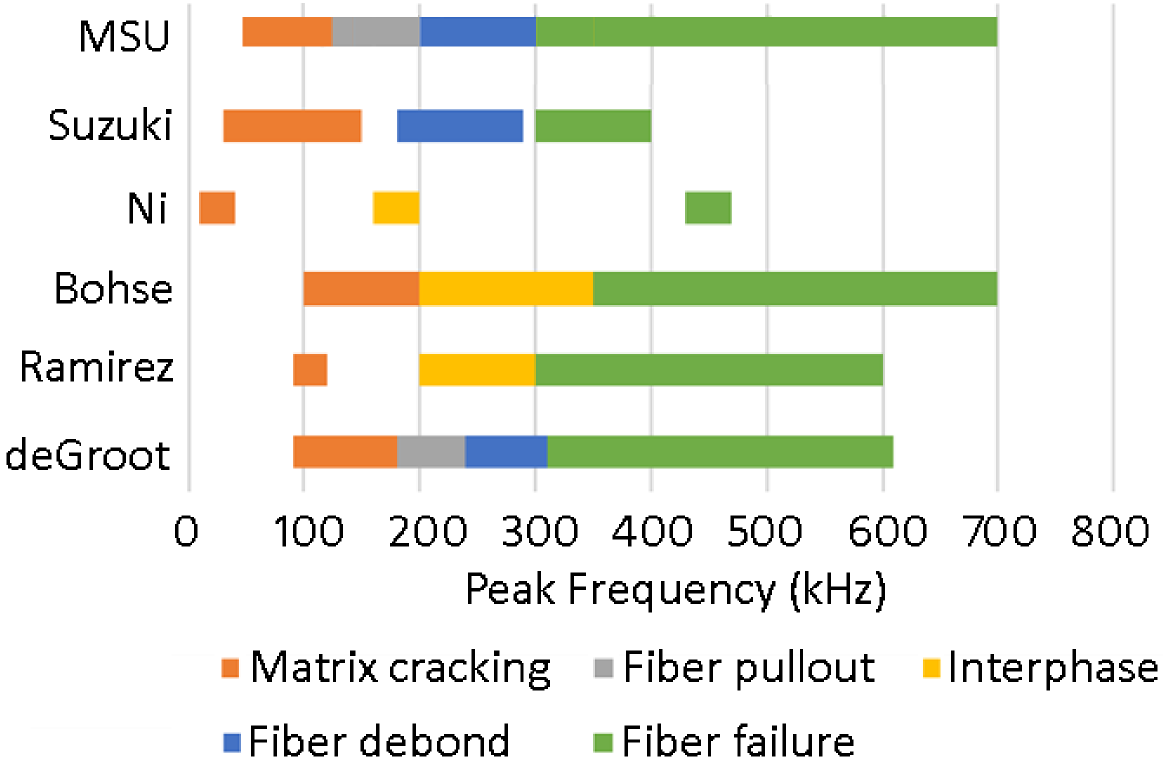
State of the Material

- **Identify damage that has occurred**
- **Determined severity**
- **Predict remaining life**

Single Parameter Analysis

Single parameters may be used to characterize damage behavior in the composite.

- Number of events
- Signal energy
- Frequency: Damage Mechanisms
  - Frequencies correlate to damage mechanisms



AE and Hygrothermal Aging

AE monitoring is NDE technique that could aid in understanding hygrothermal affects on damage behavior.

- AE is an indirect measure of damage
- How is AE response affected by hygrothermal aging?
  - Changes in damage behavior
  - Changes in Lamb wave behavior

Methods and Results Outline

- Matrix Characterization
  - Thermal analysis
  - Diffusion and swelling
- Composite Characterization
  - Diffusion and swelling
  - Hygrothermal damage evaluation
  - Mechanical testing and characterization
  - Damage progression characterization: constitutive stress-strain response and AE monitoring
- Wave Propagation and Attenuation
  - Guided ultra-sonic testing

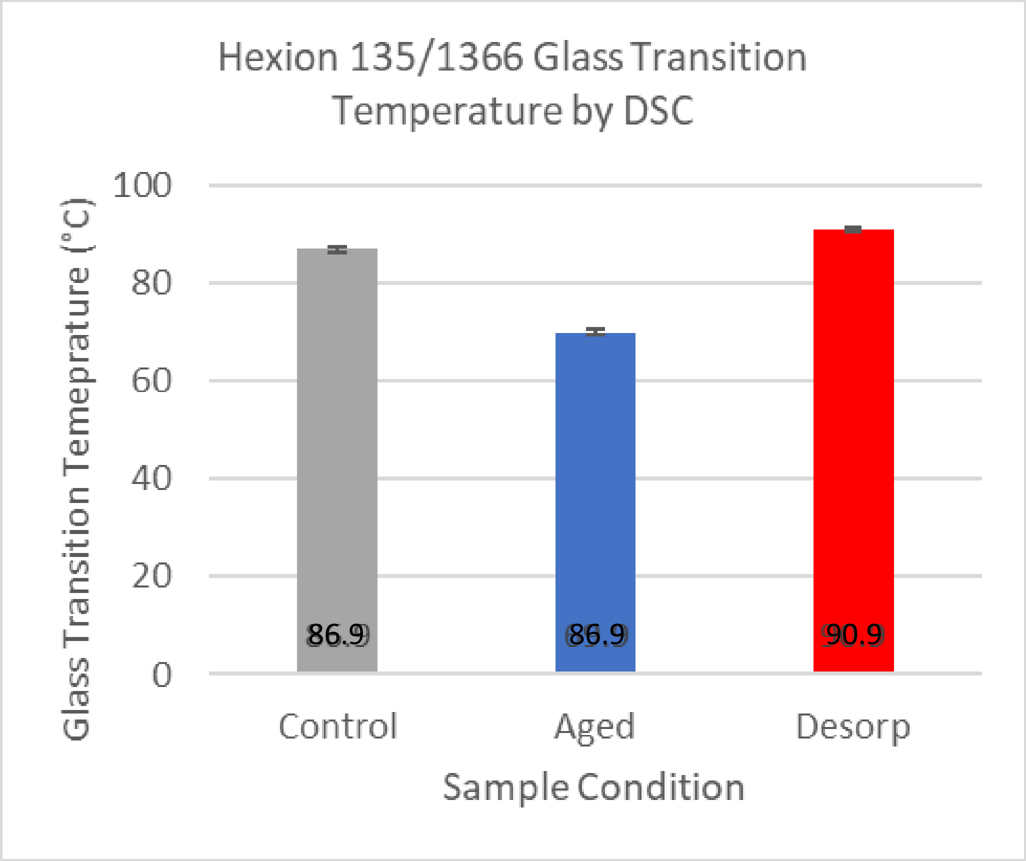
Matrix Characterization: Thermal Analysis Methods

DSC Test matrix

Sample Type	Conditioning	Number of Samples	Tested bulk moisture content (%)
Control	none	5	0.0%
Aged	312 hrs. 50°C distilled water	5	4.0%
Desorb	1) 312 hrs. 50°C distilled water 2) dried 620 hrs. 50°C	5	0.1%

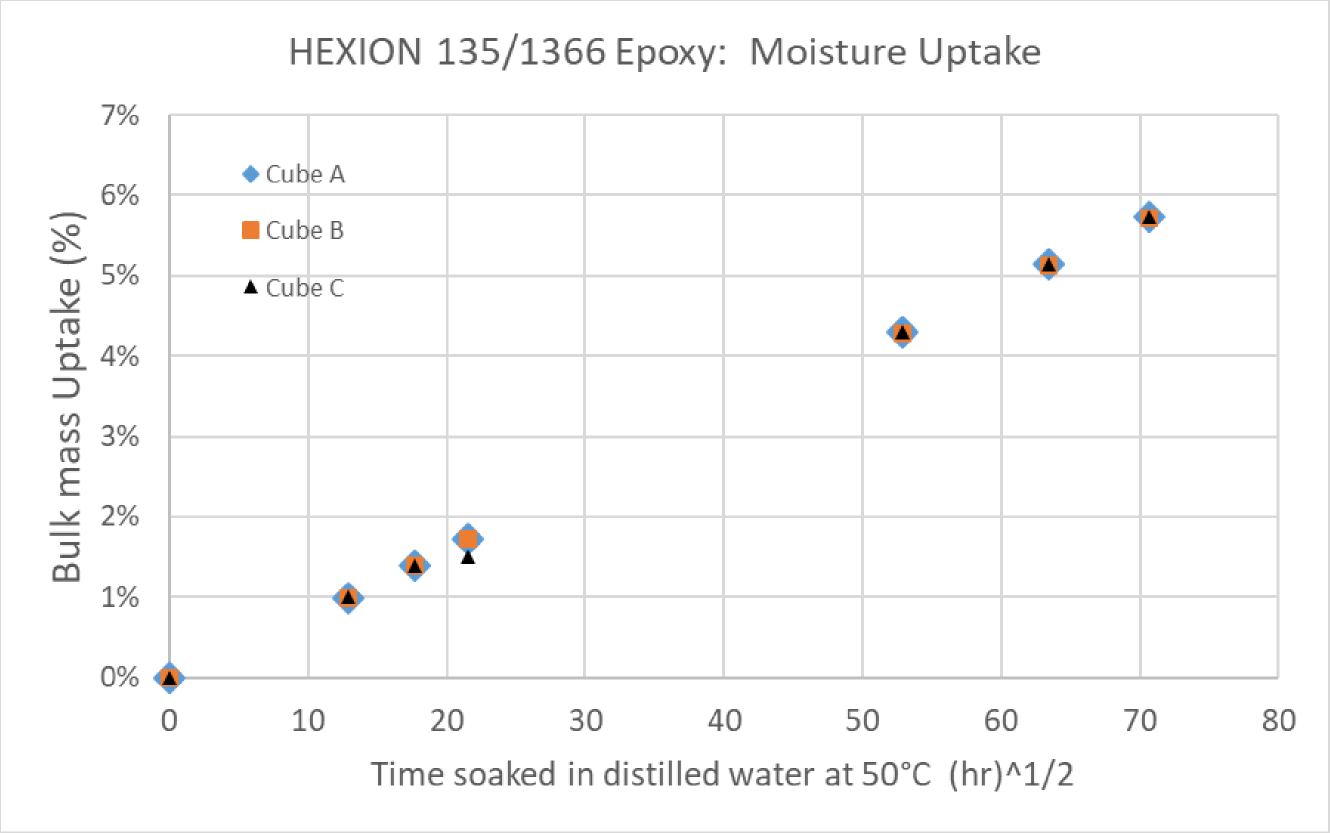
Matrix Characterization: Thermal Analysis

- T_g was reduced from hygrothermal aging by 17°C which suggests that plasticization is present
- Nearly all moisture was expelled during the drying/desorbing process
- T_g is fully recovered after desorption/drying



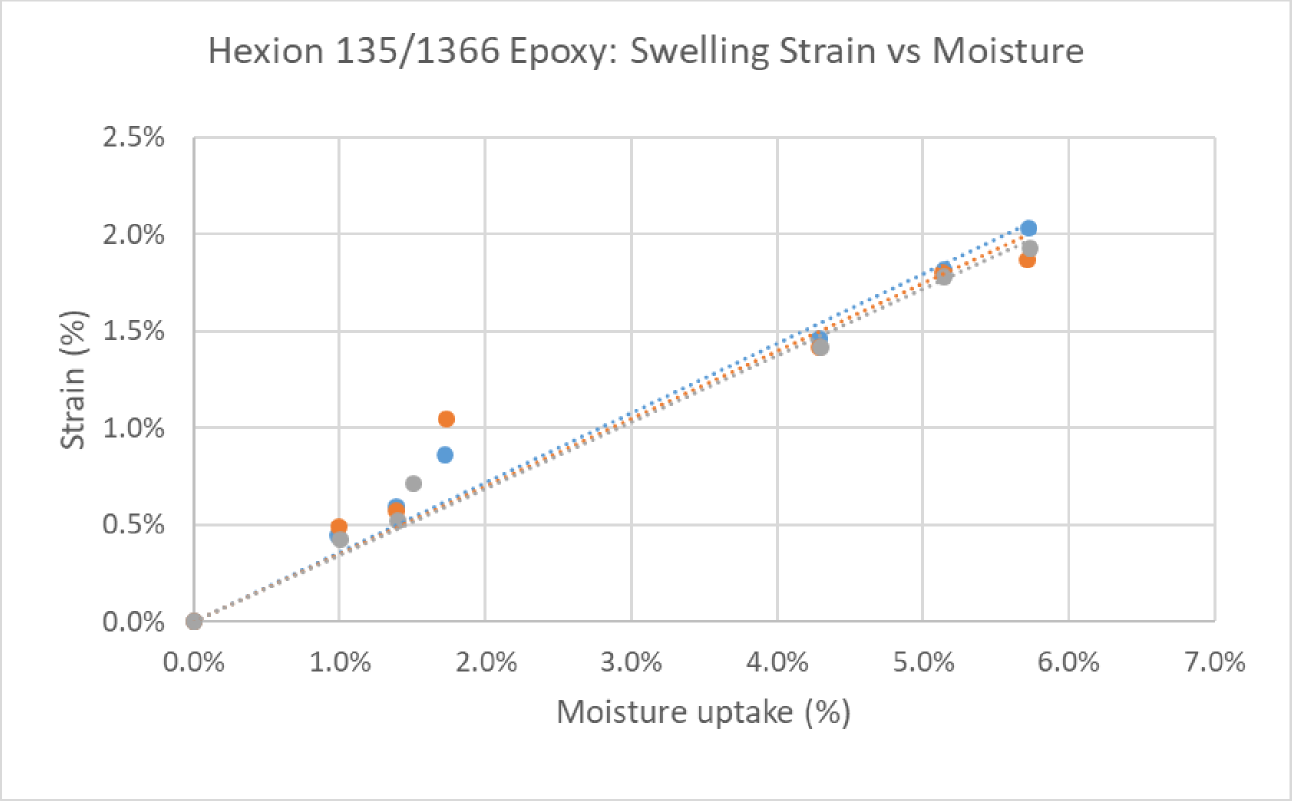
Matrix Characterization: Diffusion and Swelling Results

- Fickian behavior
  - Linear with $\sqrt{t}$
- Moisture uptake 5.7%+ and increasing
  - Typical uptake for epoxy: 2-7%

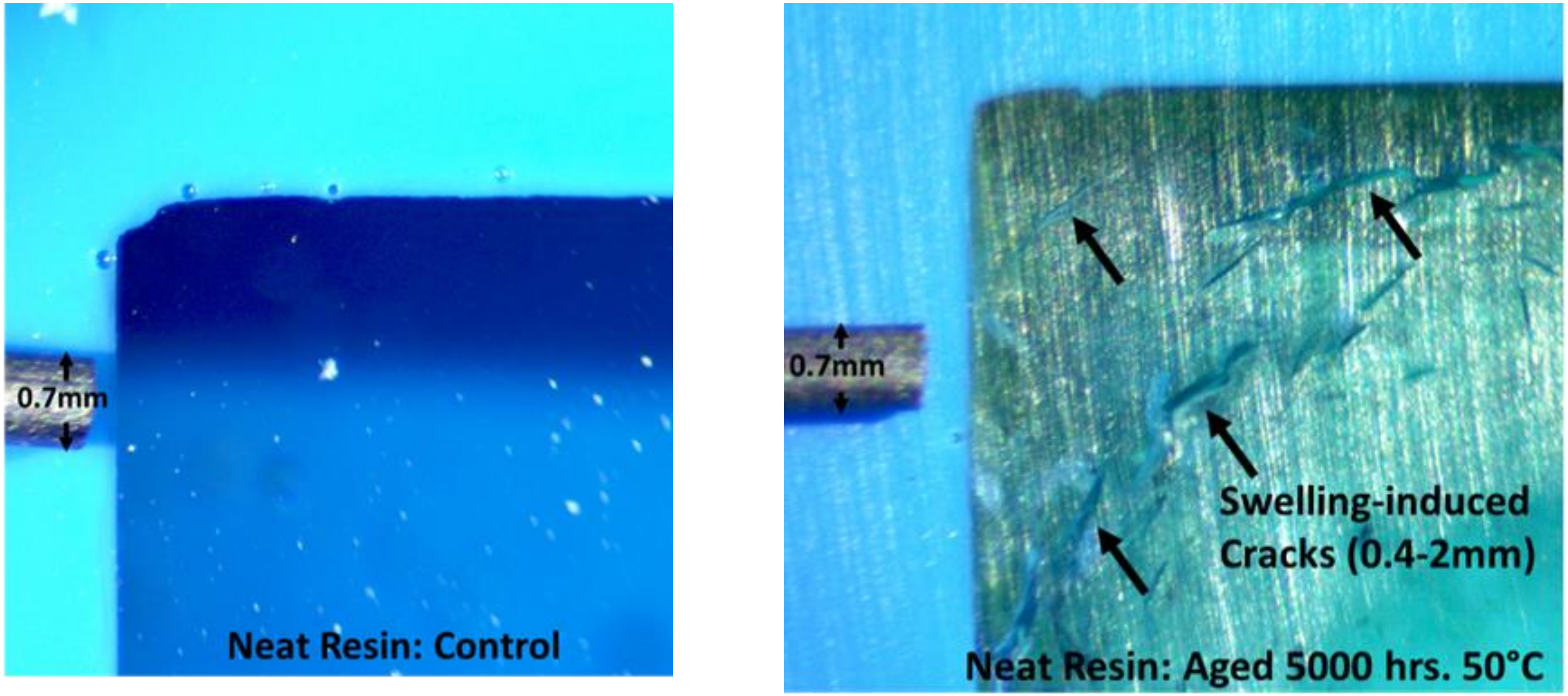


Matrix Characterization: Diffusion and Swelling Results

- Swelling strains were significant ~2%ε at 5.7% bulk moisture uptake
- Matrix Swelling coefficient
  - →
    ε
  - β_m = 0.35 (%ε / %m)



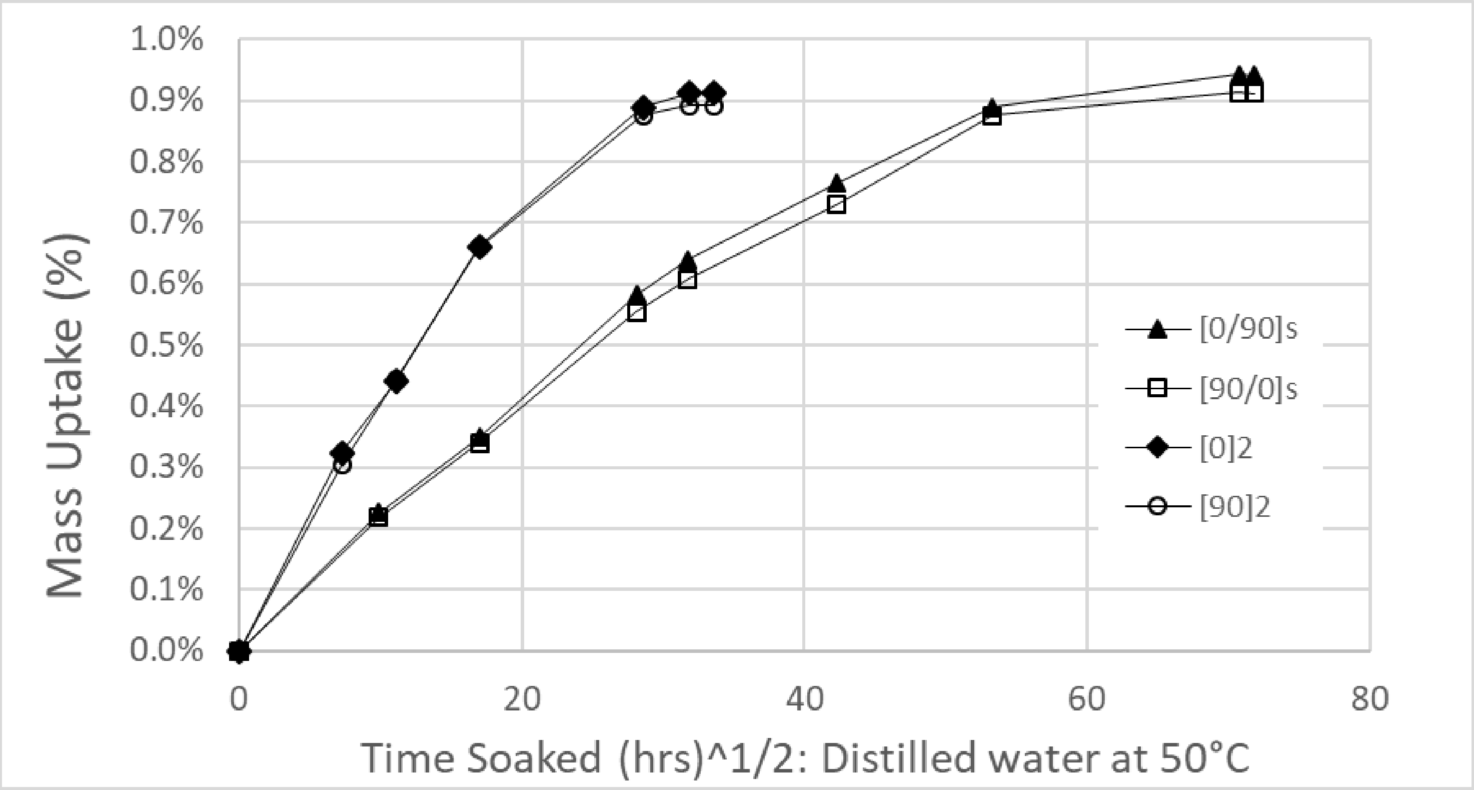
Matrix Characterization: Diffusion and Swelling Results



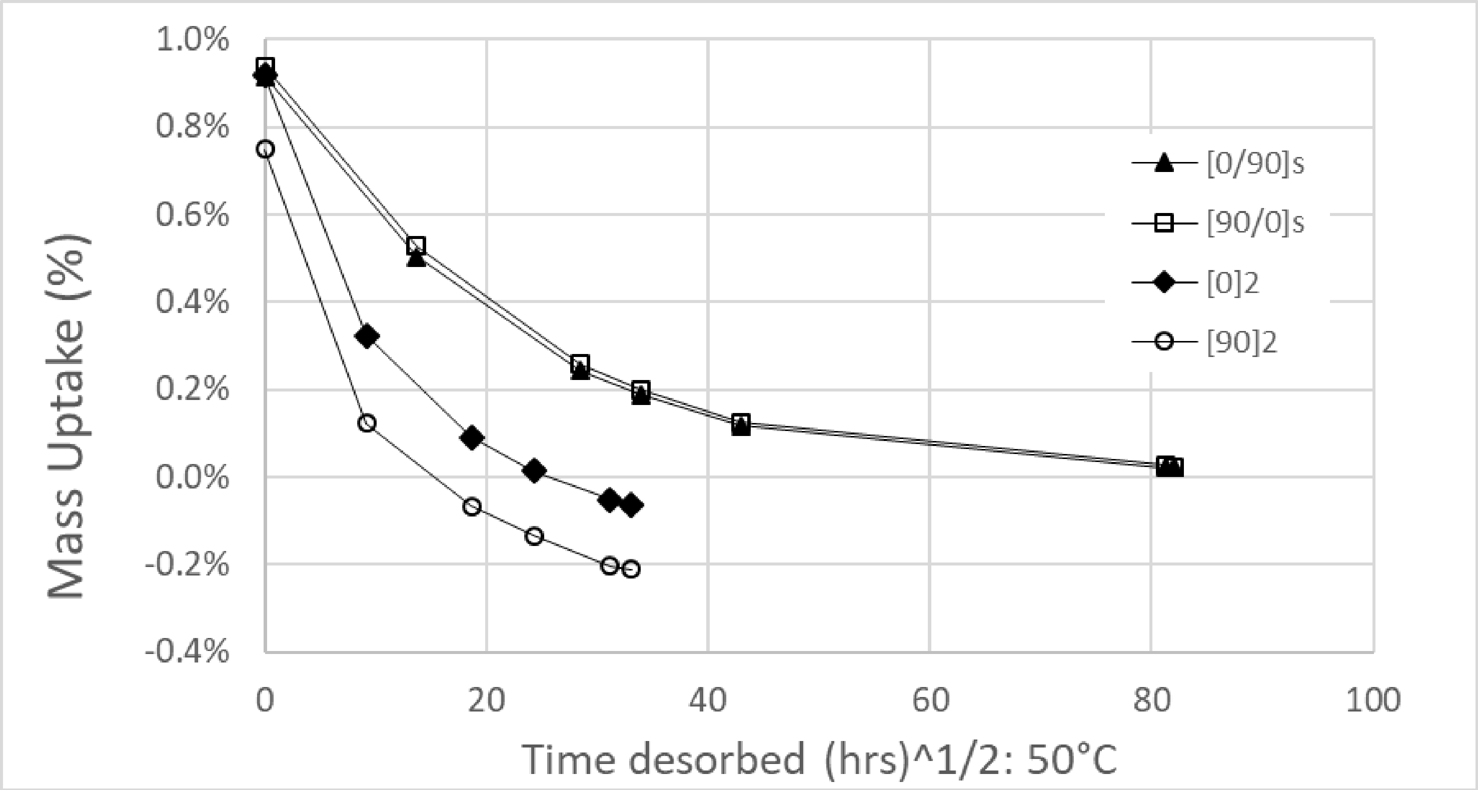
Swelling strains resulted in damage

Composite Characterization: Moisture Uptake Results

- Moisture uptake 0.9% by mass
- *In situ* matrix absorption (ROM): 2.7%

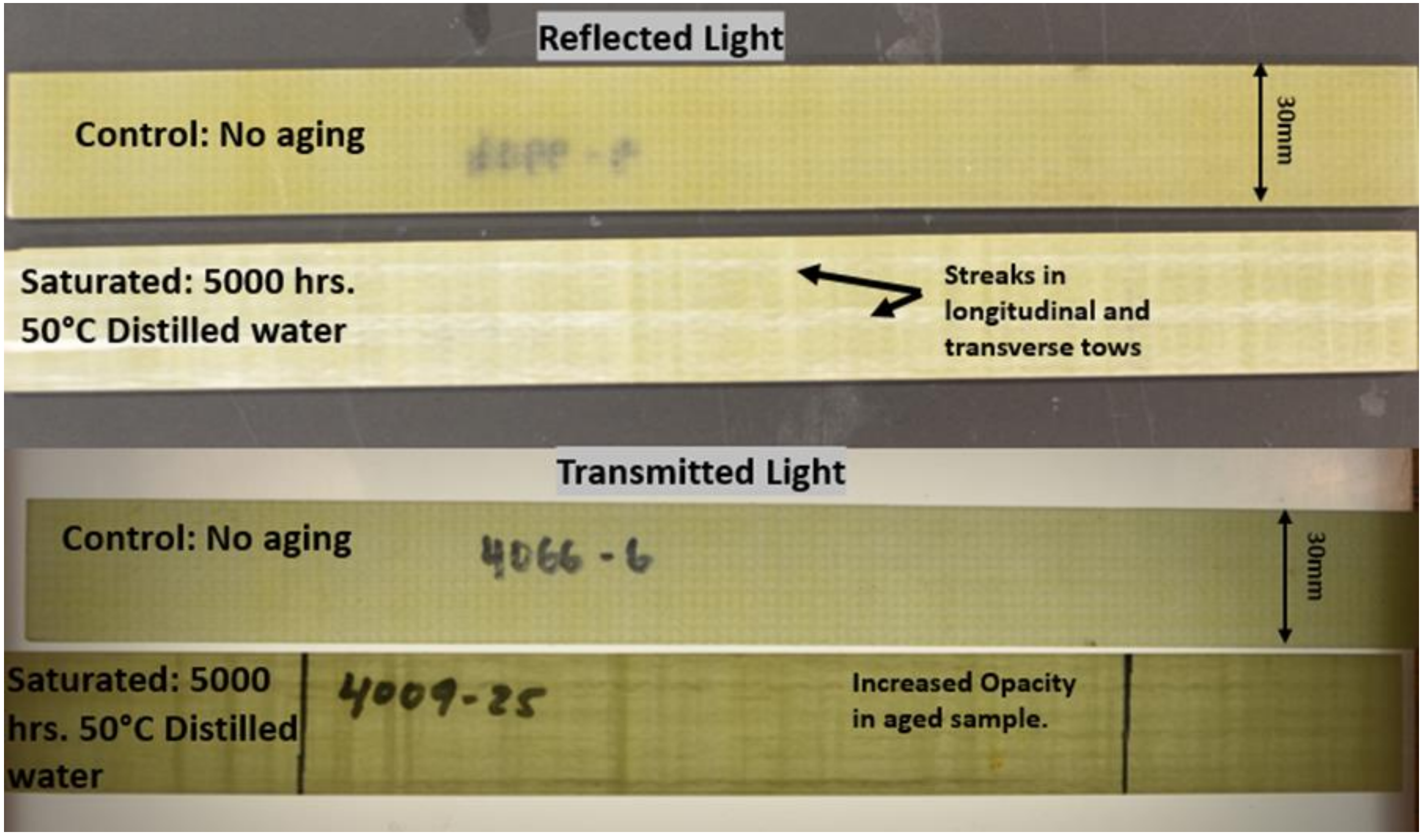


Composite Characterization: Moisture Desorption Results



Dan Samborsky: Summary of Vectorply E-LT 3800 Fabric Properties

Composite Characterization: Hygrothermal Damage



Reflected Light

Control: No aging

Saturated: 5000 hrs. 50°C Distilled water

Streaks in longitudinal and transverse tows

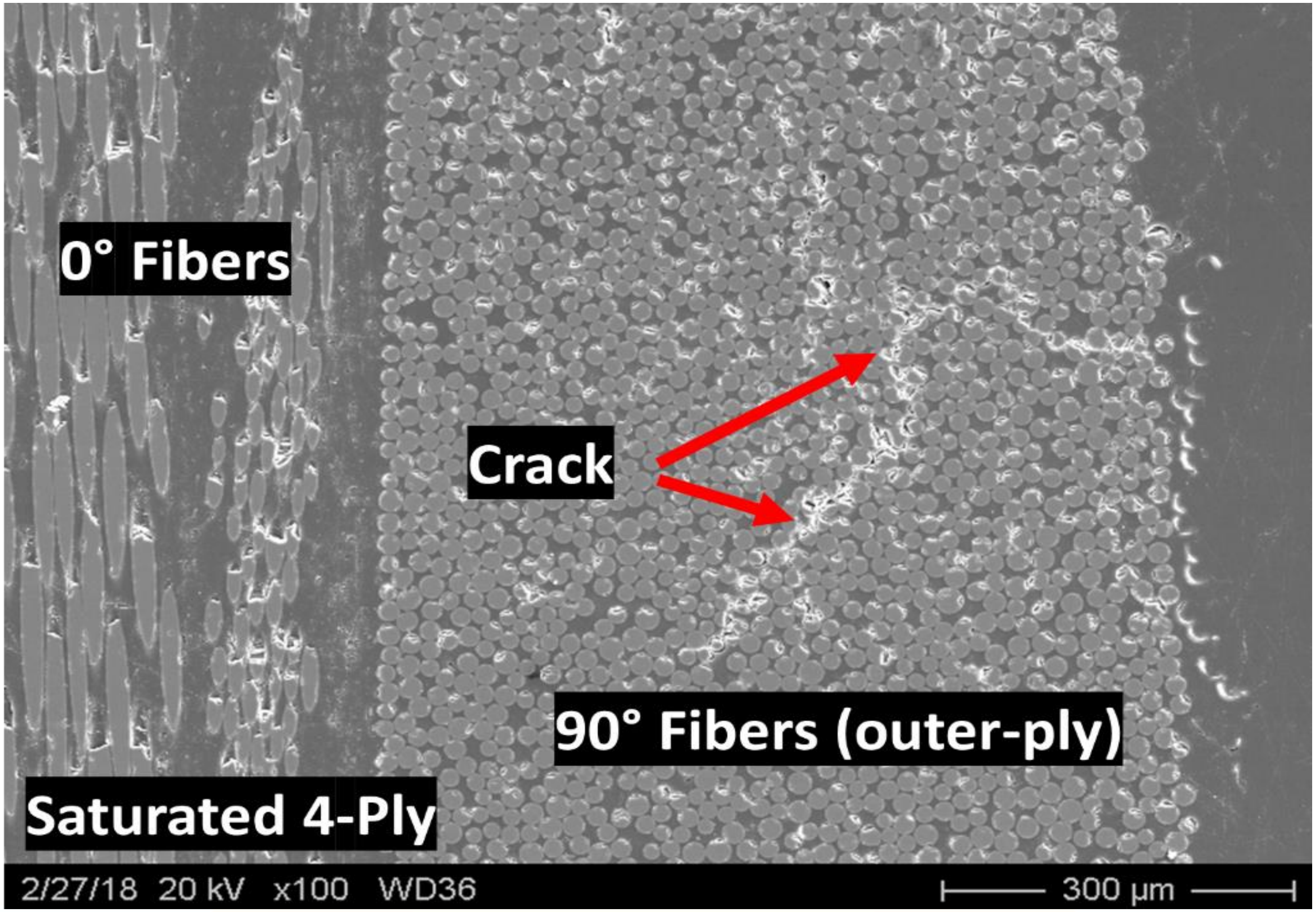
Transmitted Light

Control: No aging

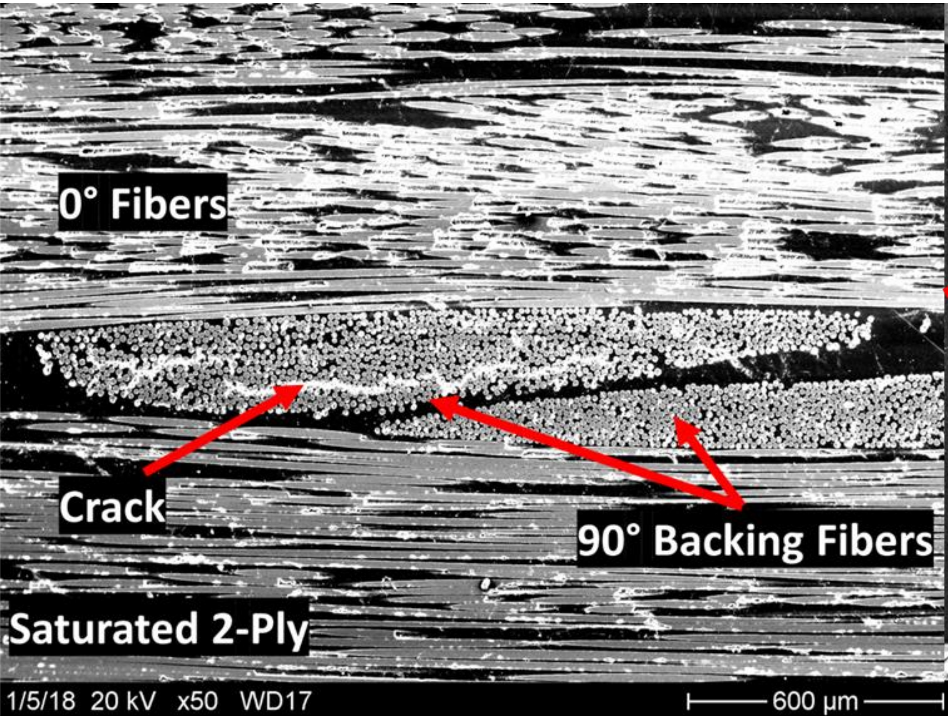
Saturated: 5000 hrs. 50°C Distilled water

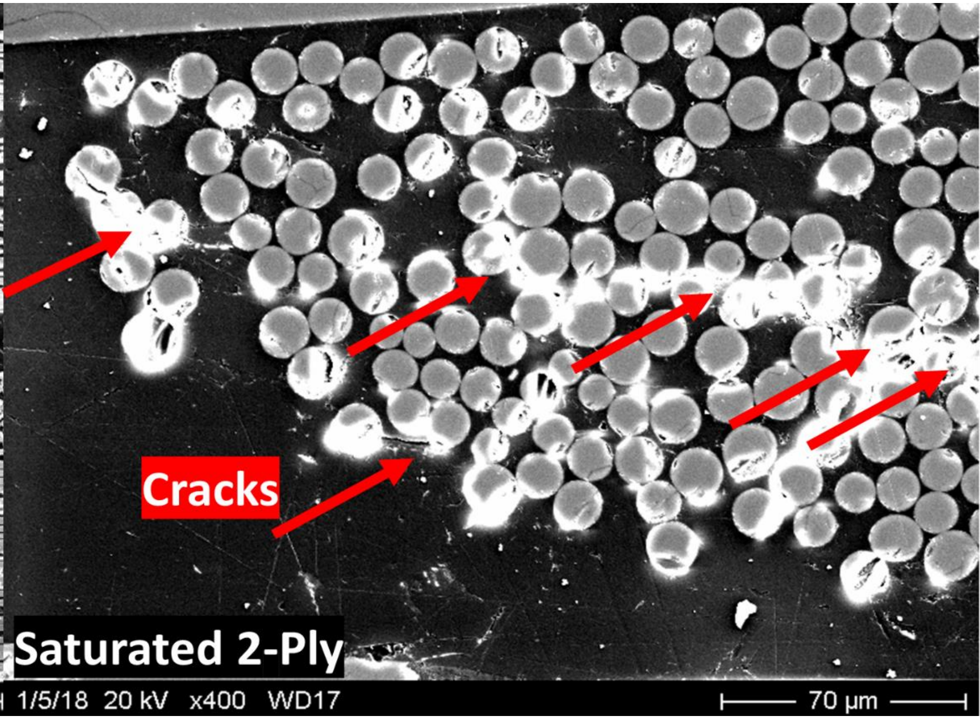
Increased Opacity in aged sample.

Composite Characterization: Hygrothermal Damage



Composite Characterization: Hygrothermal Damage





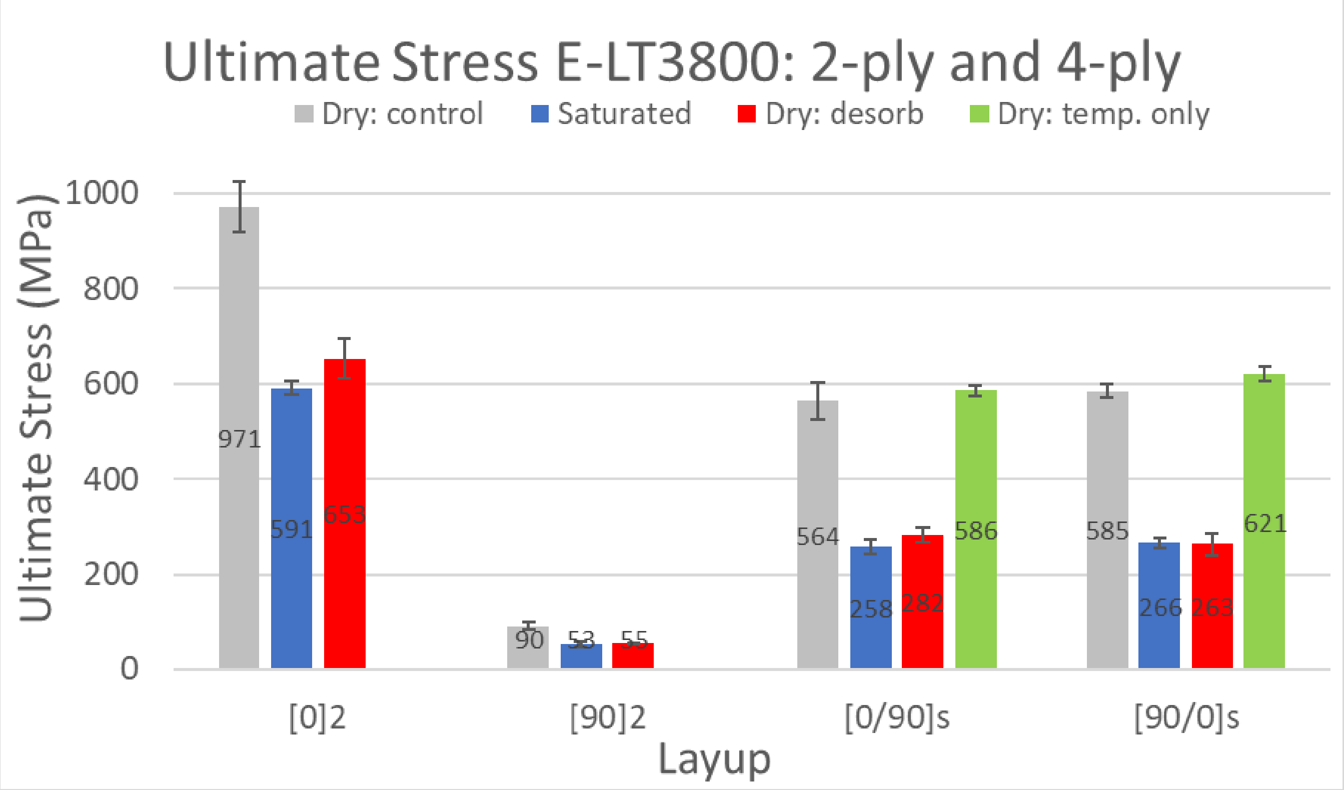
Composite Characterization: Mechanical Properties Results- Strength

Unidirectional
- 40% strength reduction

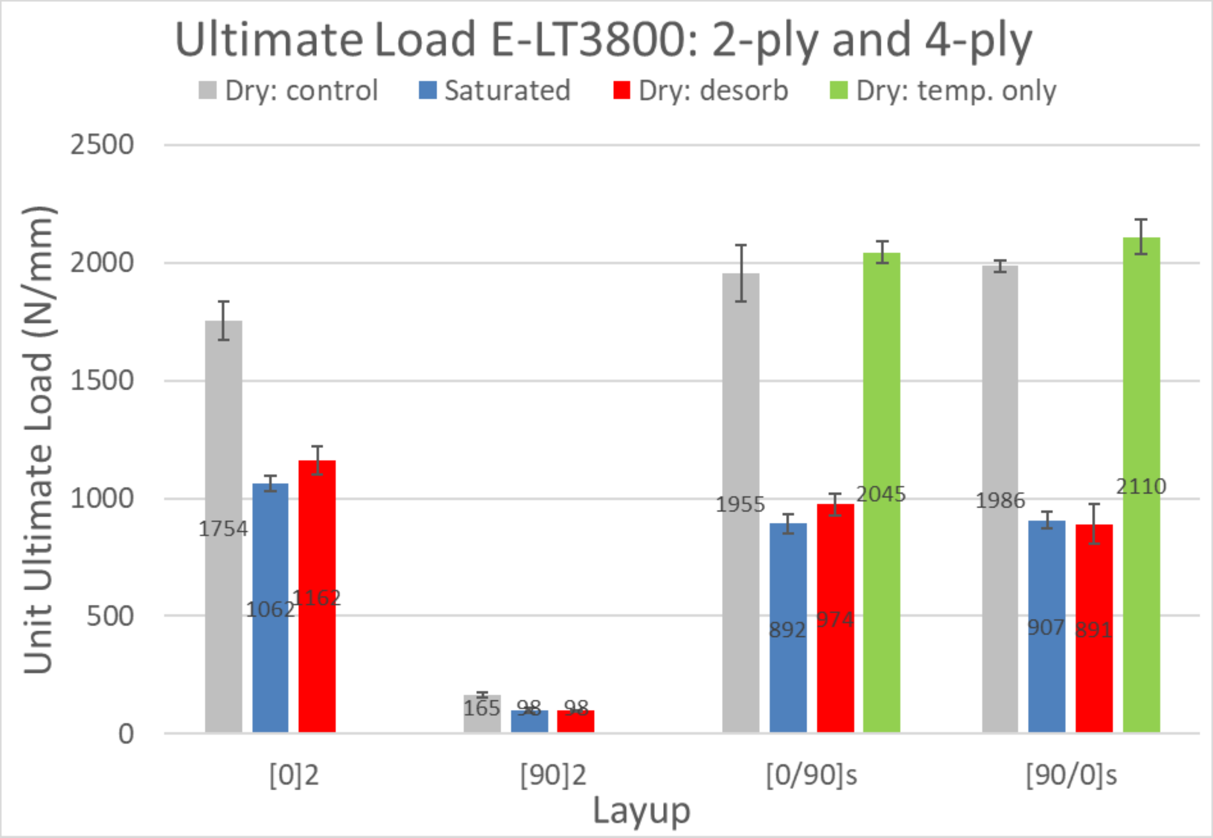
Cross-ply
- 54% strength reduction

Temperature effects
- Strength not affected

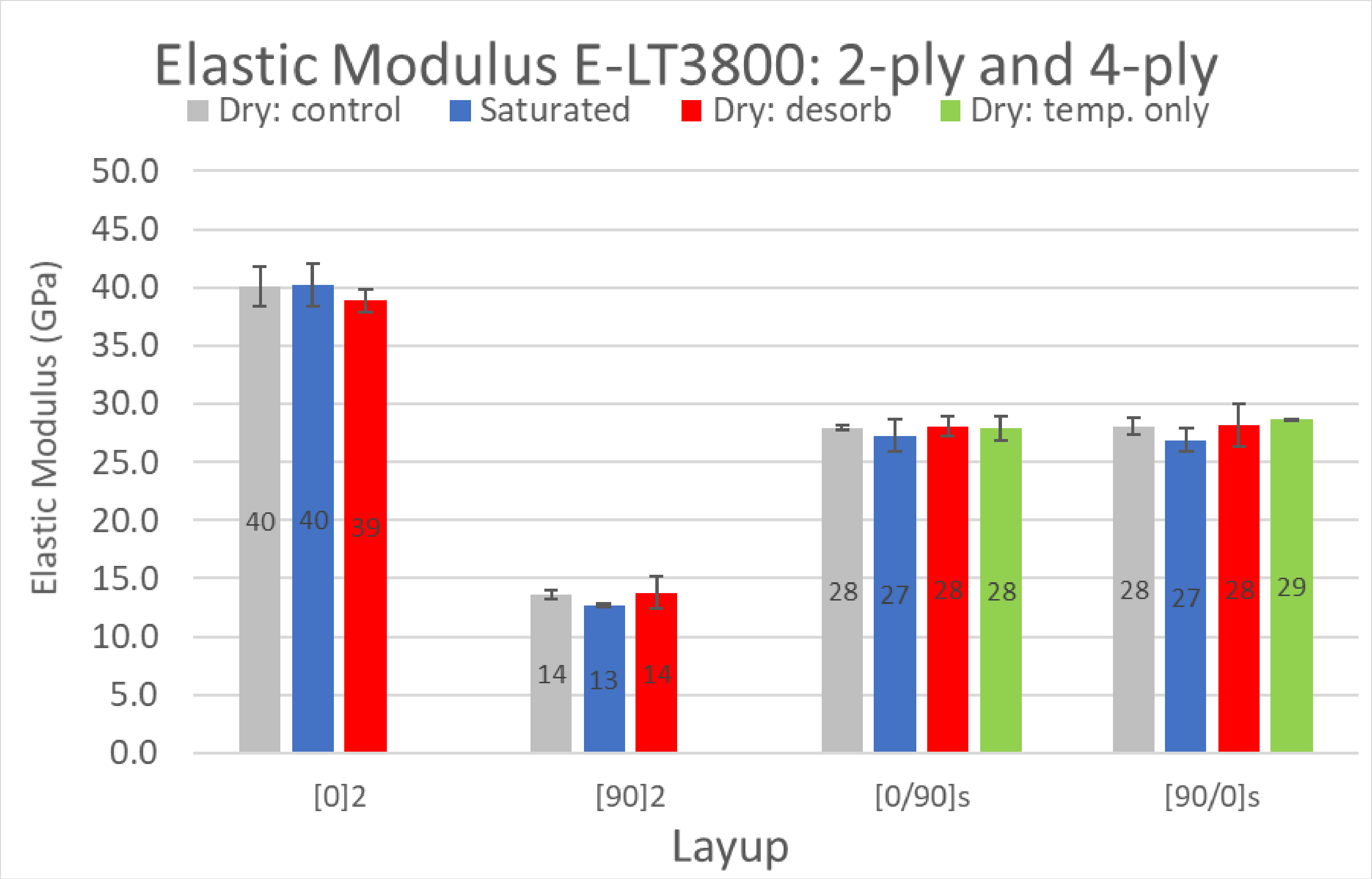
Reversibility
- Strength did not recover with desorbing/drying



Composite Characterization: Mechanical Properties Results



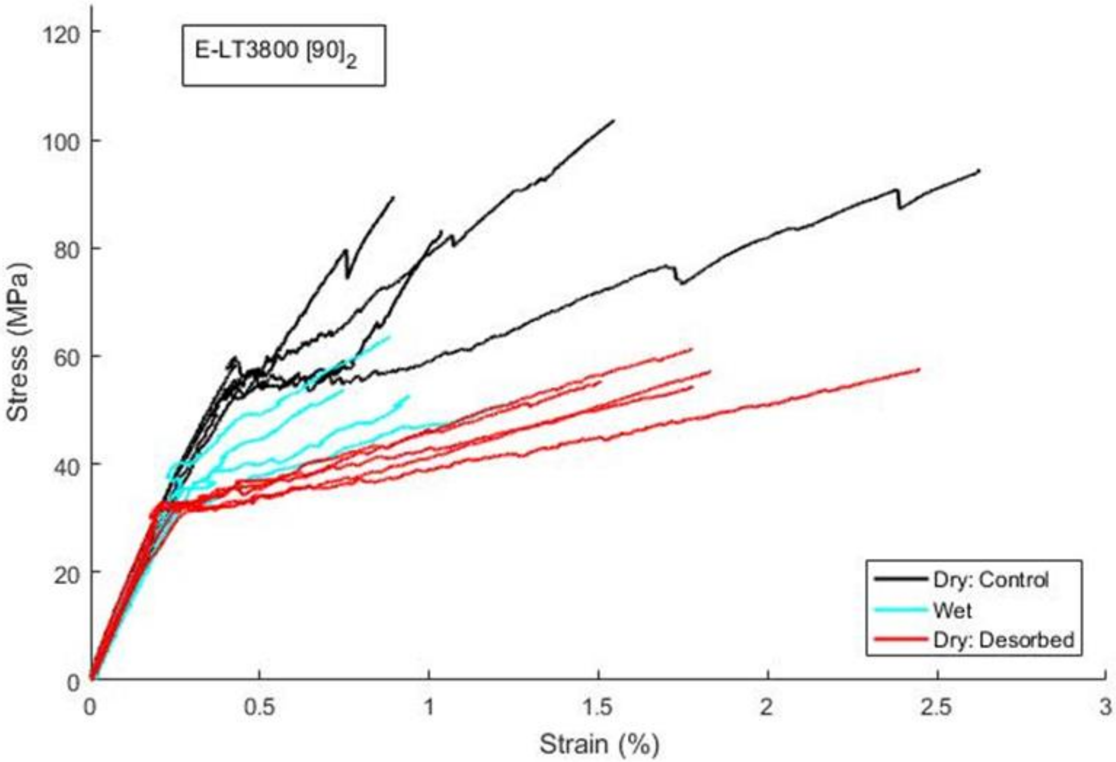
Composite Characterization: Mechanical Properties Results-Modulus



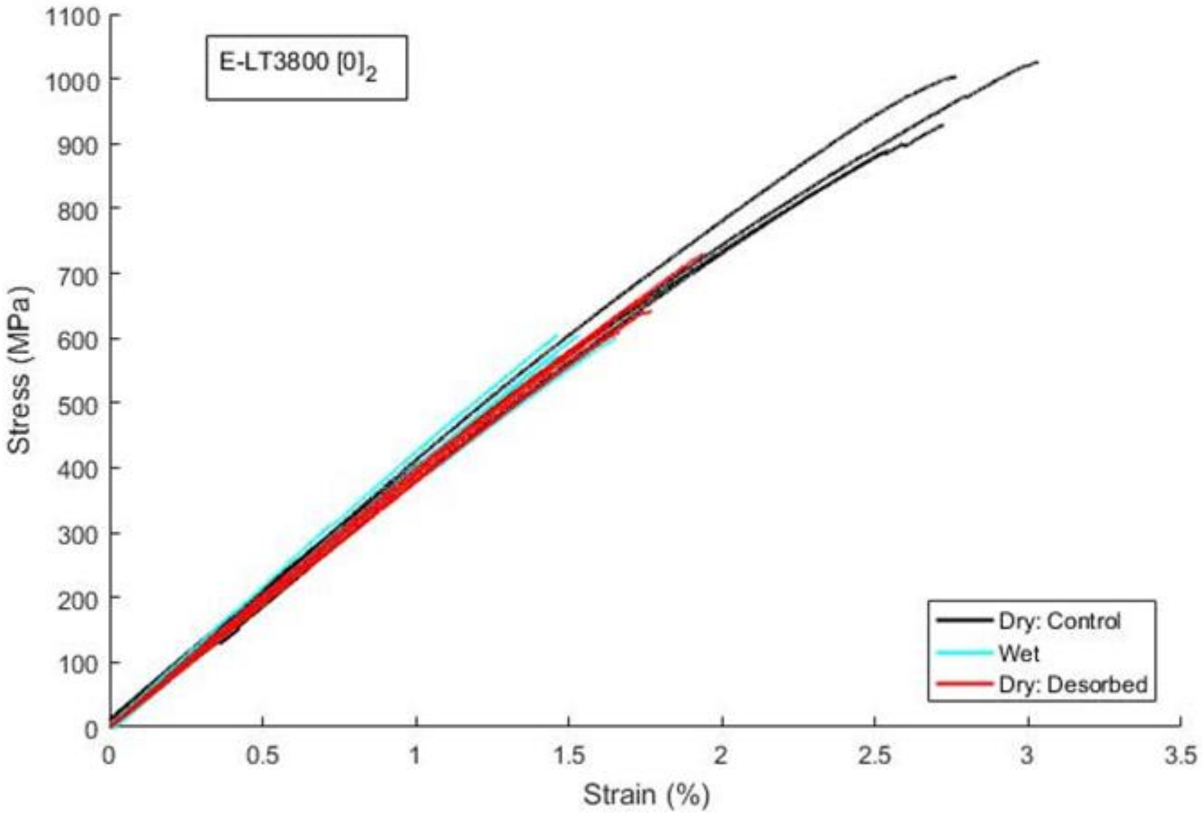
Composite Characterization: Stress-Strain Results

Reduced bi-linear “knee” in conditioned samples

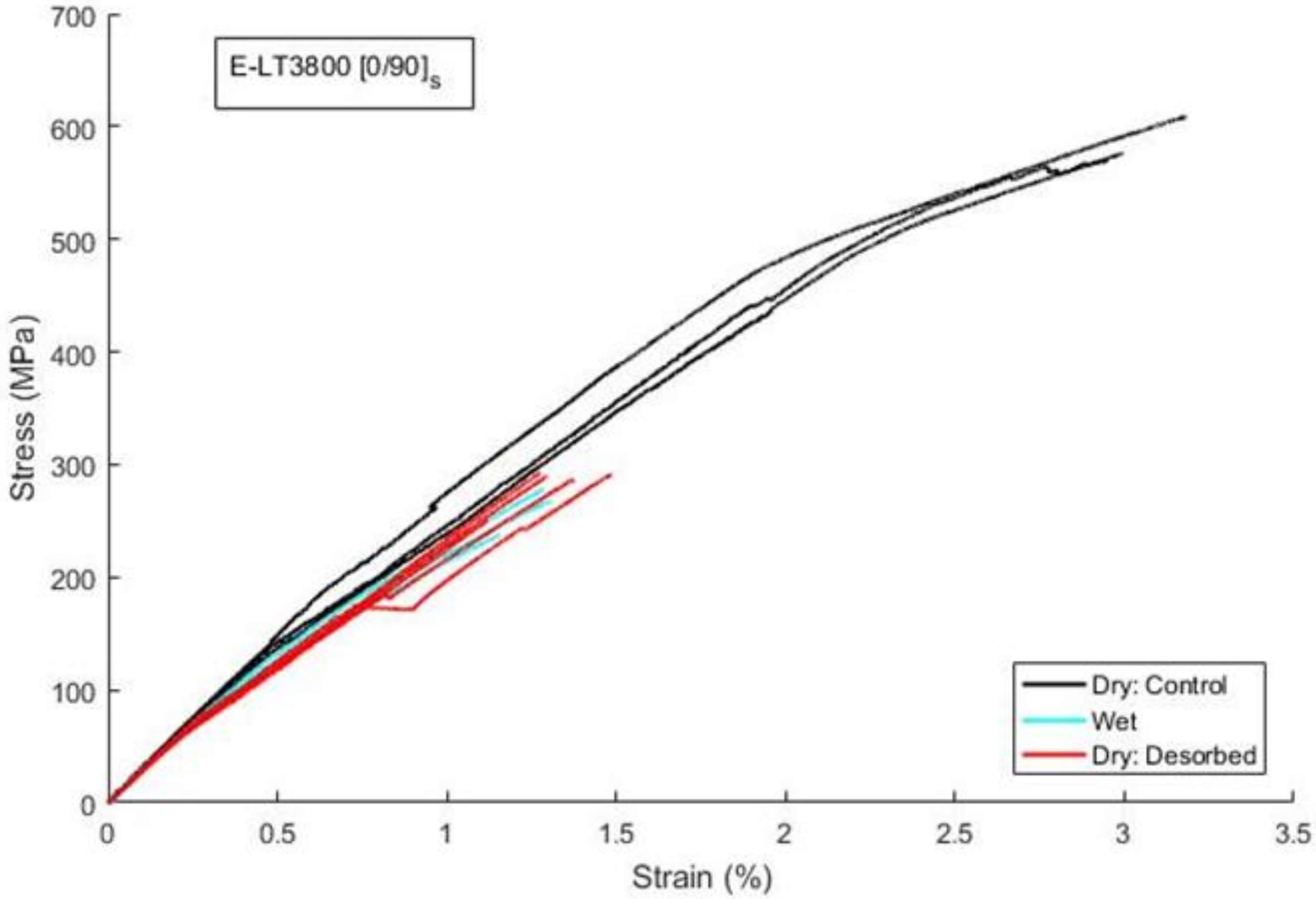
- Marks the onset of transverse failures
- Initiation vs growth



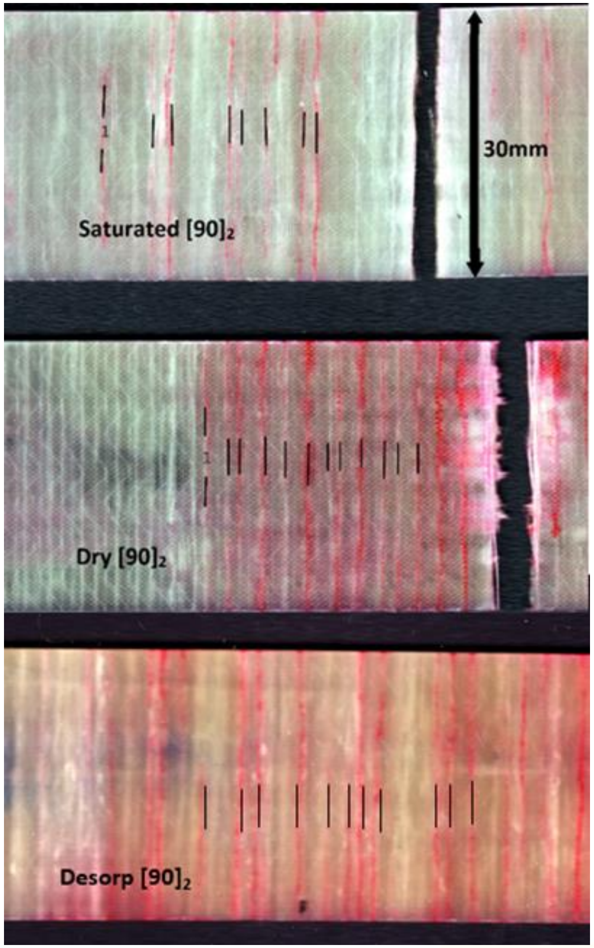
Composite Characterization: Stress-Strain Results

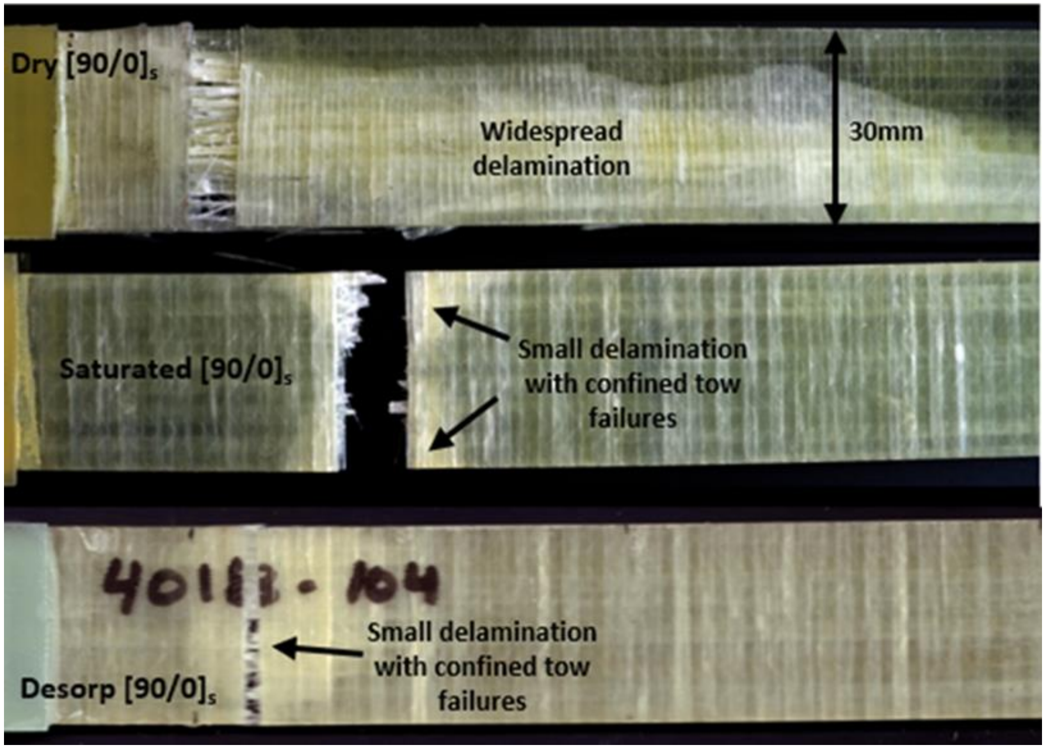


Composite Characterization: Stress-Strain Results

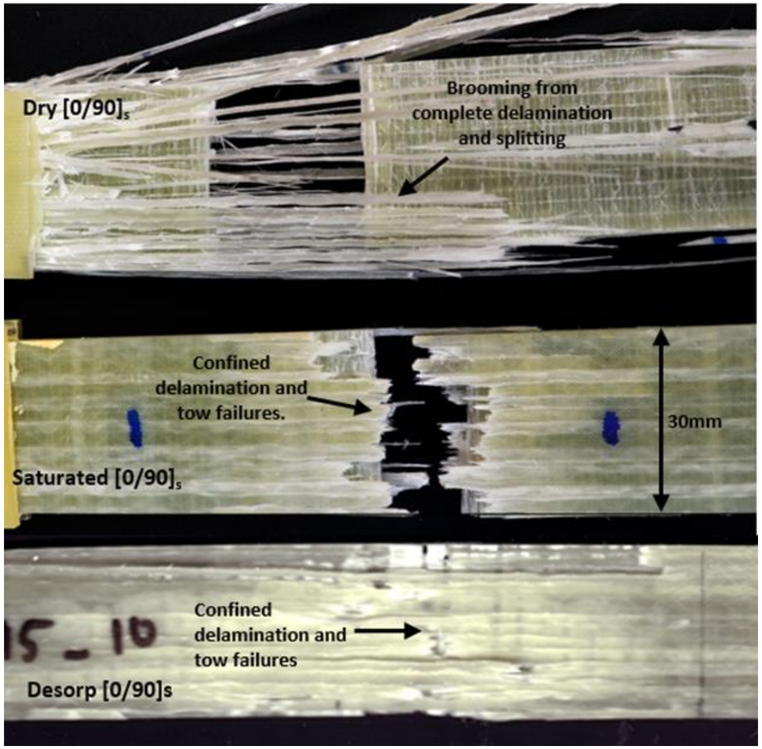


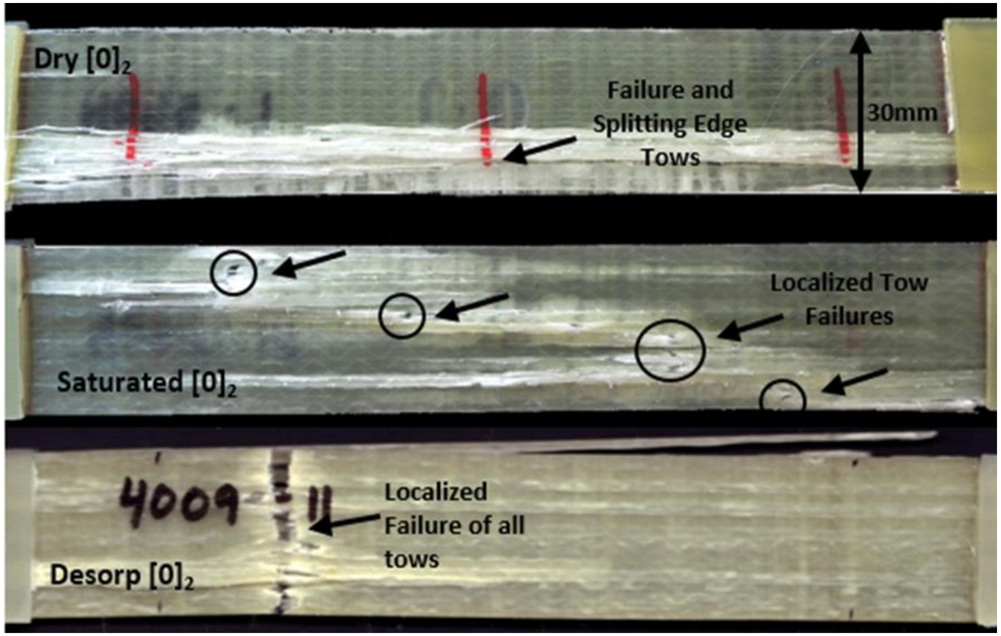
Composite Characterization: Failed Coupon Inspection



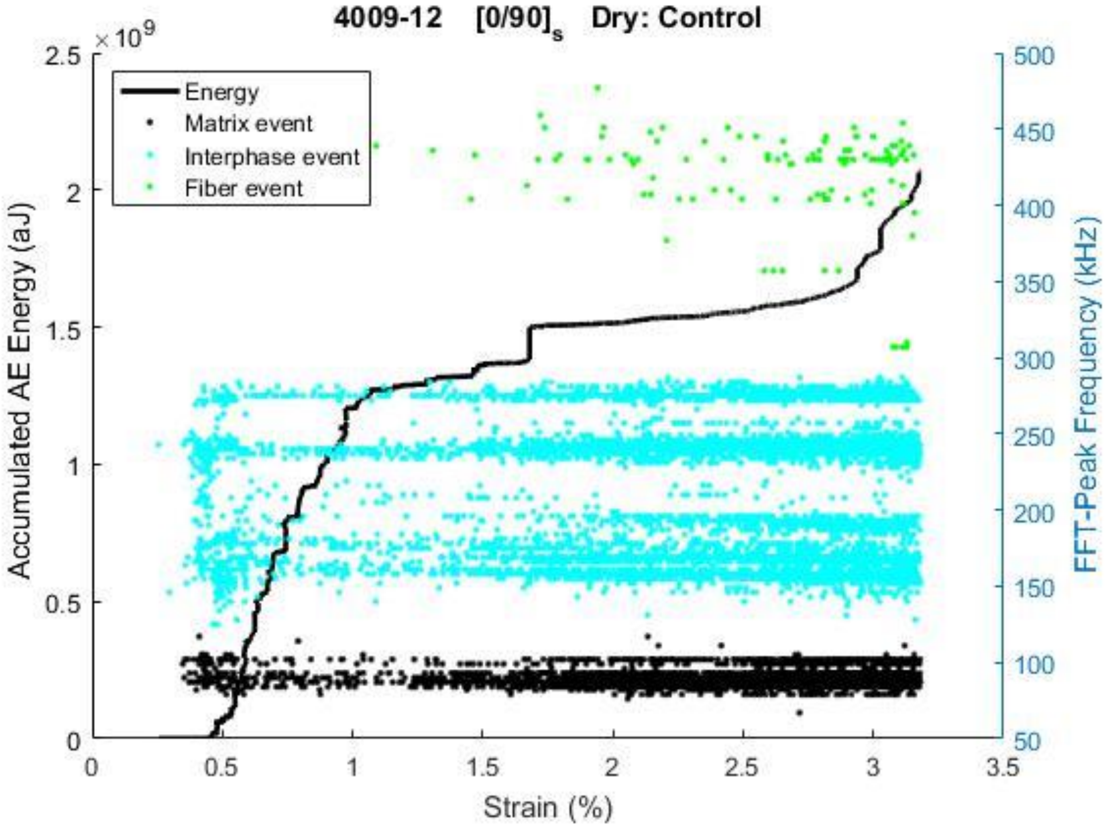


Composite Characterization: Failed Coupon Inspection





Composite Characterization: Acoustic Emission Results



	Damage Mechanism	FFT Peak Frequency Range (kHz)
Bin 1	Matrix	<120
Bin 2	Fiber/matrix interphase	120-300
Bin 3	Fiber	>300

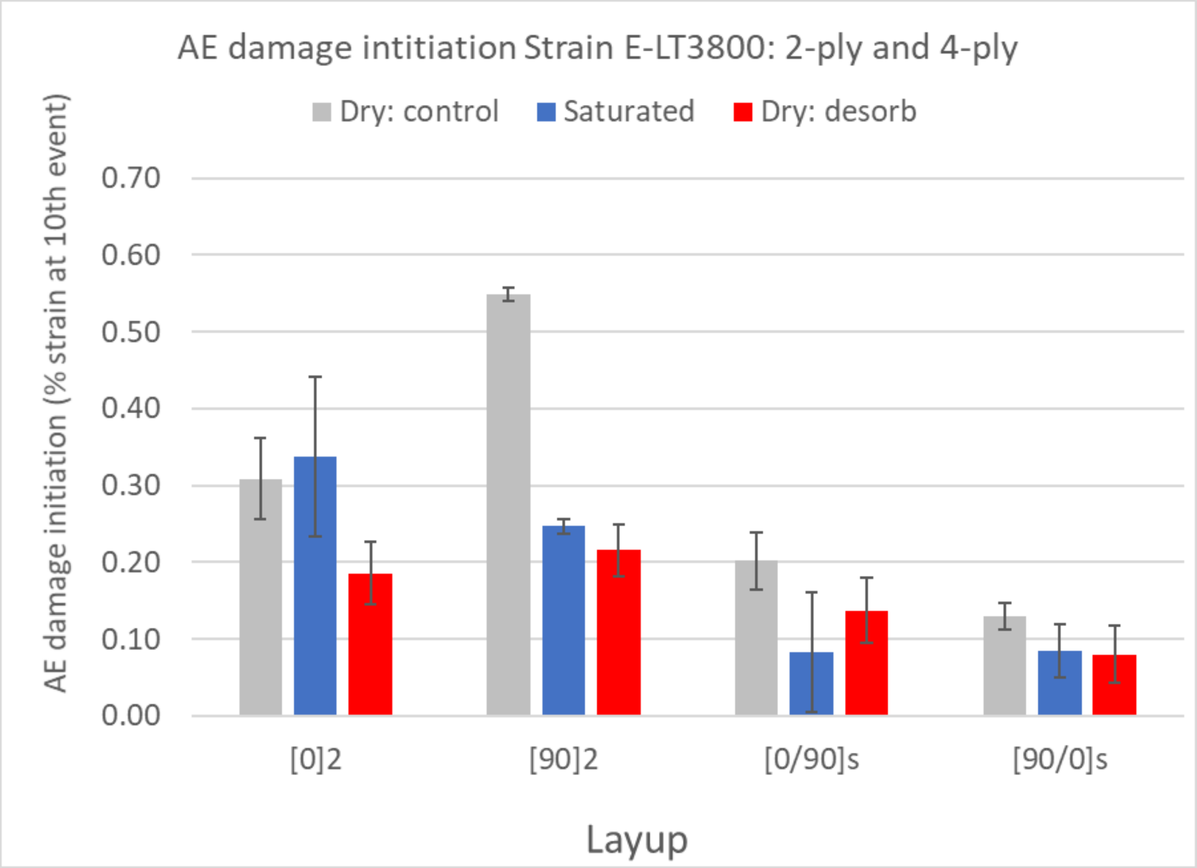
Example of AE energy and frequency results from a static tensile test

- Empirical validation of frequency-damage mechanism correlation is an ongoing work

Composite Characterization: Acoustic Emission Results

Quantify damage onset: Onset of AE activity

- Damage onset was reduced with hygrothermal conditioning
- $[90]_2$ correlates to damage onset in stress-strain response
- Damage onset was obtained for $[0]_2$ laminates



Conclusions

Change in Mechanical Properties

- Strength and damage tolerance was significantly reduced with hygrothermal aging: 40-54% reduction in strength.
- Variation in strength reductions between strength of unidirectional and cross-ply laminates suggests inter-ply behavior is affected by hygrothermal aging.

Conclusions Continued

Damage Behavior

- Reduced damage onset with hygrothermal aging
- Reduced damage tolerance

Hygrothermal affects on AE

- Changes in AE behavior relate to changes in damage behavior, not changes in wave propagation behavior.

Effects of Moisture Absorption on Static Strength and Acoustic Emission Signatures of Off-Axis Fiberglass-Epoxy Composites

Off-Axis Test Matrix

Layup	Fabric	# of tests	Conditioning
$[15]_2$	E-LT 3900	6	3 dry, 3 sat.
$[30]_2$	E-LT 3900	6	3 dry, 3 sat.
$[45]_2$	E-LT 3900	6	3 dry, 3 sat.
[±15]	E-LT 3900	6	3 dry, 3 sat.
[±30]	E-LT 3900	6	3 dry, 3 sat.
[±45]	E-LT 3900	6	3 dry, 3 sat.

Notes:

- 0.05”/min load rate

Partial Saturation Test Matrix

Layup	Fabric	# of tests	Conditioning
$[0/90]_s$	E-LT 3800	5	0.0% Moisture
$[0/90]_s$	E-LT 3800	5	0.2% Moisture
$[0/90]_s$	E-LT 3800	5	0.51% Moisture
$[0/90]_s$	E-LT 3800	5	0.71% Moisture
$[0/90]_s$	E-LT 3800	5	Fully Saturated[1]
$[90/0]_s$	E-LT 3800	5	0.0% Moisture
$[90/0]_s$	E-LT 3800	5	0.2% Moisture
$[90/0]_s$	E-LT 3800	5	0.46% Moisture
$[90/0]_s$	E-LT 3800	5	0.67% Moisture
$[90/0]_s$	E-LT 3800	5	Fully Saturated[1]

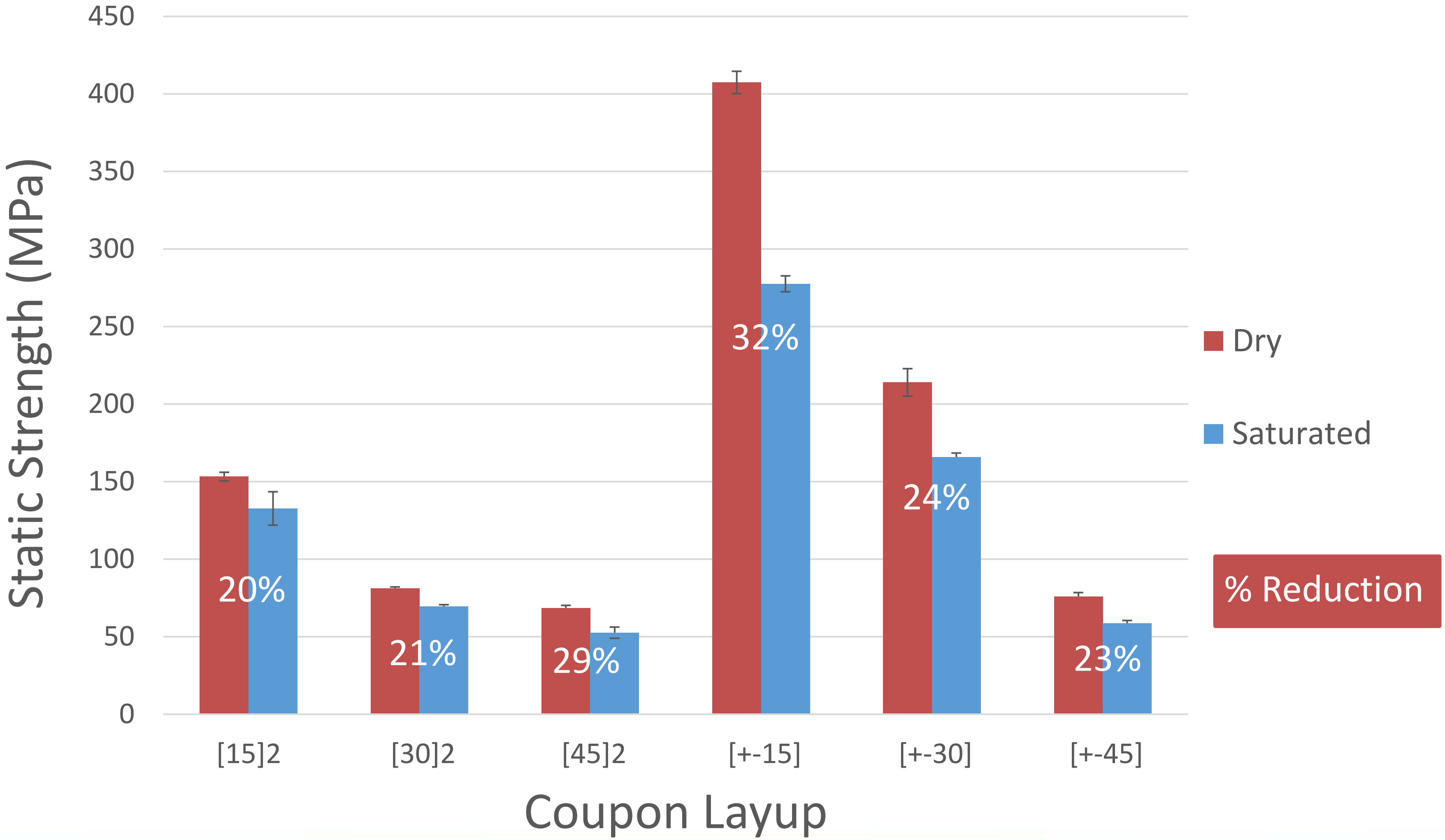
Notes:

- 0.06”/min load rate
- [1] Still undergoing conditioning, results not in presentation

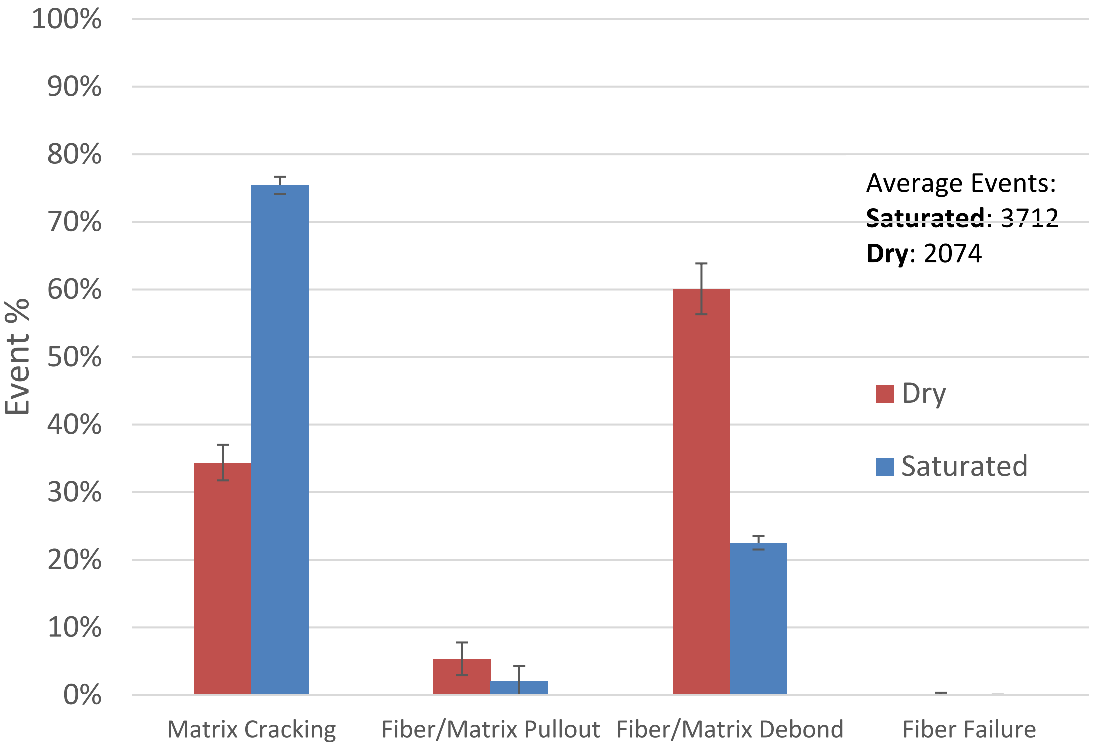
Visible Absorption Effects

- White striations visible after absorption
  - Along fiber angles
  - Consistent throughout all laminates
- Microscopic imaging inconclusive

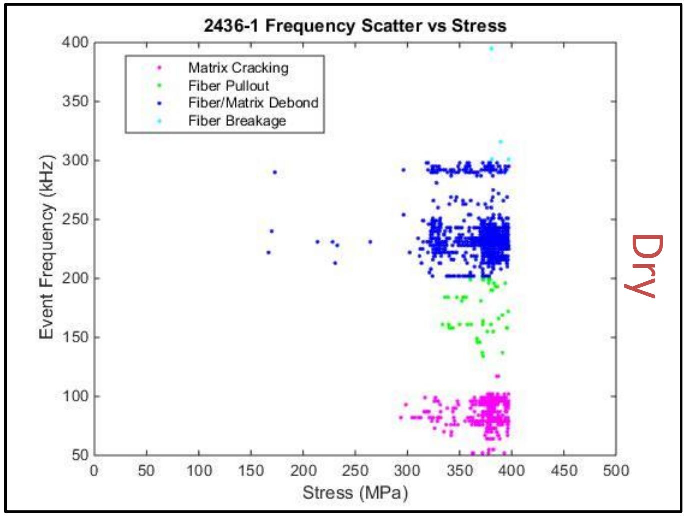
Off-Axis Static Strength

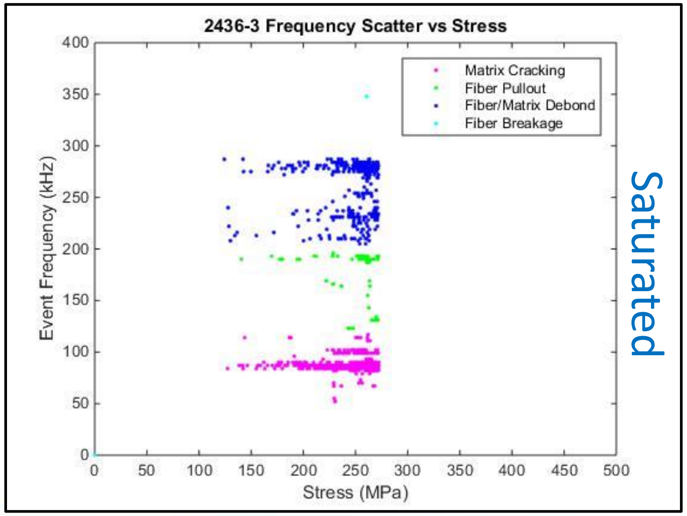


Percentage of Damage Mechanisms [±15] E-LT 3900



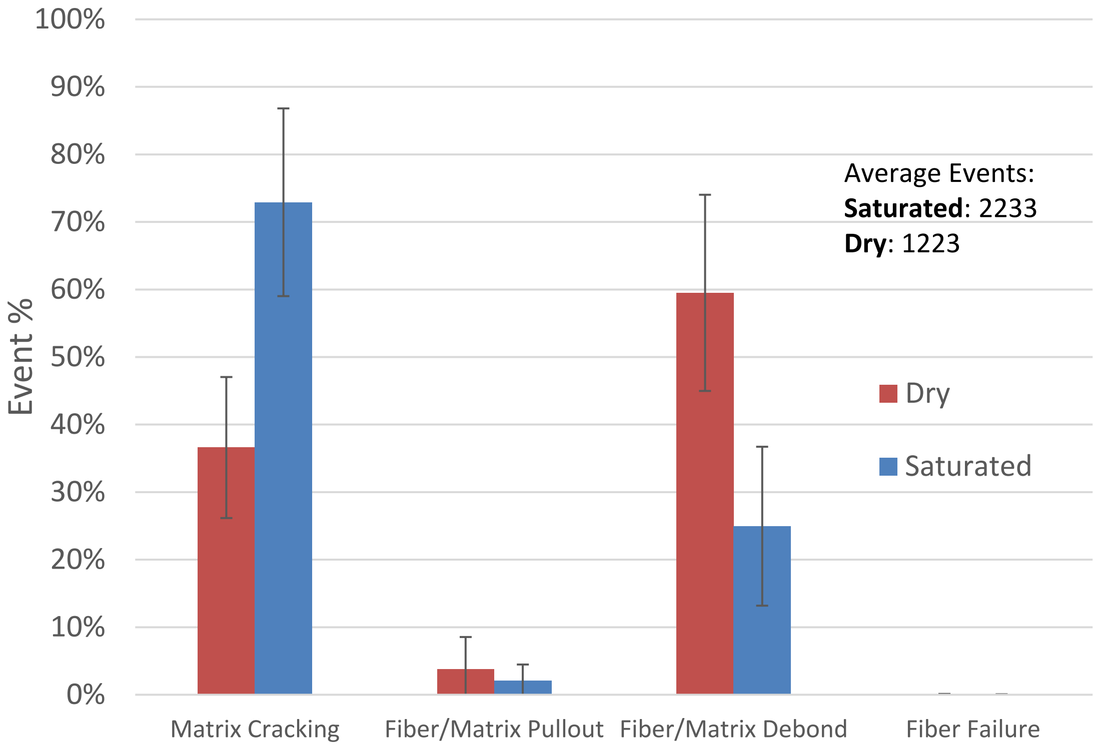
Average Events:
Saturated: 3712
Dry: 2074



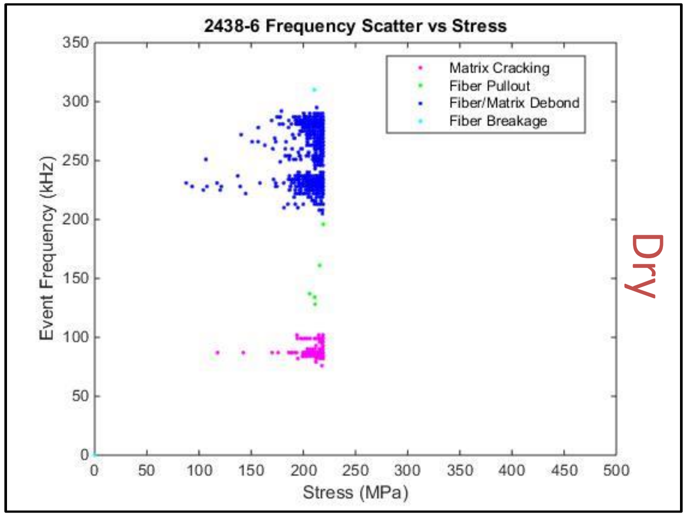


Note: 3 samples tested for saturated and dry conditions, standard deviation error bars

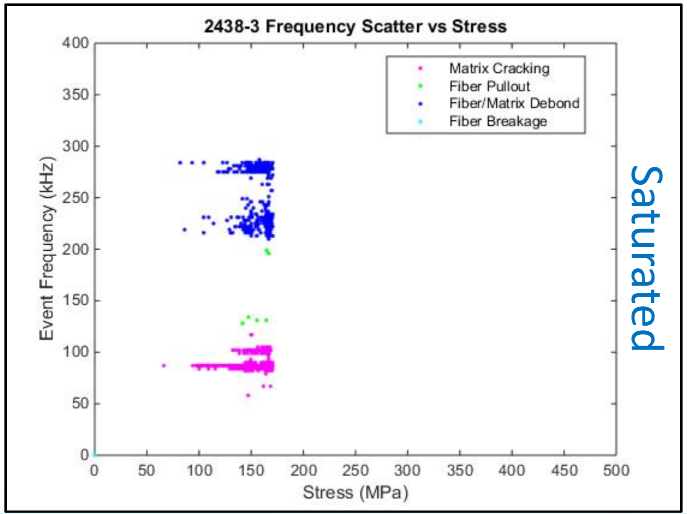
Percentage of Damage Mechanisms [±30] E-LT 3900



Average Events:
Saturated: 2233
Dry: 1223



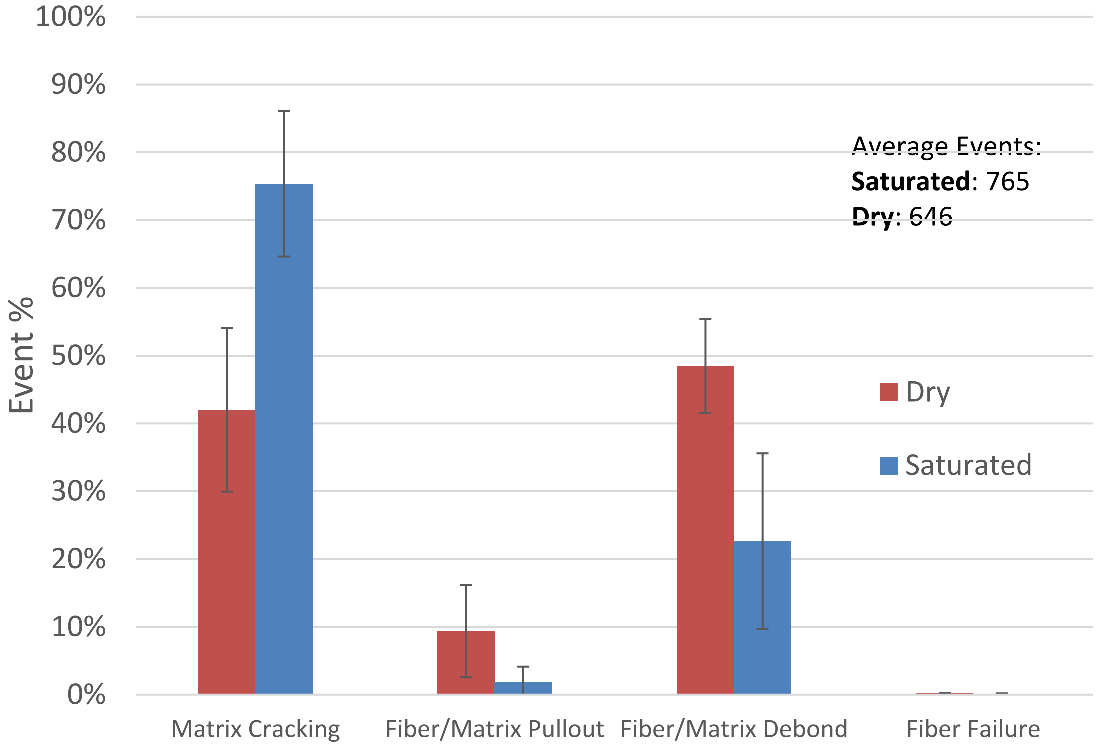
Dry



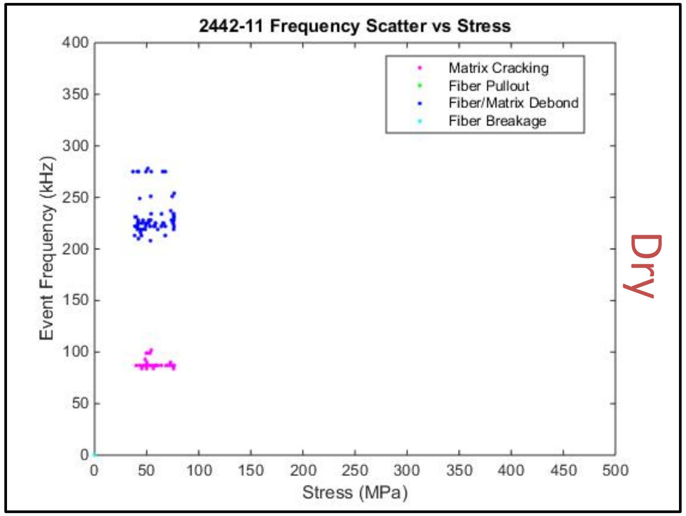
Saturated

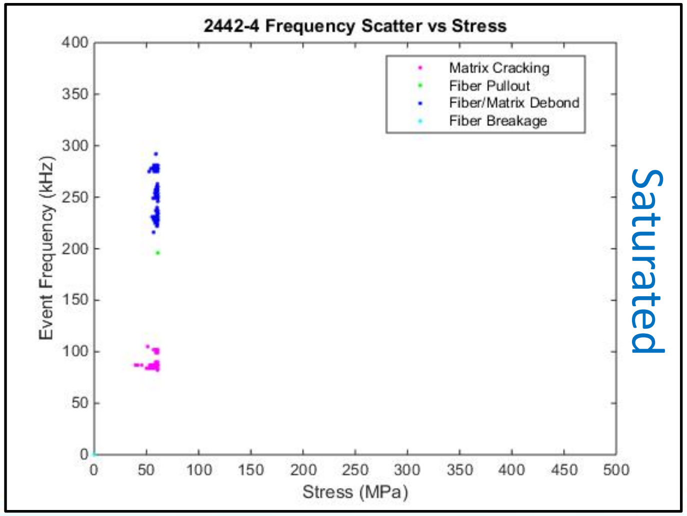
Note: 3 samples tested for saturated and dry conditions, standard deviation error bars

Percentage of Damage Mechanisms [±45] E-LT 3900



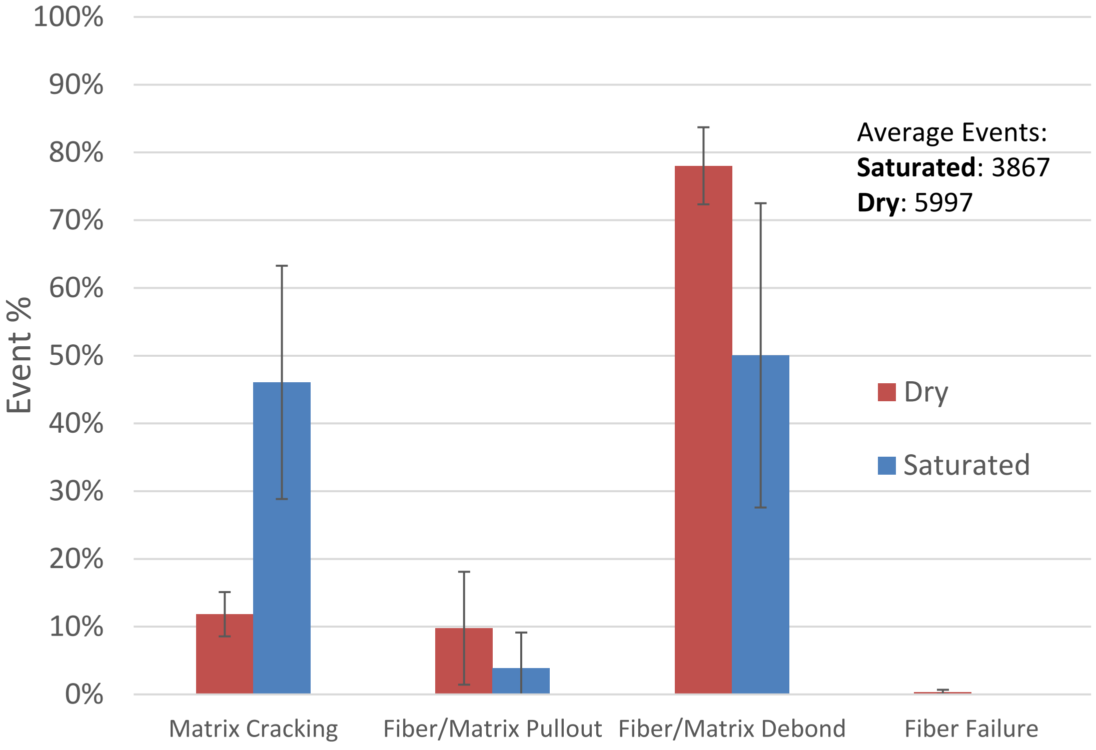
Average Events:
Saturated: 765
Dry: 646





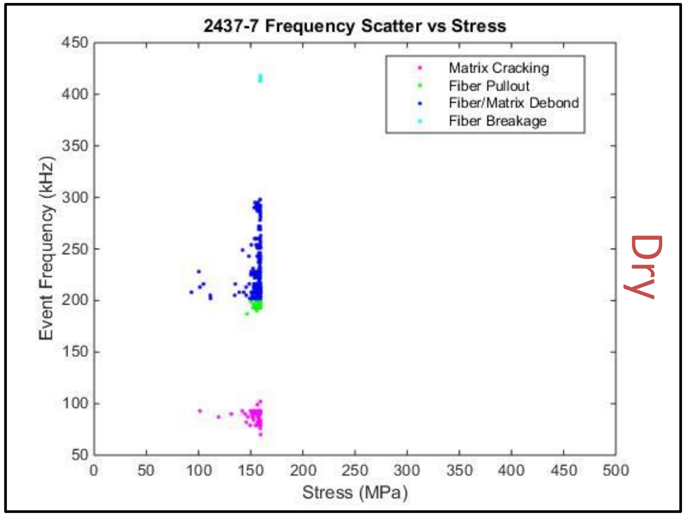
Note: 3 samples tested for saturated and dry conditions, standard deviation error bars

Percentage of Damage Mechanisms $[15]_2$ E-LT 3900

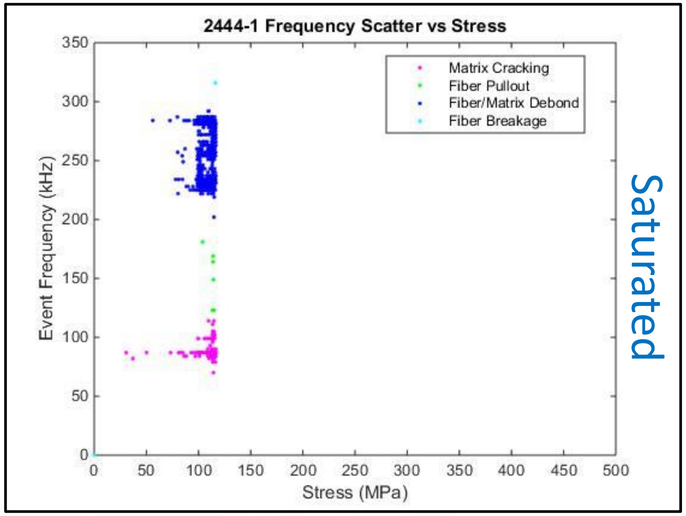


Average Events:
Saturated: 3867
Dry: 5997

Note: 3 samples tested for saturated and dry conditions, standard deviation error bars

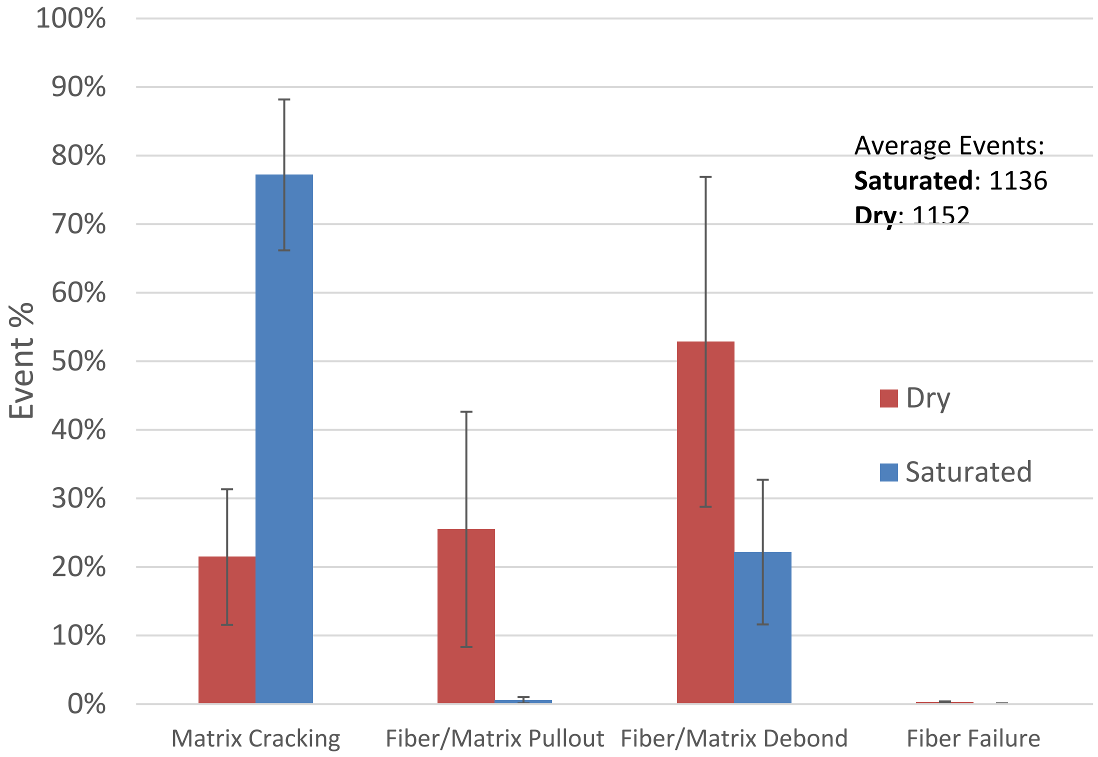


Dry



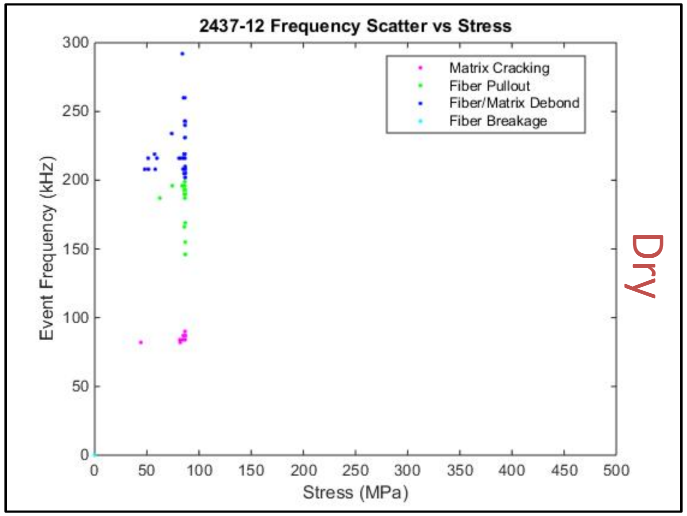
Saturated

Percentage of Damage Mechanisms $[30]_2$ E-LT 3900

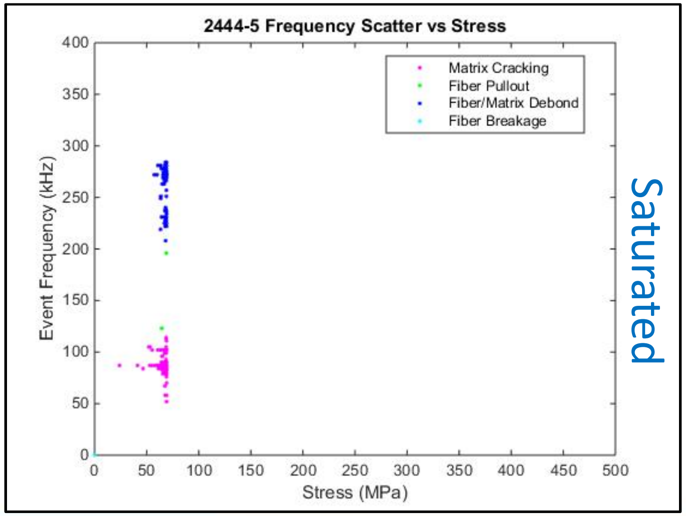


Average Events:
Saturated: 1136
Dry: 1152

Note: 3 samples tested for saturated and dry conditions, standard deviation error bars

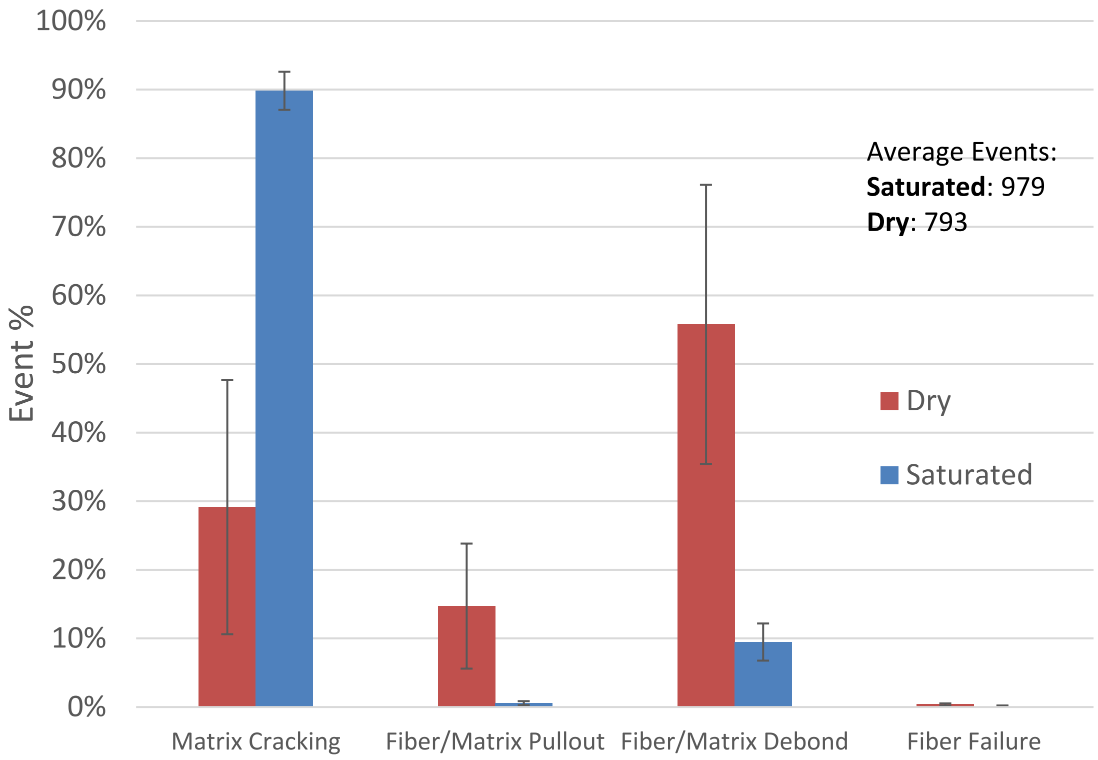

2437-12 Frequency Scatter vs Stress

Dry

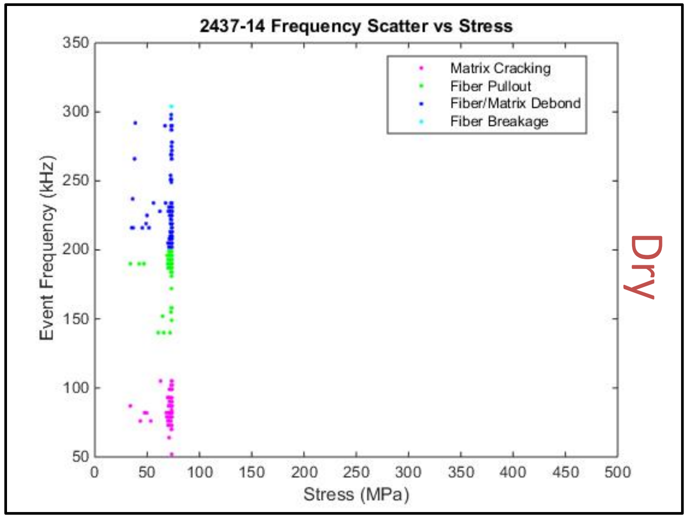

2444-5 Frequency Scatter vs Stress

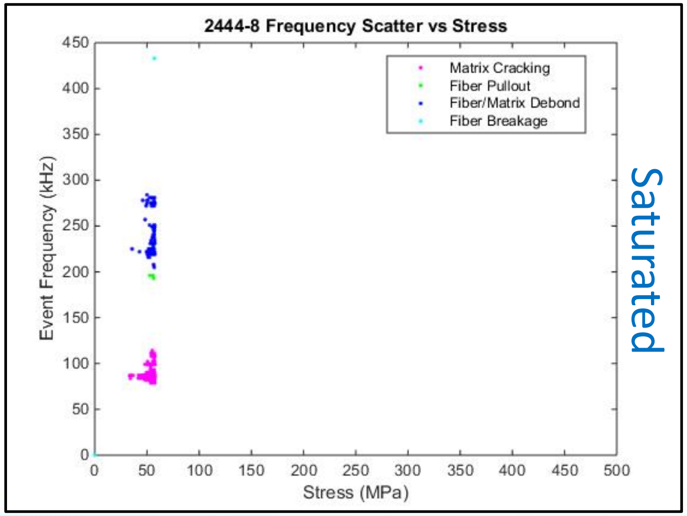
Saturated

Percentage of Damage Mechanisms $[45]_2$ E-LT 3900



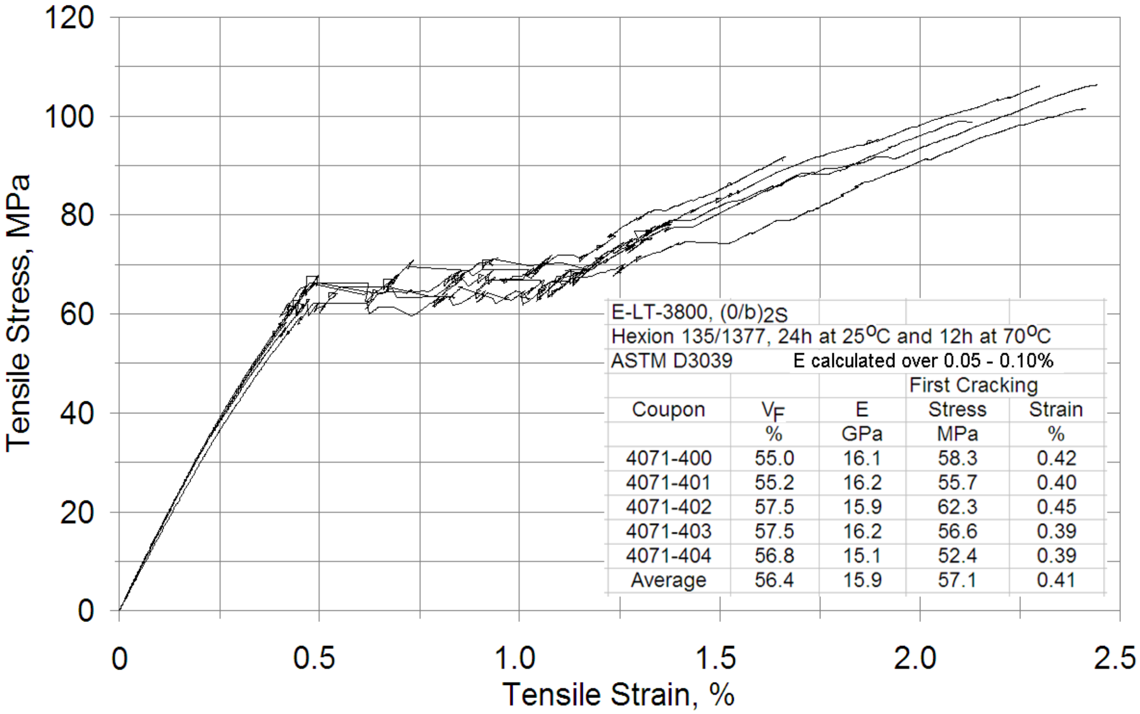
Average Events:
Saturated: 979
Dry: 793





Note: 3 samples tested for saturated and dry conditions, standard deviation error bars

Model Parameters



E-LT-3800, $(0/b)_{2S}$
Hexion 135/1377, 24h at 25°C and 12h at 70°C
ASTM D3039 E calculated over 0.05 - 0.10%

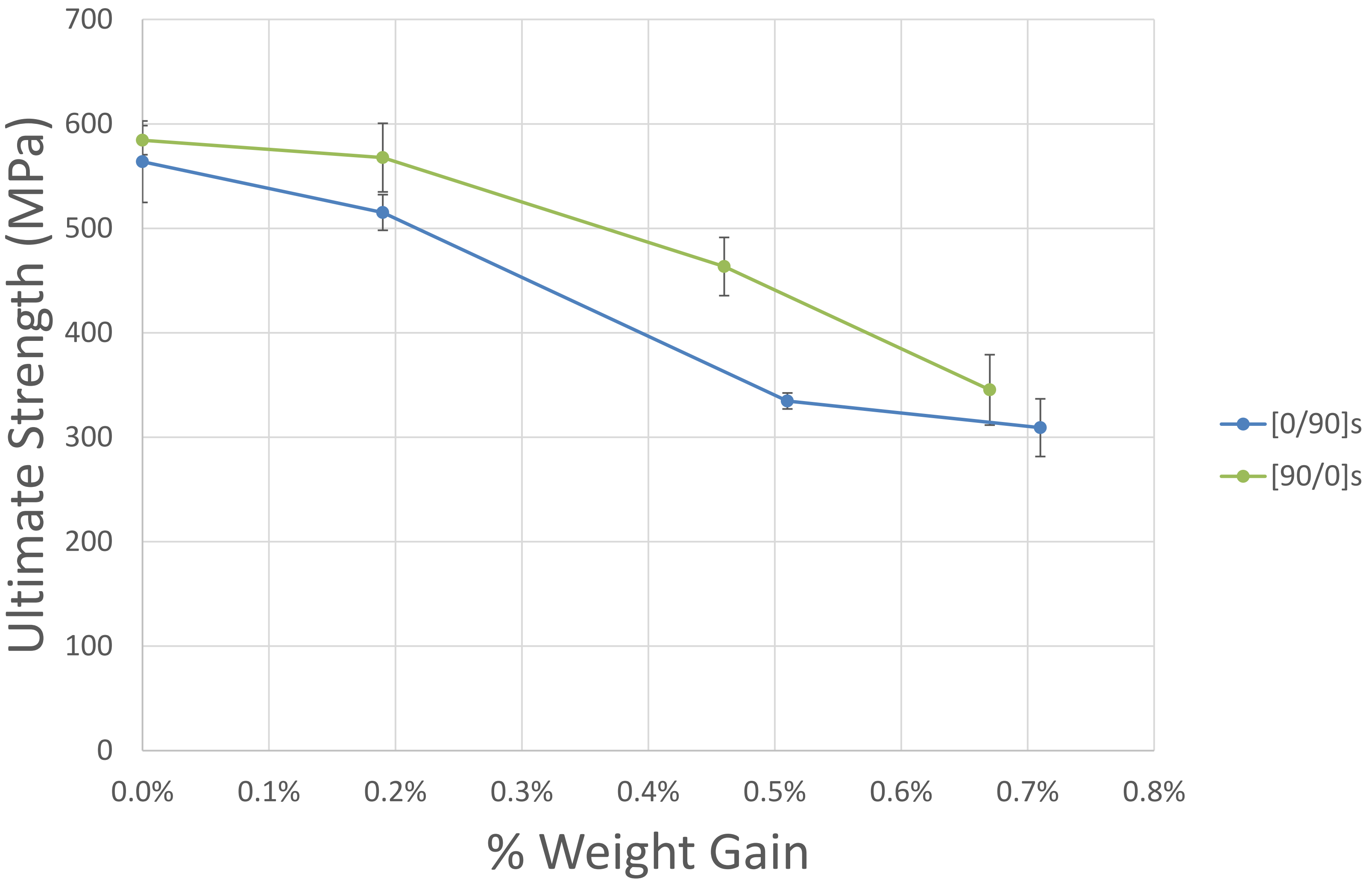
Coupon	V_F %	E GPa	First Cracking Stress MPa	First Cracking Strain %
4071-400	55.0	16.1	58.3	0.42
4071-401	55.2	16.2	55.7	0.40
4071-402	57.5	15.9	62.3	0.45
4071-403	57.5	16.2	56.6	0.39
4071-404	56.8	15.1	52.4	0.39
Average	56.4	15.9	57.1	0.41

- Laminate plate theory is usually used for true unidirectional plies
  - The addition of backing strands and stitching complicates analysis

Results

PARTIAL SATURATION

Ultimate Strength vs % Weight Gain



Conclusions

- Off-axis strength reductions similar to unidirectional
- Max stress failure criterion highlights degradation in shear strength
  - Has to be tuned to dry results
- Acoustic emission analysis indicates a change in damage progression

Conclusions cont.

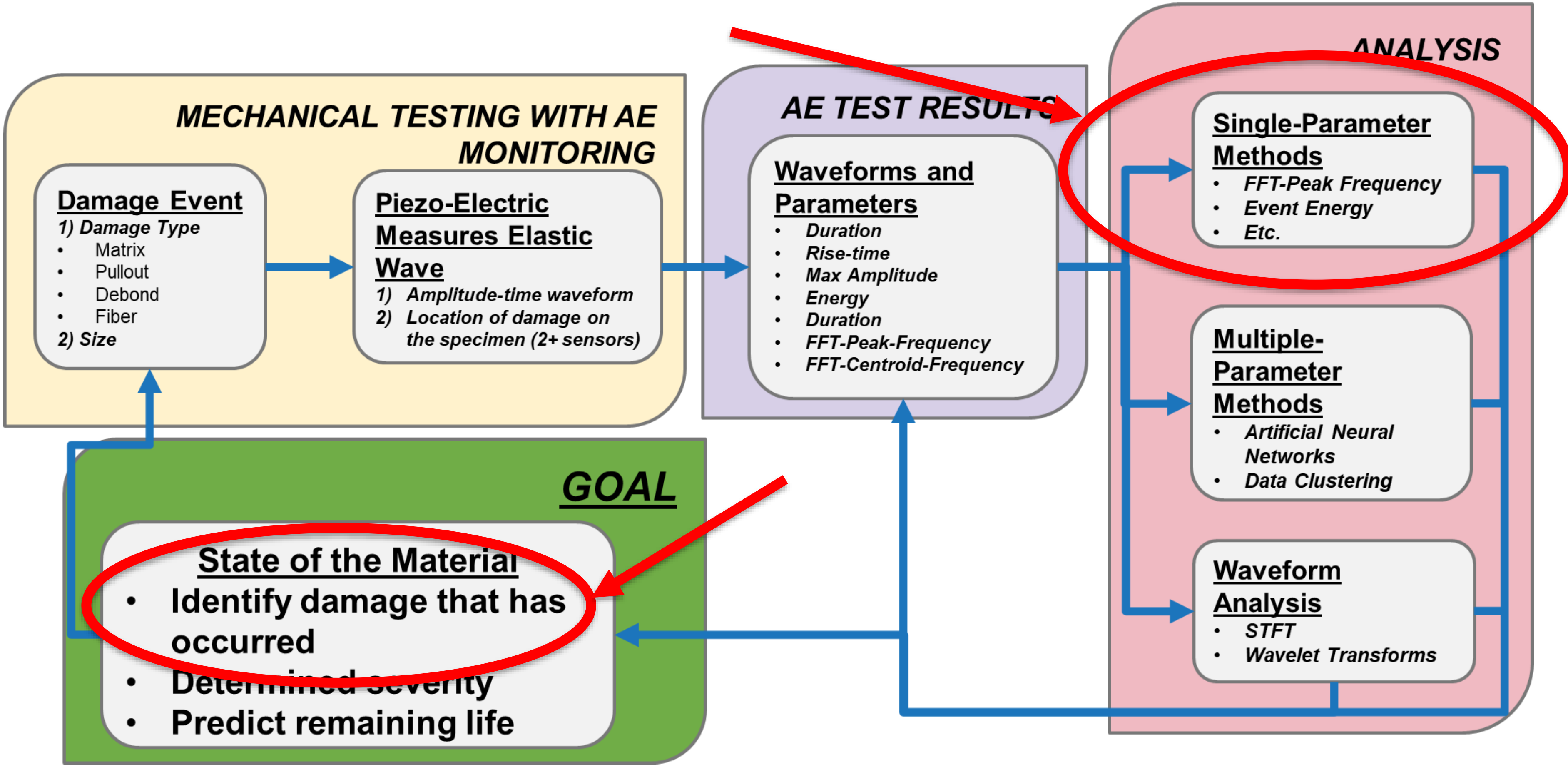
- Dry samples
  - AE analysis shows interfacial damage prior to matrix cracking
- Saturated samples
  - Change in progression indicates matrix cracking beginning prior to interface damage
- Matrix shear strength
- Matrix fracture toughness

Conclusions

- $[0/90]_s$ degraded faster initially than $[90/0]_s$
  - Verifies extension of Fickian diffusion
- Acoustic emission analysis inconclusive
  - Individual layups had different acoustic signatures
  - Comparison of two different layups yet to be successful

An Acoustic Emission and Hygrothermal Aging Study of Fiber Reinforced Polymer Composites

AE System Implementation



MECHANICAL TESTING WITH AE MONITORING

Damage Event

1) Damage Type
- Matrix
- Pullout
- Debond
- Fiber

2) Size

Piezo-Electric Measures Elastic Wave

1) *Amplitude-time waveform*
2) *Location of damage on the specimen (2+ sensors)*

AE TEST RESULTS

Waveforms and Parameters
- *Duration*
- *Rise-time*
- *Max Amplitude*
- *Energy*
- *Duration*
- *FFT-Peak-Frequency*
- *FFT-Centroid-Frequency*

ANALYSIS

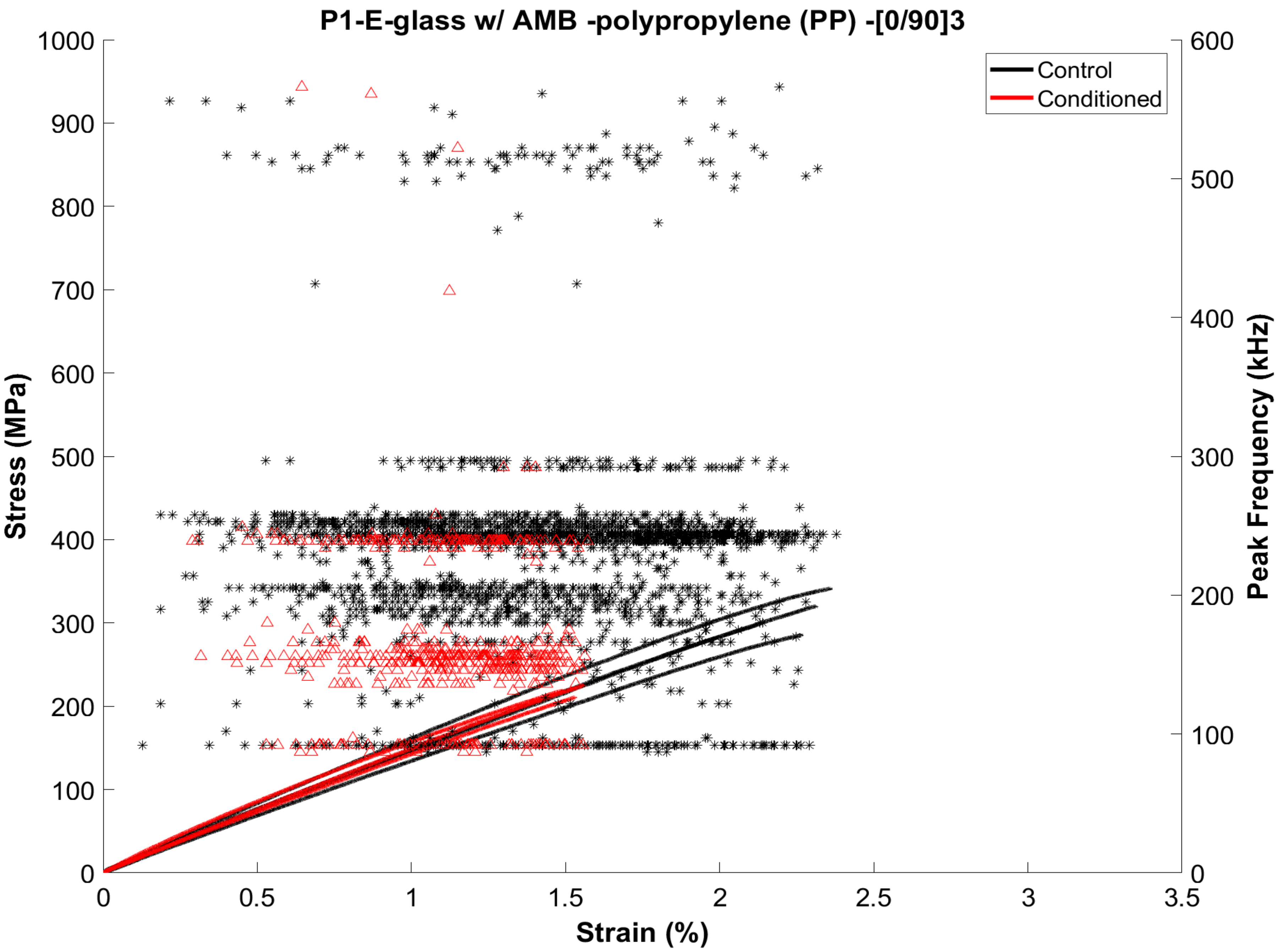
Single-Parameter Methods
- *FFT-Peak Frequency*
- *Event Energy*
- *Etc.*

Multiple-Parameter Methods
- *Artificial Neural Networks*
- *Data Clustering*

Waveform Analysis
- *STFT*
- *Wavelet Transforms*

GOAL

State of the Material
- **Identify damage that has occurred**
- **Determined severity**
- **Predict remaining life**


P1-E-glass w/ AMB -polypropylene (PP) -[0/90]3
Stress (MPa)
0
100
200
300
400
500
600
700
800
900
1000
Peak Frequency (kHz)
0
100
200
300
400
500
600
Strain (%)
0
0.5
1
1.5
2
2.5
3
3.5
Control
Conditioned

MHK Study

- Material Characterization for MHK applications
  - U.S. DOE Water Power Technologies Office
- MHK Database
  - Sandia National Laboratory & MSU
- Industry supplied material systems

MHK Material Summary

Label	Resin	Fabric	Layup
J1	Eastman Copolyester 5011, PETG	Vectorply E-QX 4800	[0/45/90/-45]4
J2	Derakane 470 HT-400 VE	Vectorply E-QX 4800	[0/45/90/-45]4
J3	Applied Poleramic SC18	Vectorply E-QX 4800	[0/45/90/-45]4
J4	Derakane 470 HT-400 VE	OCV WR27TW	[(0/90/)(45/-45)]4
J5	Applied Poleramic SC18	OCV WR27TW	[(0/90/)(45/-45)]4
J6	Applied Poleramic SC18	TPI 4582 (2x2 twill), T700 12K 670 gsm	[(0/90/)(45/-45)]4
J7	Applied Poleramic SC18	Vectorply C-QX 2300 778 gsm, T700 12K Quad	[(0/45/90/-45]4
J8	Derakane 470 HT-400 VE	TPI 4582 (2x2 twill), T700 12K 670 gsm	[(0/45/90/-45]4

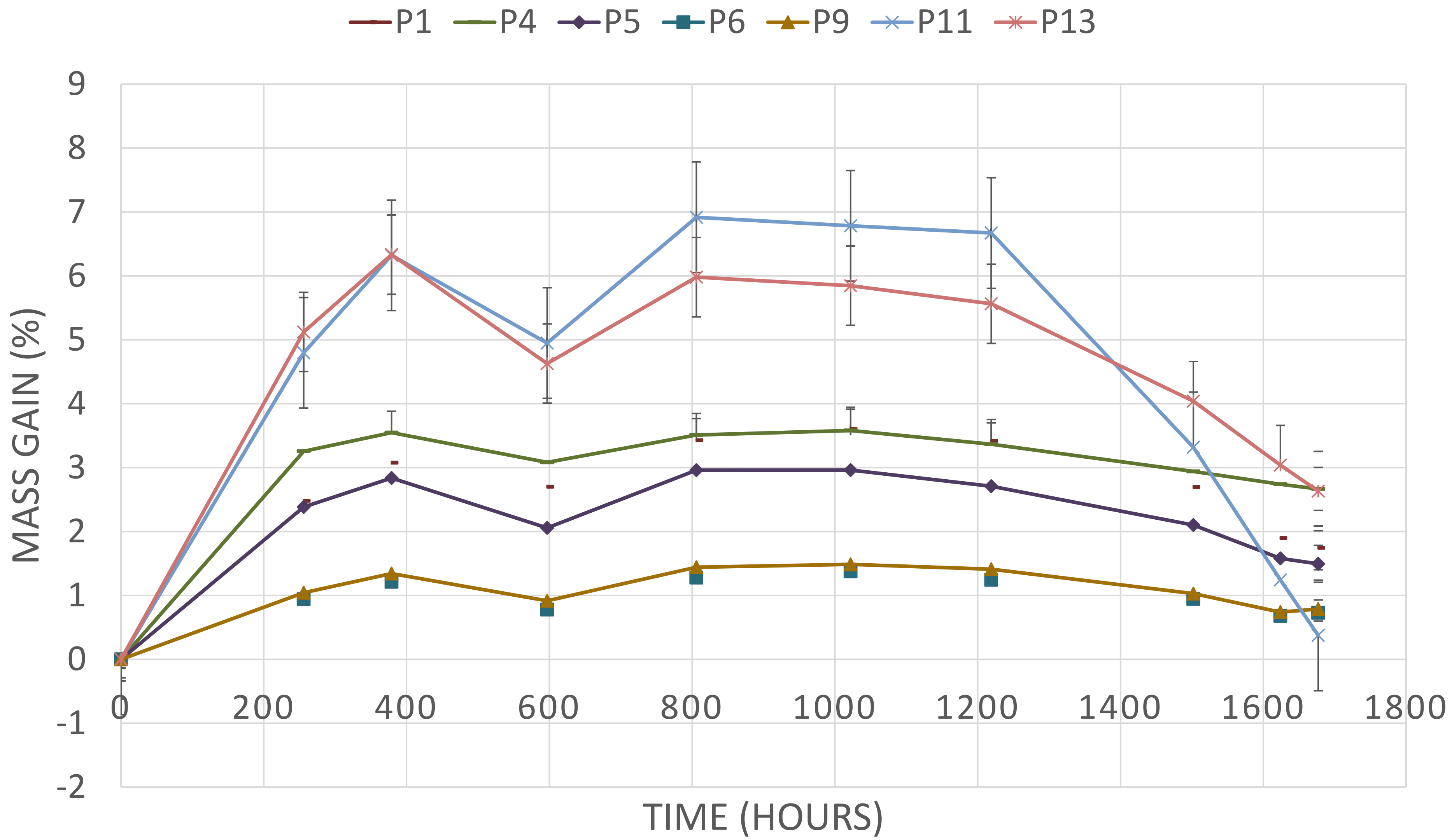
Label	Resin	Fabric (hybrids)	Layup
CE1	Pro-set INF 114/211	Zoltek UD600	[(+45/-45)g/0c]s
CE2	Pro-set INF 114/211	Vectorply CLA 1812	[(+45/-45)g/0c]s
CE3	Hexion RIMR 035c/RIMH 0366	Zoltek UD600	[(+45/-45)g/0c]s
CE4	Hexion RIMR 035c/RIMH 0366	Vectorply CLA 1812	[(+45/-45)g/0c]s
CE5	Crestapol 1250PUL urethane Acrylate	E-BX 1700, CLA 1812, Veil	[(+45/-45)g/0c]s
CE6	AME 6001 VE +1.5% MCP	ELT-2900, E-BX 1700, ELT-2900	[0/+45/-45/0]s

Label	Resin	Fabric	Layup
N1	Elium	JM 086	[0b]2s

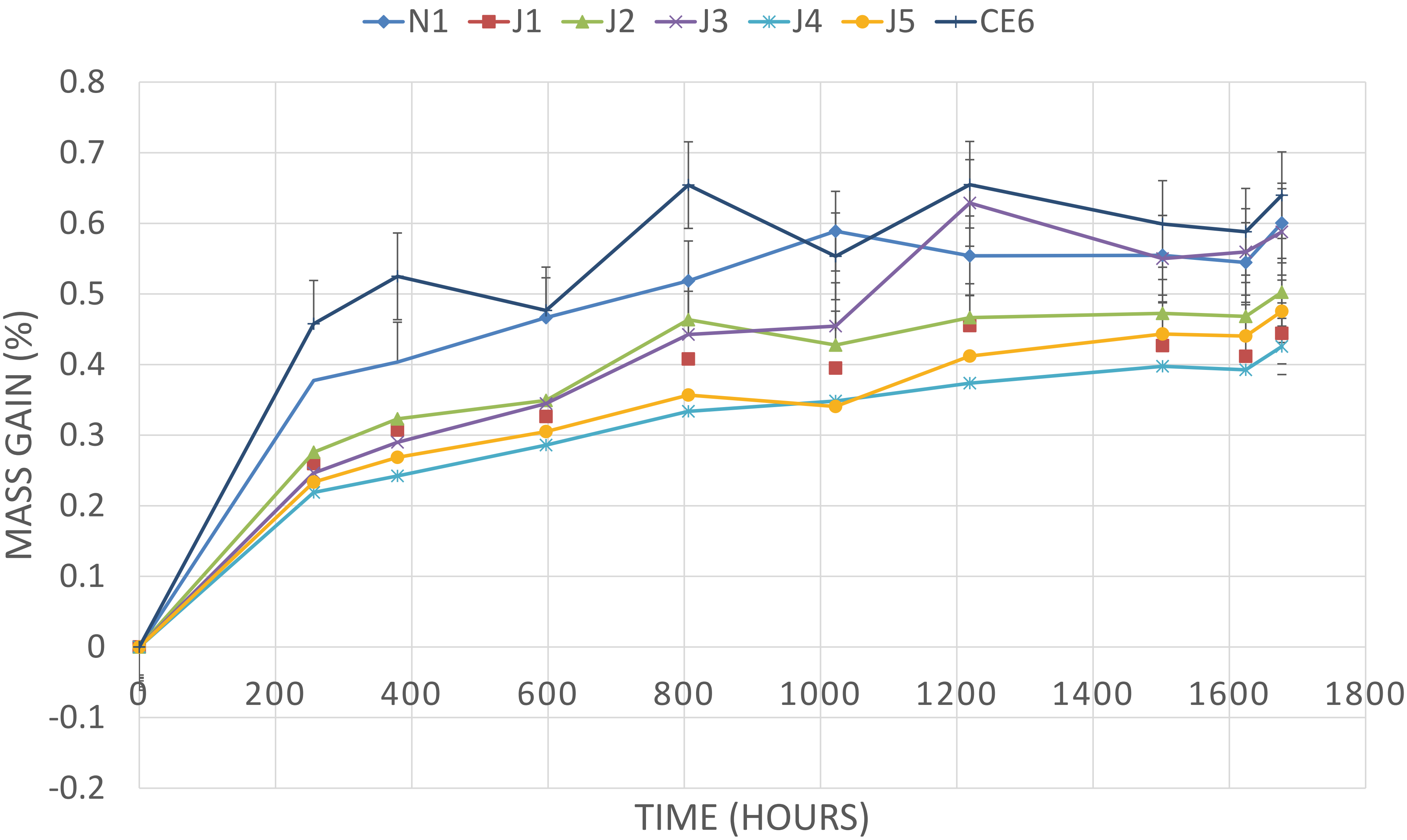
MHK Material Summary Cont.

Label	Resin	Fabric	Layup
P1	PP	E-glass w/AMB	[0/90]3
P4	PA6	E-glass	[0/90]3
P5	PA11	E-glass	[0/90]3
P6	PET	E-glass	[0/90]3
P9	PETG	E-glass	[0/90]3
P11	HDPE	E-glass	[0/90]3
P13	PP	E-glass	[0/90]3

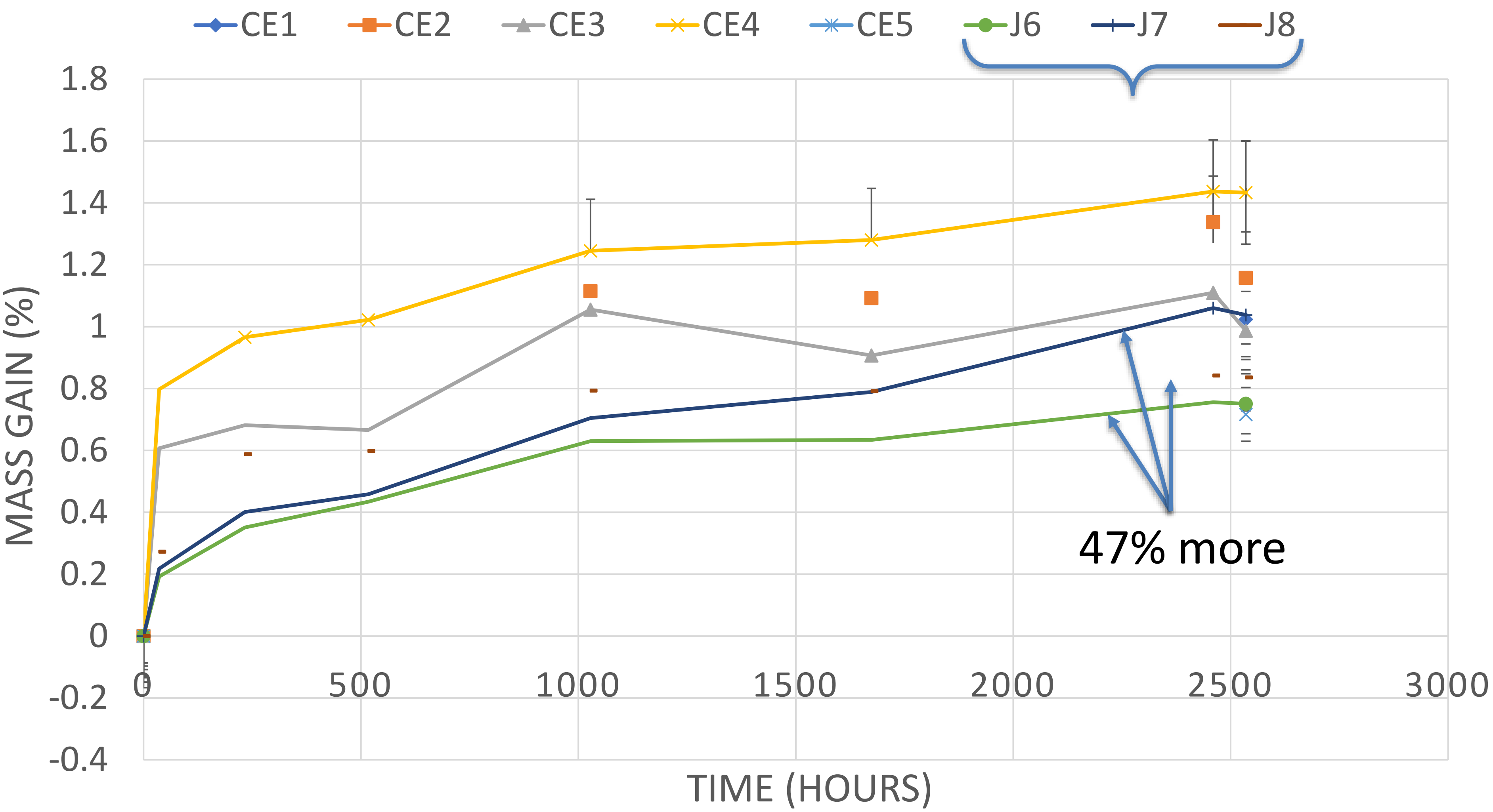
GLASS IN THERMOPLASTIC



GLASS IN THERMOSET

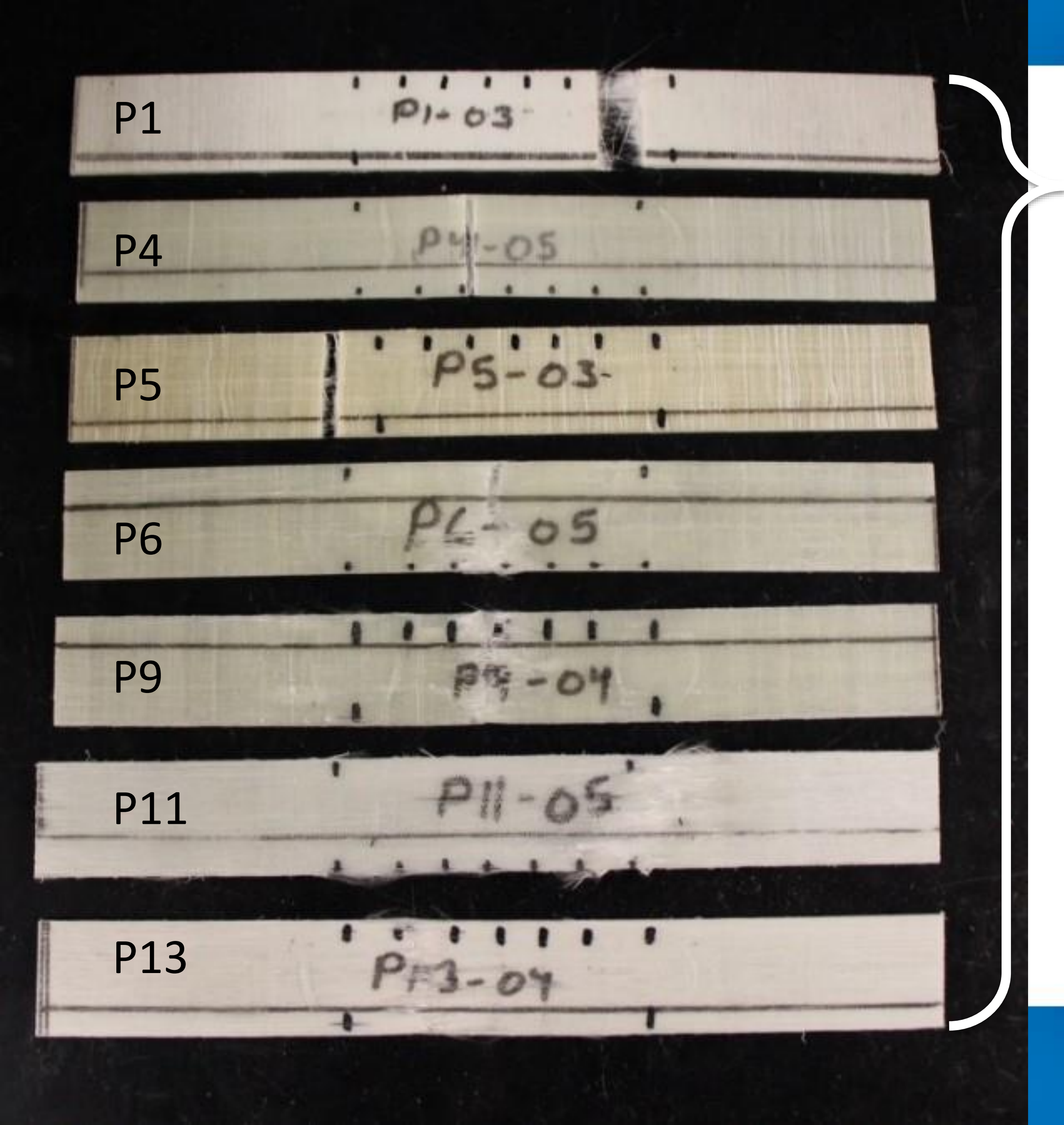


CARBON & CARBON-GLASS HYBRID SYSTEMS



Tested in quasi-static axial tension

Partial Saturation



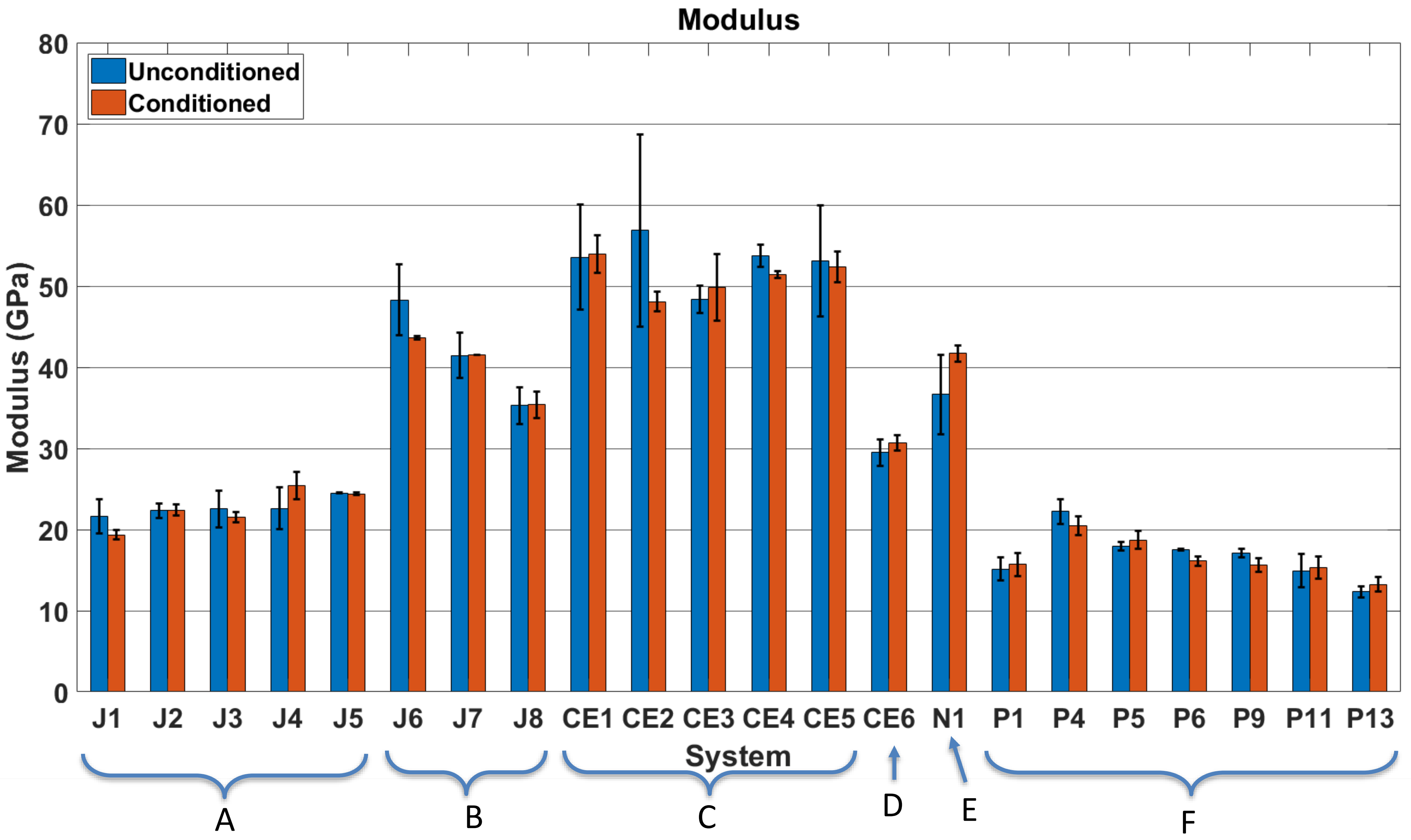
[0/90]3

quasi-static
axial tension

Partial
Saturation

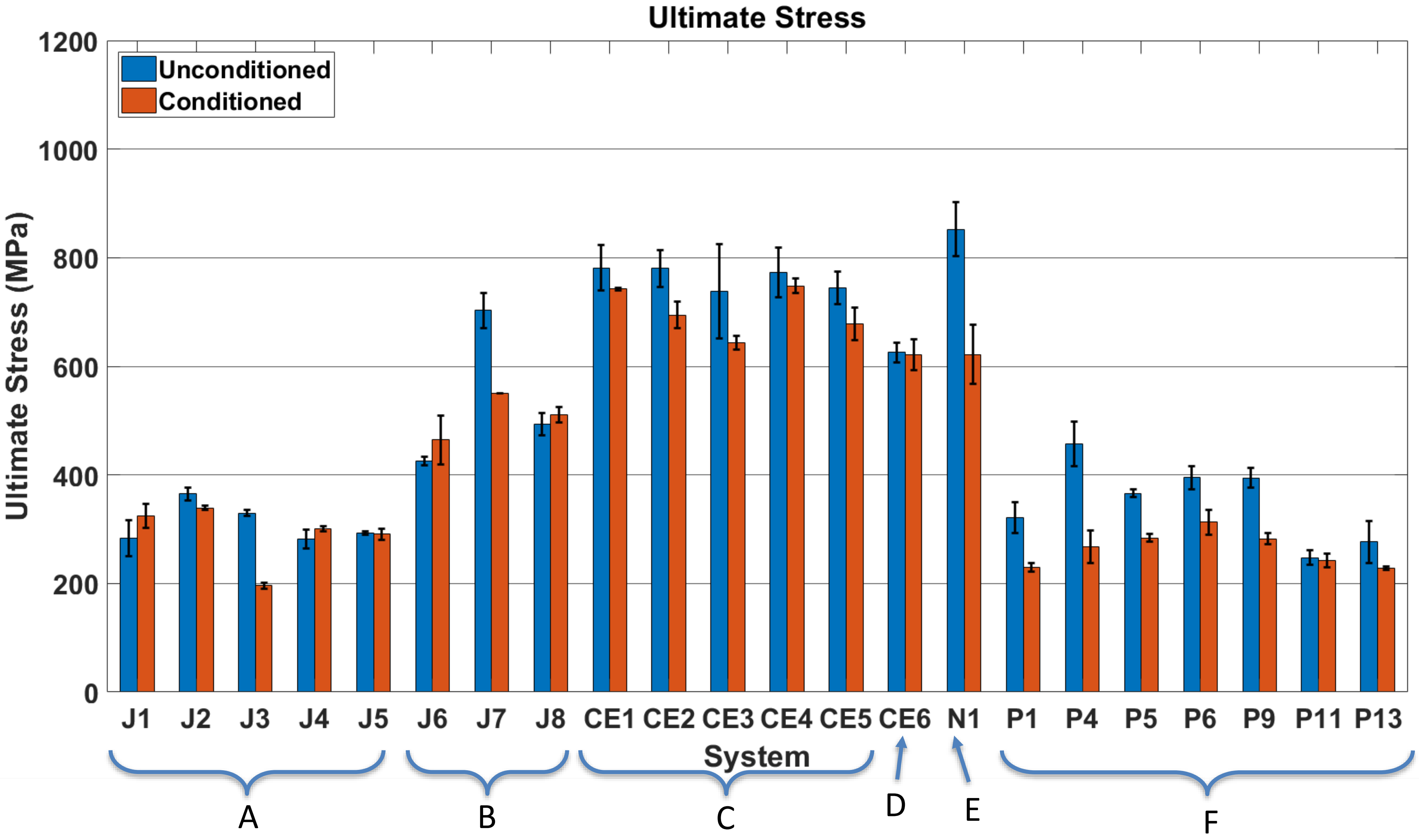
Tested in quasi-static axial tension

Partial Saturation



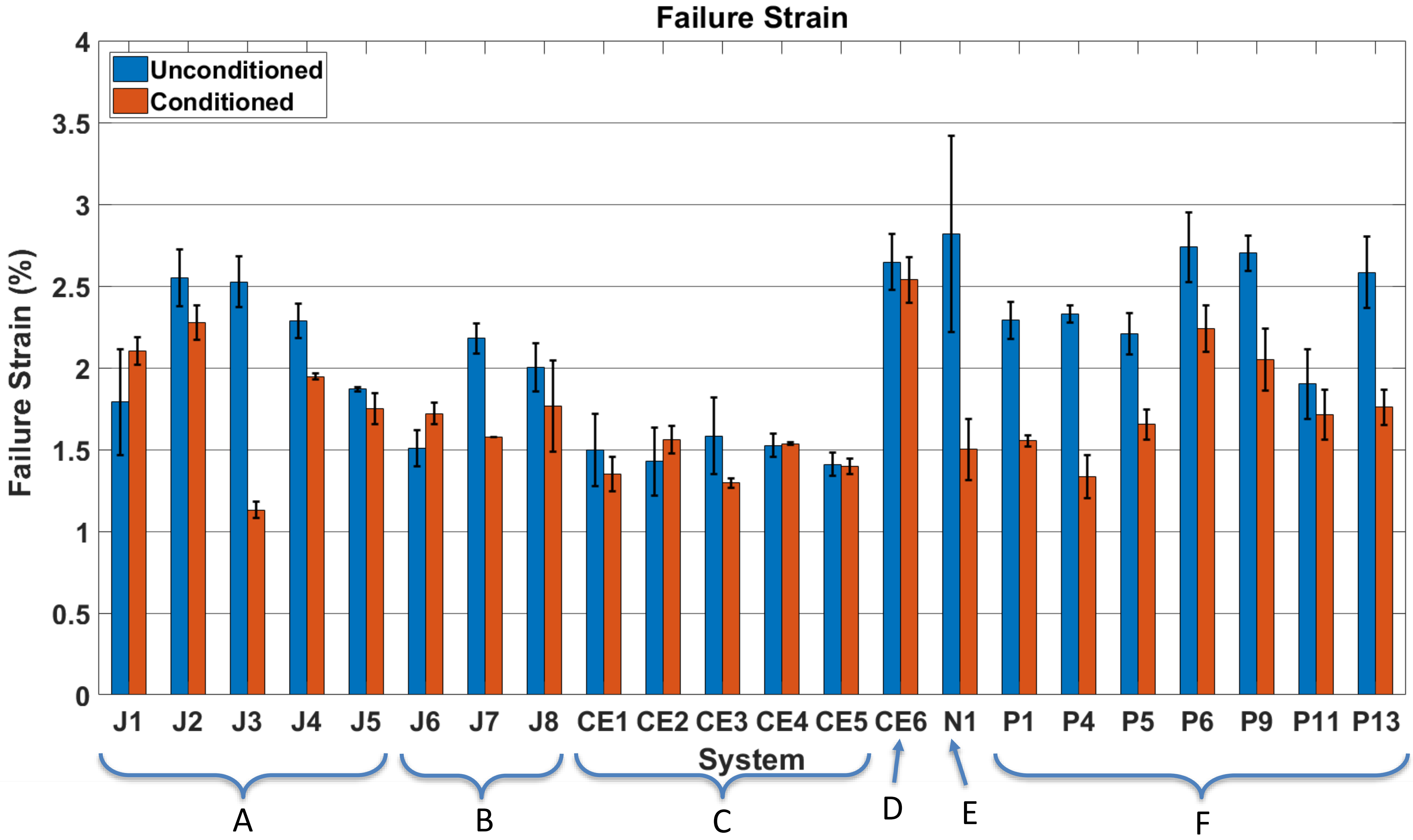
Group	Fiber	Matrix	Layup Type
A	Glass	Thermoset	Quasi-Isotropic
B	Carbon	Thermoset	Quasi-isotropic
C	Hybrid	Thermoset	[45/-45/0]s

Group	Fiber	Matrix Type	Layup Type
D	Glass	Vinyl ester	[0/45/-45/0]
E	Glass	Elium	[0b]s
F	Glass	Thermoplastic	[0/90]n



Group	Fiber	Matrix	Layup Type
A	Glass	Thermoset	Quasi-Isotropic
B	Carbon	Thermoset	Quasi-isotropic
C	Hybrid	Thermoset	[45/-45/0]s

Group	Fiber	Matrix Type	Layup Type
D	Glass	Vinyl ester	[0/45/-45/0]
E	Glass	Elium	[0b]s
F	Glass	Thermoplastic	[0/90]n

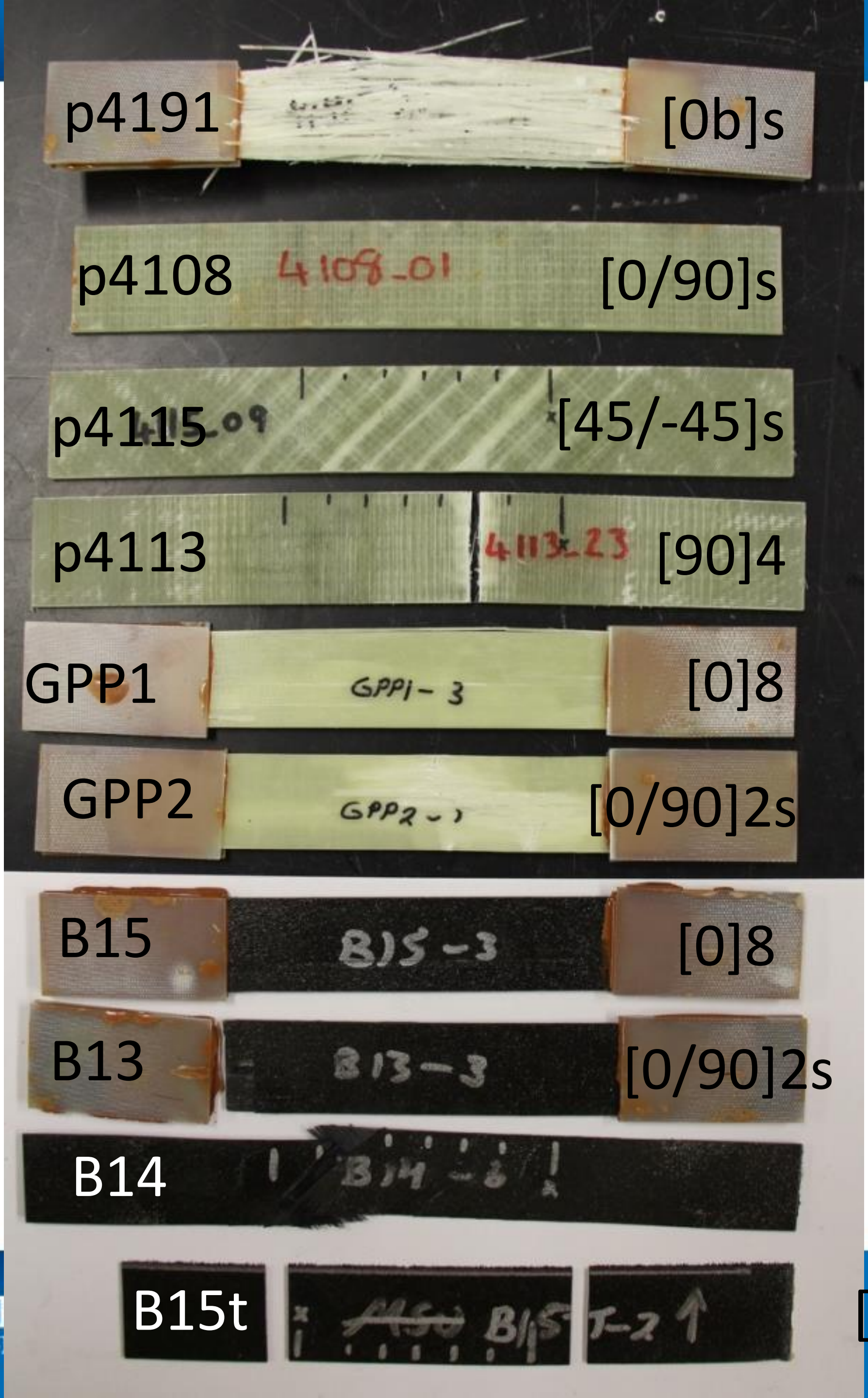


Group	Fiber	Matrix	Layup Type
A	Glass	Thermoset	Quasi-Isotropic
B	Carbon	Thermoset	Quasi-isotropic
C	Hybrid	Thermoset	[45/-45/0]s

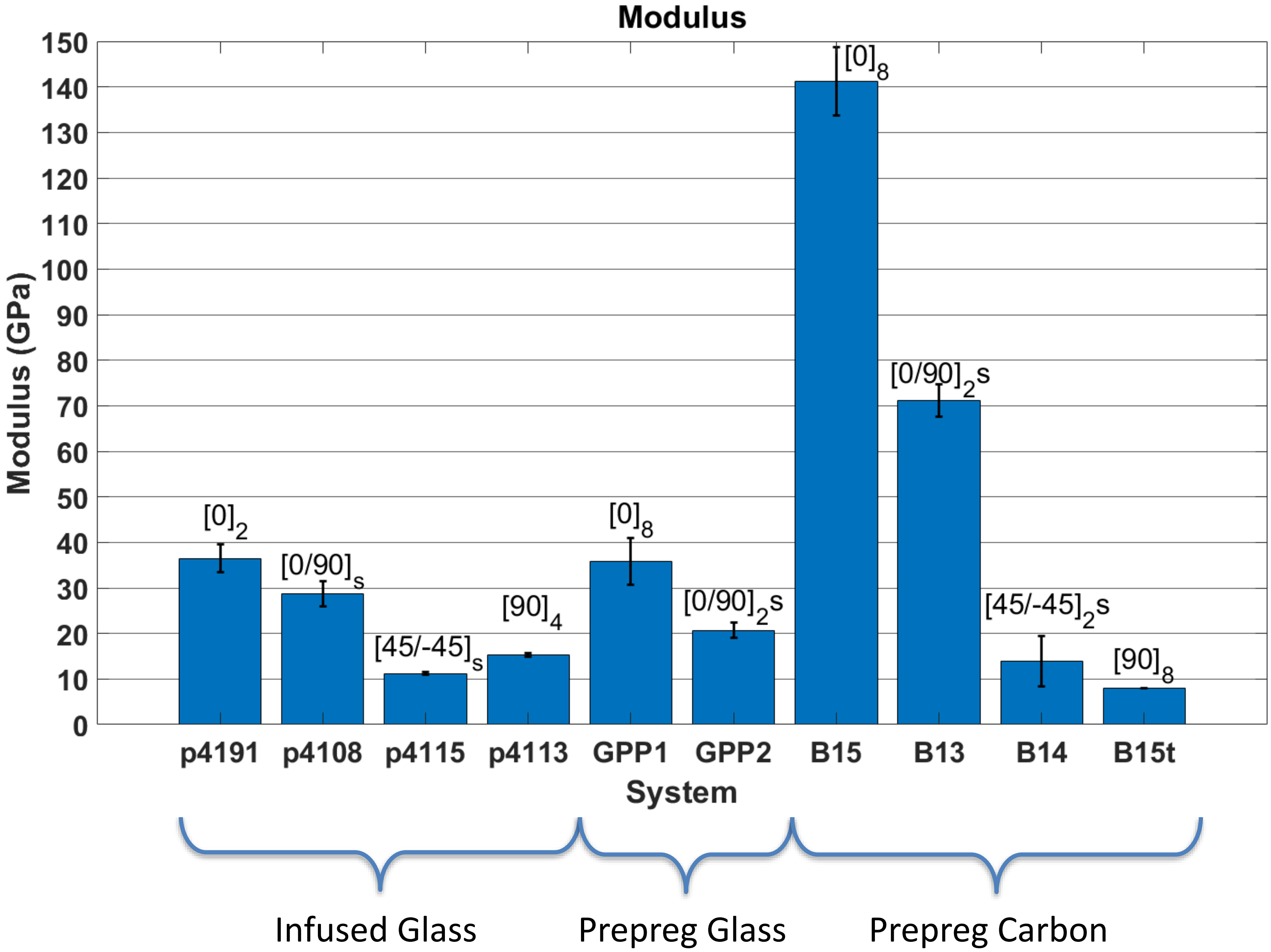
Group	Fiber	Matrix Type	Layup Type
D	Glass	Vinyl ester	[0/45/-45/0]
E	Glass	Elium	[0b]s
F	Glass	Thermoplastic	[0/90]n

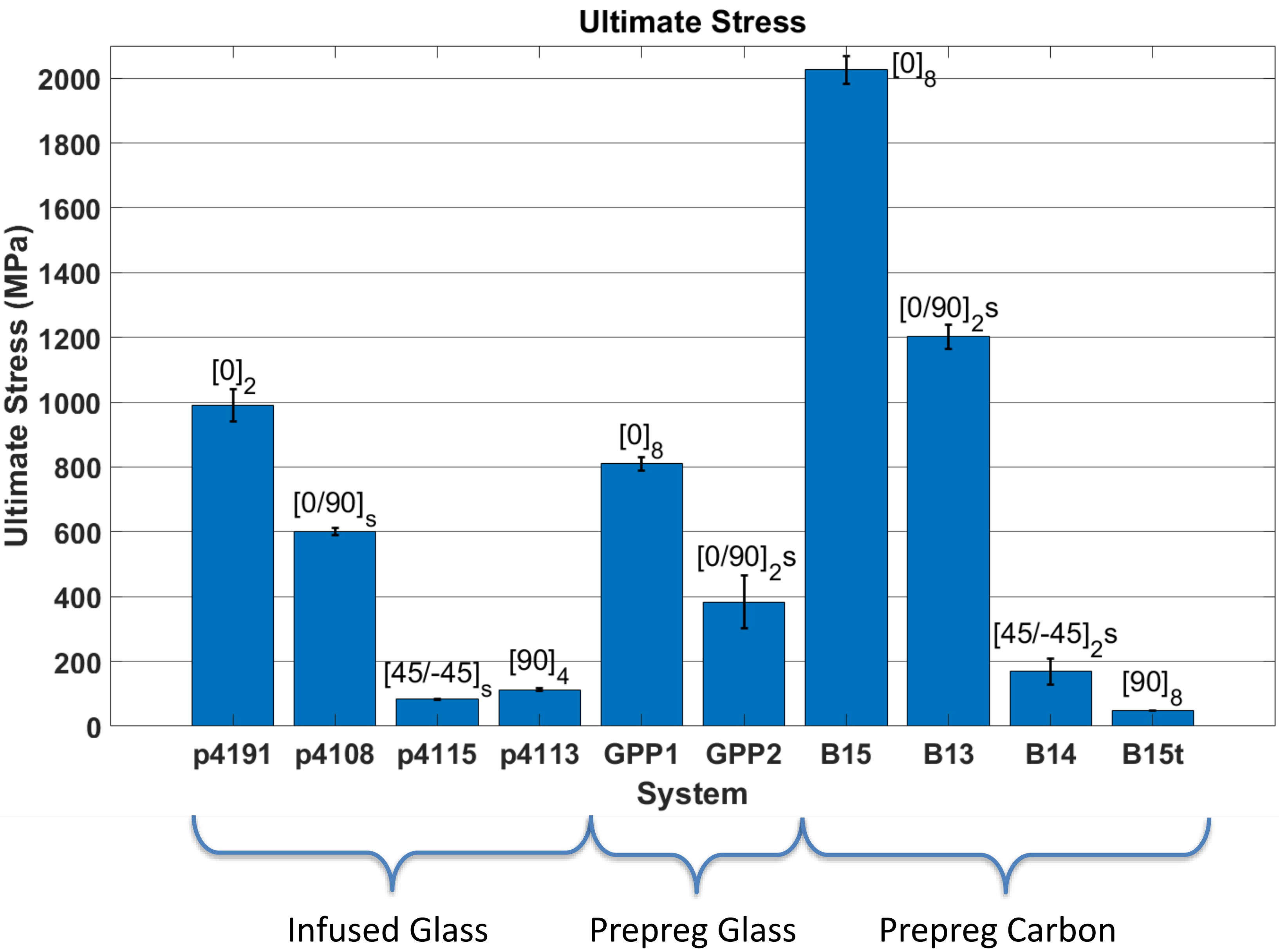
Conclusions

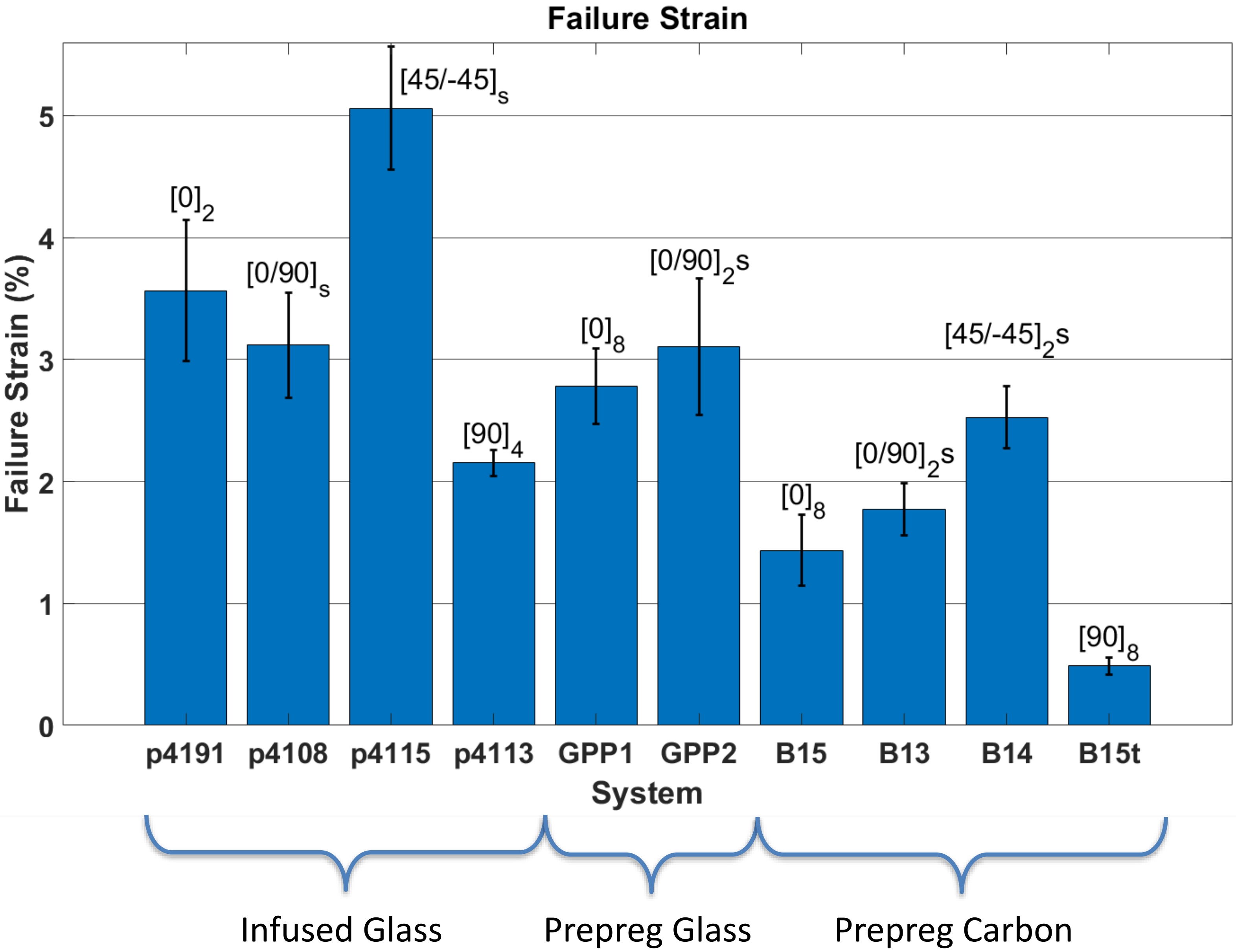
- Moisture Uptake
  - Thermoplastics have higher diffusion constants and free volumes than thermosets
  - Carbon Laminates absorb more moisture than glass laminates
    - Normalized by volume fraction comparing matrix
  - Thermoplastic laminates are observed to degrade (lose mass) in heated SSW after ~1000 hours



Tested in quasi-static axial tension


Modulus
Modulus (GPa)
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
$[0]_2$
$[0/90]_s$
$[45/-45]_s$
$[90]_4$
$[0]_8$
$[0/90]_2s$
$[0]_8$
$[0/90]_2s$
$[45/-45]_2s$
$[90]_8$
p4191
p4108
p4115
p4113
GPP1
GPP2
B15
B13
B14
B15t
System
Infused Glass
Prepreg Glass
Prepreg Carbon


Ultimate Stress
Ultimate Stress (MPa)
0
200
400
600
800
1000
1200
1400
1600
1800
2000
[0]$_2$
[0/90]$_s$
[45/-45]$_s$
[90]$_4$
[0]$_8$
[0/90]$_2$s
[0]$_8$
[0/90]$_2$s
[45/-45]$_2$s
[90]$_8$
p4191
p4108
p4115
p4113
GPP1
GPP2
B15
B13
B14
B15t
System
Infused Glass
Prepreg Glass
Prepreg Carbon


Failure Strain
Failure Strain (%)
0
1
2
3
4
5
[0]2
[0/90]s
[45/-45]s
[90]4
[0]8
[0/90]2s
[0]8
[0/90]2s
[45/-45]2s
[90]8
p4191
p4108
p4115
p4113
GPP1
GPP2
B15
B13
B14
B15t
System
Infused Glass
Prepreg Glass
Prepreg Carbon

Conclusions

- Mechanical
  - Moduli are generally unaffected by moisture uptake
  - Strength and failure strain generally decrease with moisture
    - Some exceptions
  - Low quality laminates are affected less

Summary

- MSU and Sandia have performed many tests to characterize and quantify the effects of moisture on composite materials
- Broad range of tests and materials to investigate amount and type of damage
- Still many unanswered questions.