Degradation of Mechanical Properties in Composite Materials Subjected to Salt Water Environments

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MONTANA STATE UNIVERSITY

Mountains & Minds

MSU MHK Program

TASK SUMMARY

- Task 1: Laminate fabrication and characterization
  - **Fabricate laminates by resin infusion**, which are of interest for MHK applications, including multidirectional (0 and ±45 plies) and biax (±45 only) glass fabrics, with epoxy, polyester and vinyl ester resins. **Use materials from the SNL/MSU/DOE Fatigue of Composite Materials Database** which show the best mechanical properties, supplemented with additional materials of interest to the MHK application. **Characterize these materials for strength and fatigue resistance** under ambient laboratory conditions if data are not already available in the Database. Appropriate MHK coatings (to be determined) will be included in selected cases to establish coating cracking strains.
- Task 2: Testing for environmental effects
  - For the laminates in Task 1, condition coupons of approximately 6-mm thickness for 1000 hours in synthetic sea water at an elevated temperature of 40^{o}C; **record weight changes over time**. After 1000 hours of conditioning, shift coupons to the synthetic sea water at approximately 20^{o}C until testing. **Characterize static and fatigue properties at temperatures of approximately 5, 20 and 40^{o}C**. Wet conditioned specimens to be tested in a local coupon environment of synthetic sea water; control specimens to be tested in heated or cooled laboratory air. The effects of coatings will be included for selected cases.
- Task 3: Adhesive joints
  - Using the recently developed thick adhesive fatigue test methods for lap shear and cracked lap shear (CLS) (2010 AIAA SDM paper AIAA-2010-2821, Sears, et al), **determine the effect of MHK environment on at least two adhesive systems of interest to MHK applications.** Follow the parameters in Task 2 for specimen preparation, conditioning and testing. Fatigue testing will be carried out under reversed loading (R = -1) to characterize: (a) lap shear force vs. lifetime trends, and (b) CLS crack fracture toughness and fatigue crack growth rates.

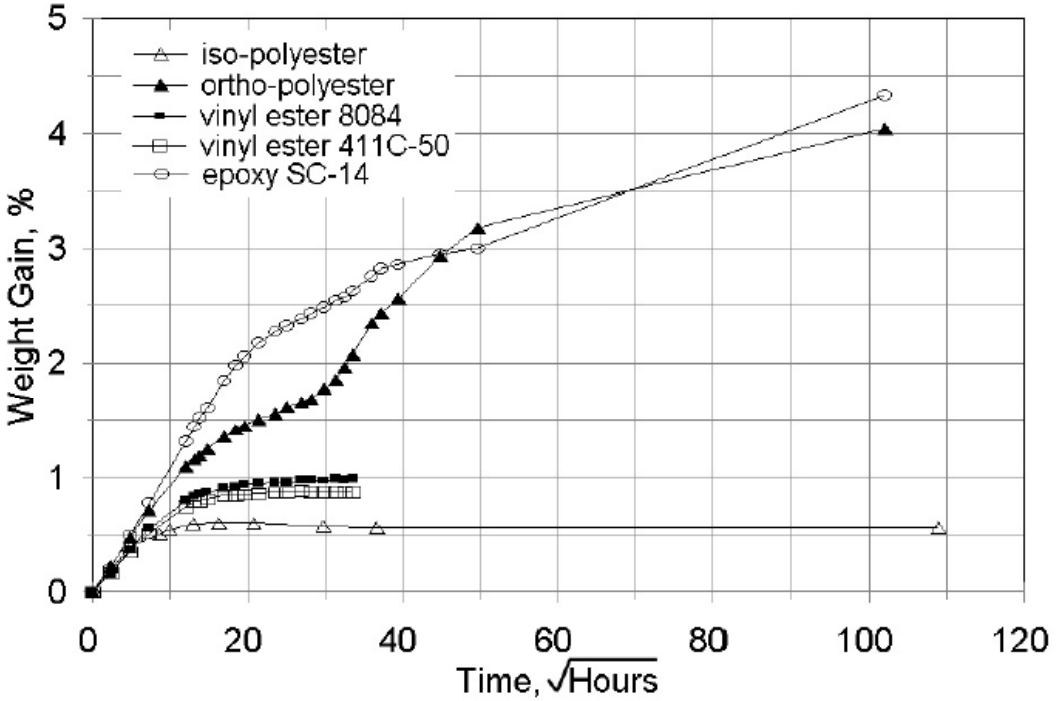
MHK Historical Perspectives

- Issues of composites in water not really new….
- Decades of data ….. mainly focused around aircraft and ship industries from 70's – 90's
- Industries where manufacturing costs are significantly greater *and allowable* than energy technologies
- Continually have new materials
  - Changes are constantly being made in matrix and fiber materials requiring new characterization.

Water Absorption for Neat Resin

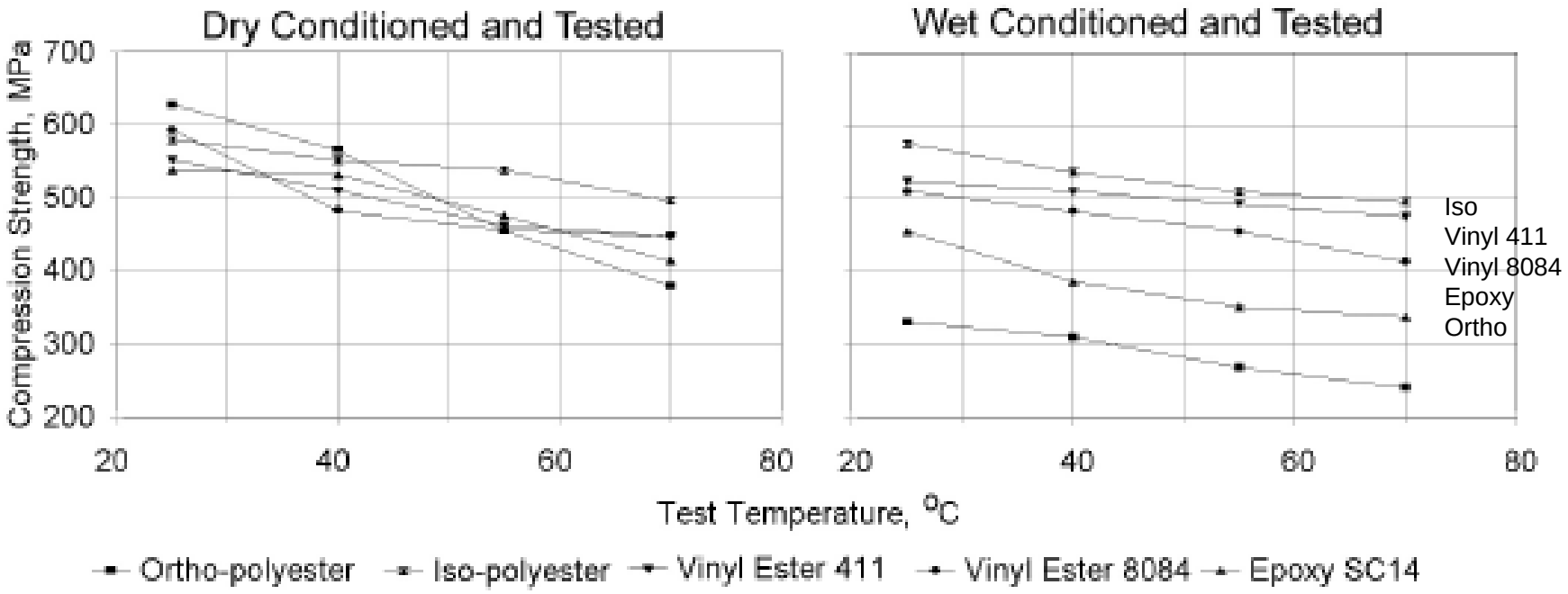
MSU/SNL/DOE 2002 Report

- Distilled water soaking
- Accelerated aging at 50°C
- Vinyl esters and iso-polyesters less uptake than ortho-polyester and epoxy
- Bi-linear saturation mode for epoxy and ortho-polyester
- Fickian diffusion until saturation for 3 systems
- 8084- rubber toughened



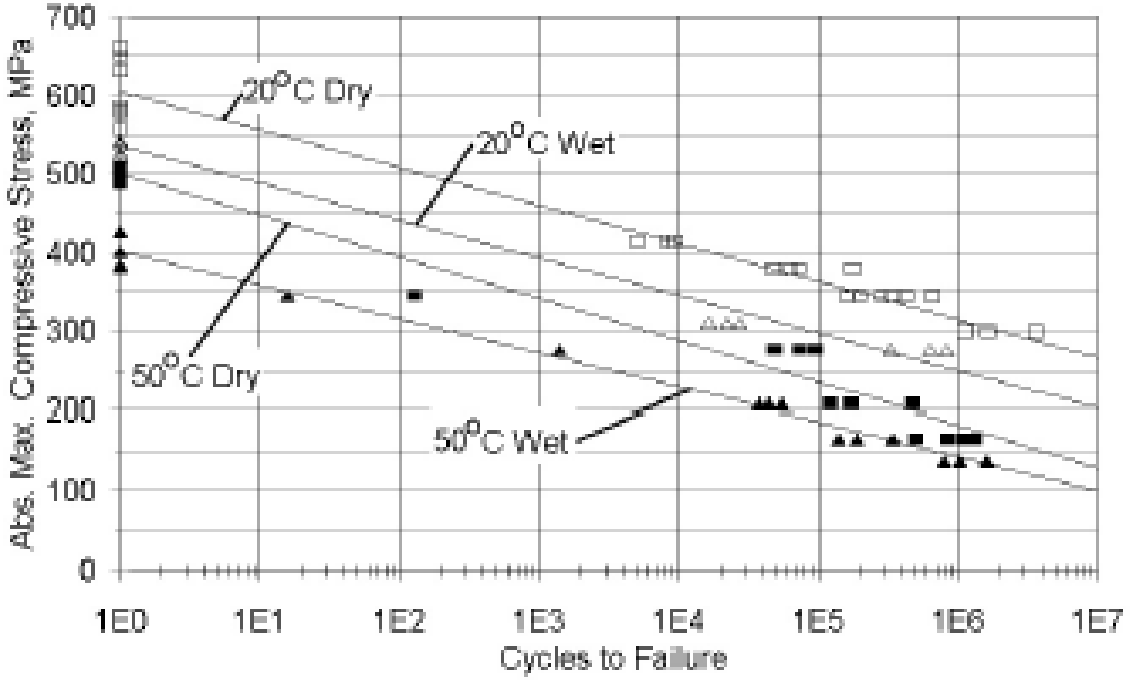
Water Absorption for Composite
MSU/SNL/DOE 2002 Report

- Compressive Strength, 0° Dir, of $[0/\pm 45/0]_S$, $V_f = 0.36$



Water Absorption for Composite
MSU/SNL/DOE 2002 Report

- Tensile Fatigue, R =10
  - Ortho-polyester, 0° Dir, of $[0/\pm 45/0]_S$, $V_f = 0.36$
  - ~1 % weight gain

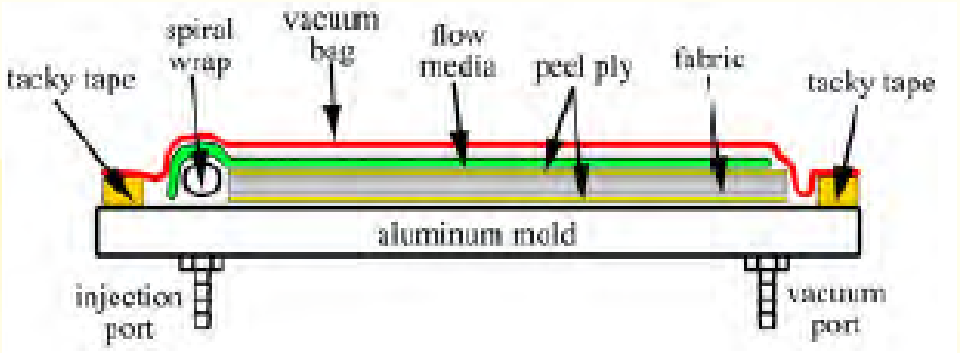


Water Absorption for Composites
MSU/SNL/DOE 2002 Report

- Summary
  - Hot/wet properties were better for iso-polyester and vinyl-ester system than otho-polyester or epoxy.
  - Vinyl-esters provided better toughness and structural integrity for slightly increased cost.
  - Epoxy systems middle of the class.
  - Fatigue strengths followed the quasi-static results in hot/wet conditions.

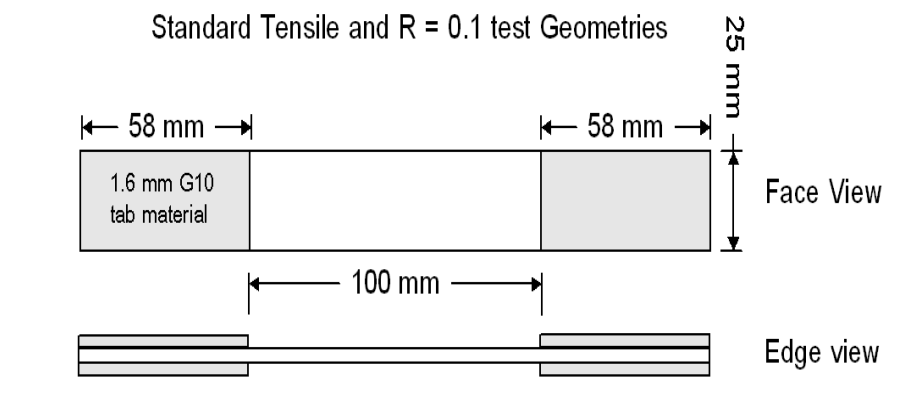
Current MHK Program

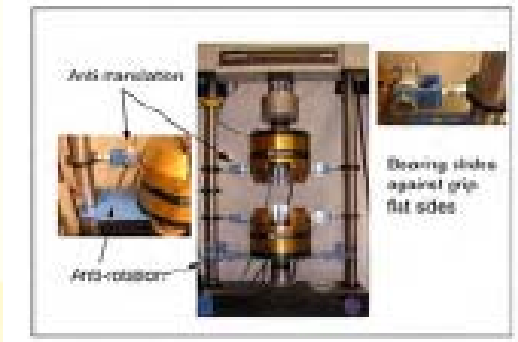
- Extend the knowledge to current wind material systems to understand exposure to salt water.
  - Fabricated Test Samples
    - 180 samples:
      - Resin: Momentive RIMR135/RIMH1366; UD glass fabric: PPG-Devold L1200/G30-E07
      - Processing: Infusion, curing at 20°C for 24 hrs followed by 80°C for 12 hrs; Layups: $[0]_2$, $[0]_6$, $[90]_6$, $[\pm 45]_6$
      - Saltwater Conditioned
    - 120 samples:
      - Resin : Hexion RIMR135/RIMH1366; UD glass fabric PPG-Devold L1200/G50-E07 glass fabric
      - Processing: Infusion, curing at 25°C for 24 hrs followed by 70°C,for 12 hrs; Layups: $[0]_2$, $[0]_6$, $[90]_6$
      - Salt Fog Conditioned at Sandia National Laboratory
    - 120 samples:
      - Resin: Ashland 601; UD glass fabric PPG-Devold L1200/G50-E07 glass fabric
      - Processing: Infusion, curing at 25°C for 24 hrs followed by 70°C,for 12 hrs; Layups: $[0]_2$, $[0]_6$, $[90]_6$, $[\pm 45]_6$
      - Saltwater Conditioned



Current MHK Program

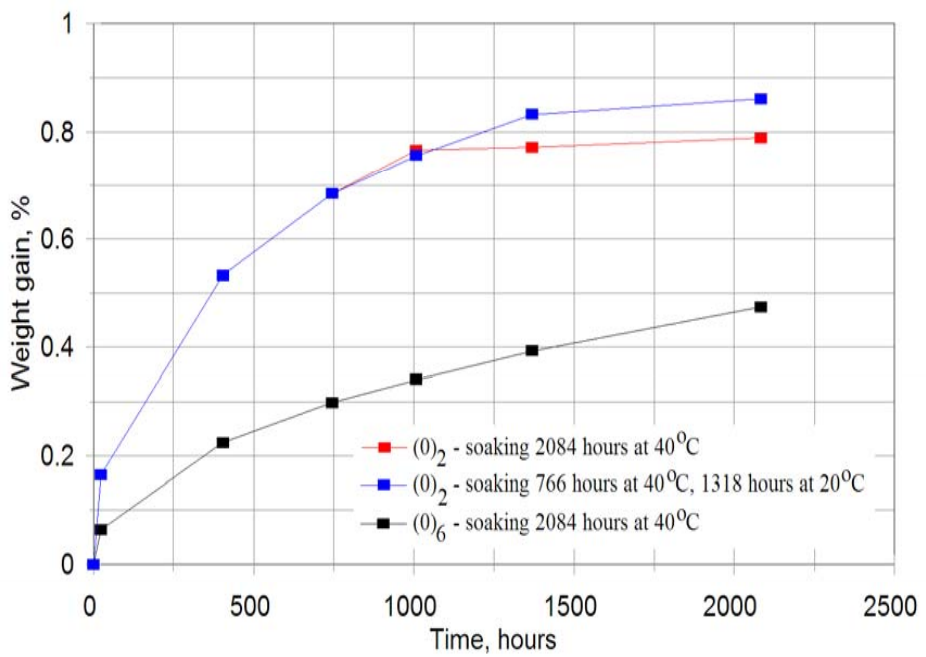
- Mechanical Testing:
  - Quasi-static testing at 0°(5°), 20° and 40° C, with and without environmental treatment.
  - Tensile testing of $[0]_2$ and $[90]_6$ layups by ASTM D3039 and compressive testing of $[0]_6$ and $[90]_6$ layups by ASTM D6641.

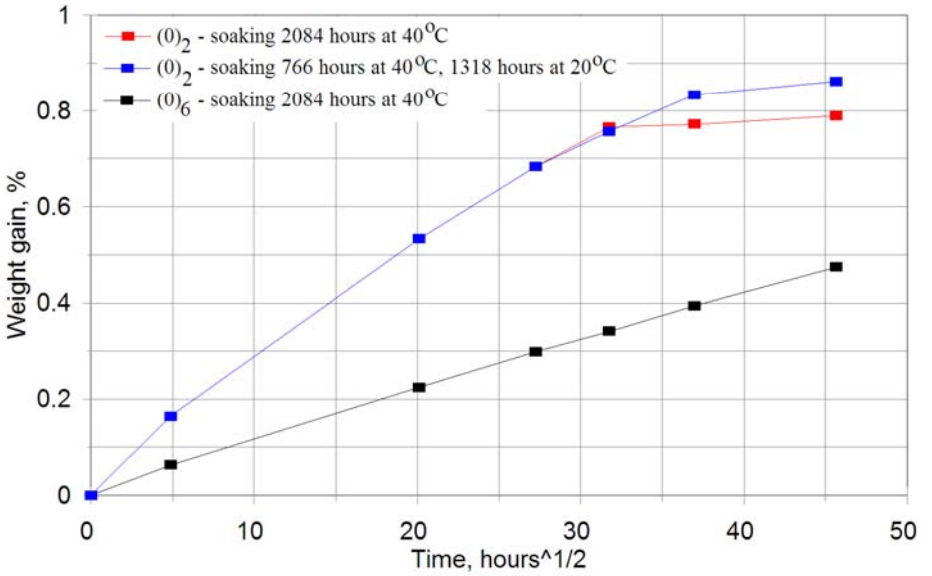




Initial Composite Moisture Absorption Trial – 40°C (Task2)

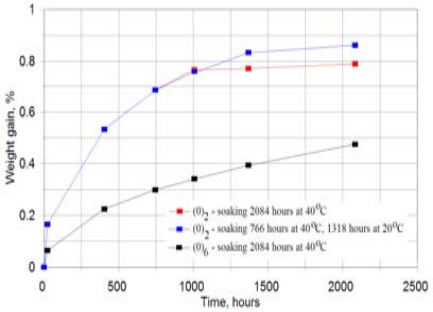
- $[0]_2$ samples are approaching an equilibrium moisture content at 1000 hours (41 days)
- thicker $[0]_6$ samples have lower moisture absorption
- Initial testing protocol was to remove sample after 1000 hrs at 40°C and store in SSW at 20°C until testing
- Weight gain trends as standard diffusion mechanism

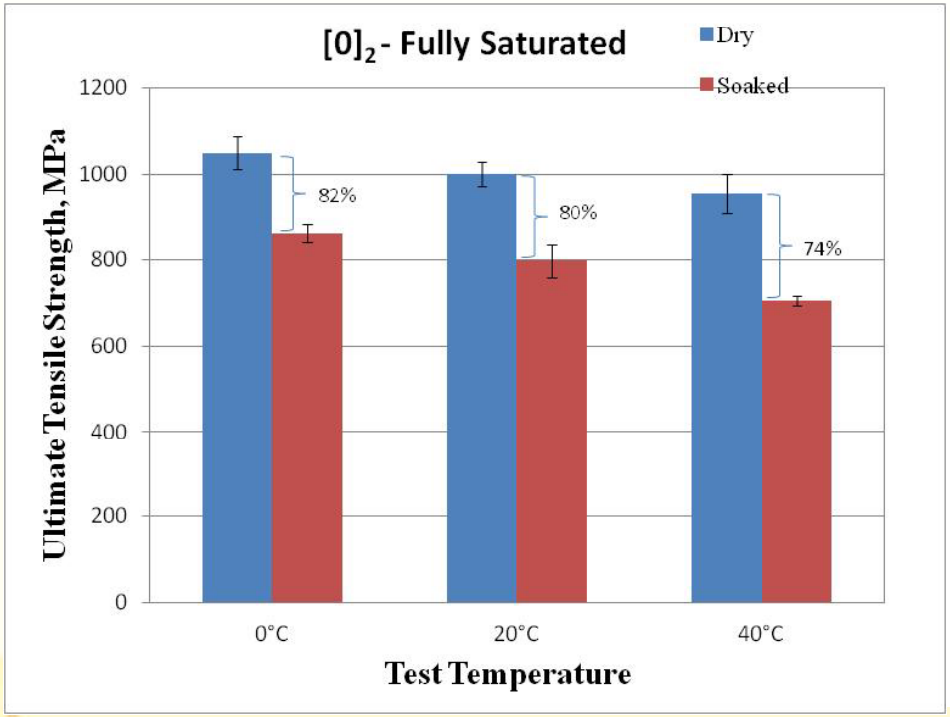


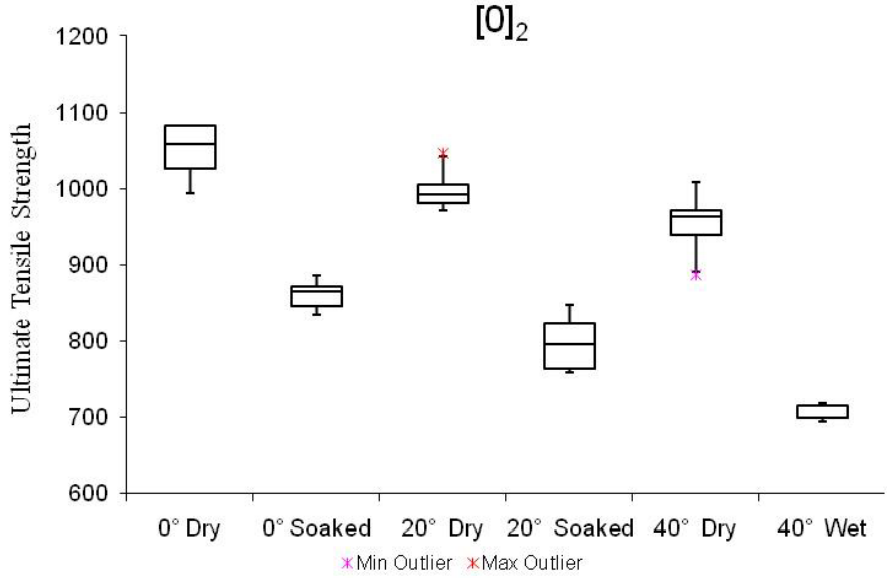


Tensile Mechanical Testing: longitudinal direction

- reduced tensile strength with increasing moisture and temperature
- Fully saturated with 0.86 % Wt. Gain, $V_f = 0.56$
  - Average values plotted with standard deviation
  - Box plot showing median, quartiles, max/min and outliers

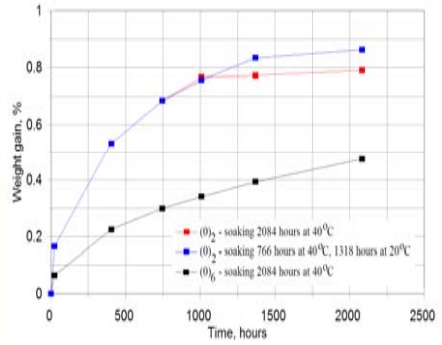


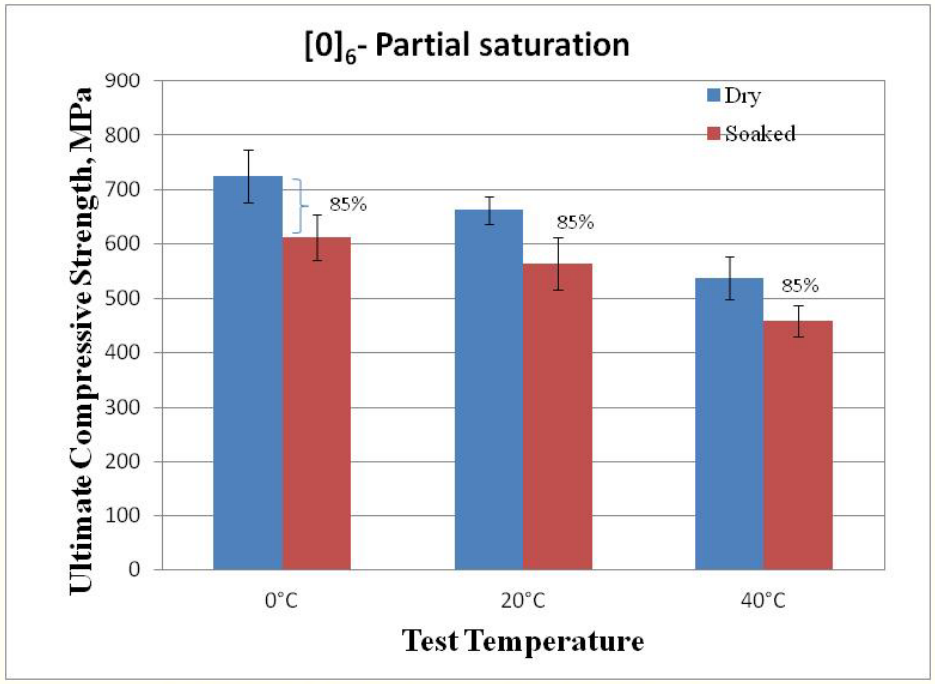


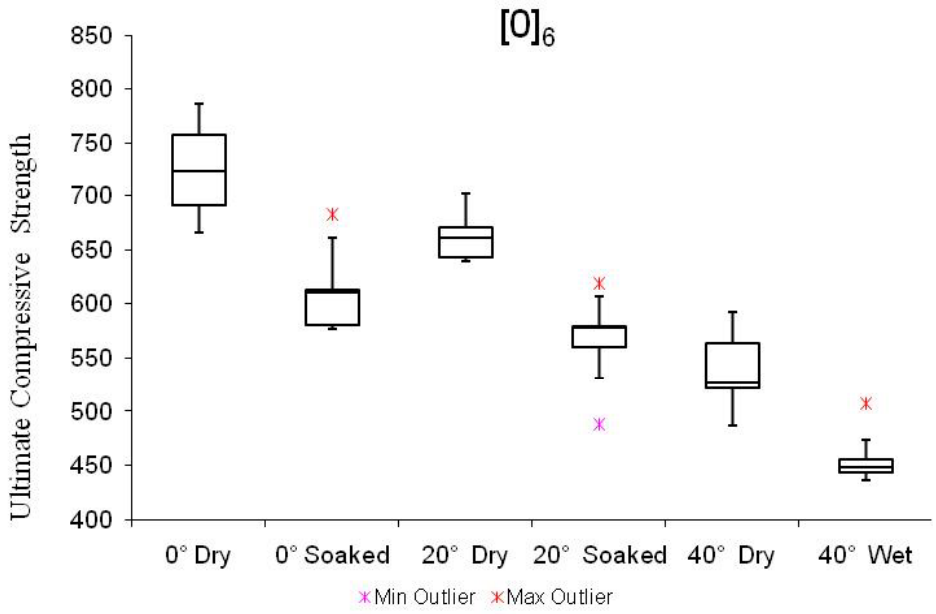


Compressive Mechanical Testing: longitudinal direction

- Reduced compressive strength with increasing moisture and temperature in the 0º (longitudinal) direction.
- NOT fully saturated with 0.47 % Wt. Gain, $V_f = 0.56$
  - Average values plotted with standard deviation
  - Box plot showing median, quartiles, max/min and outliers

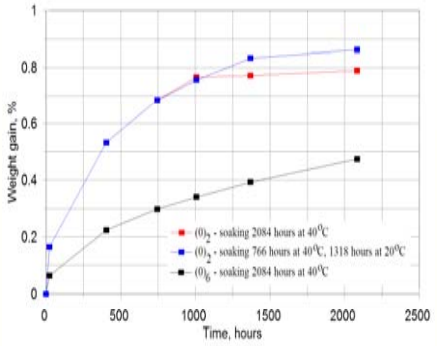


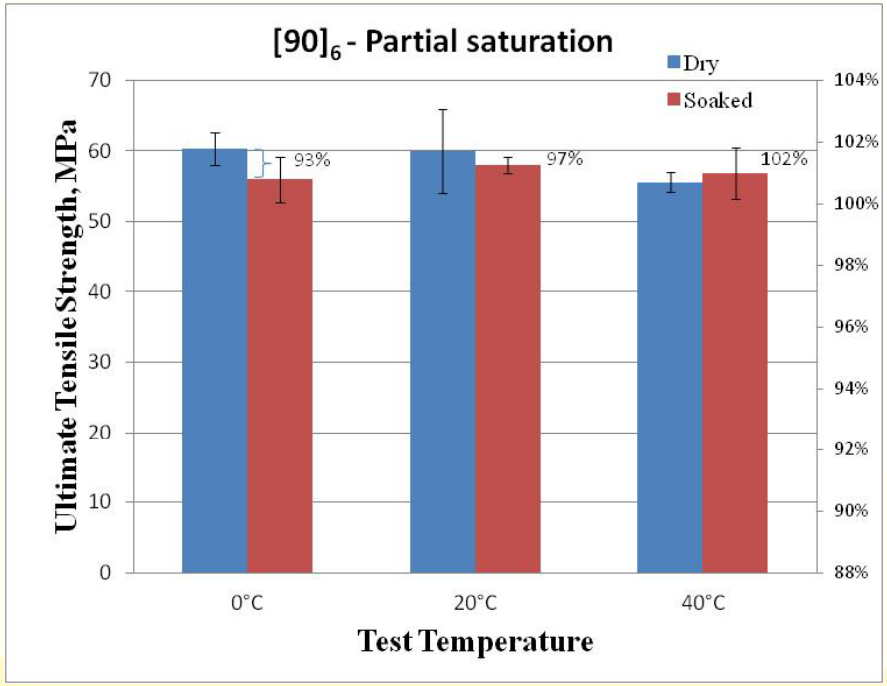


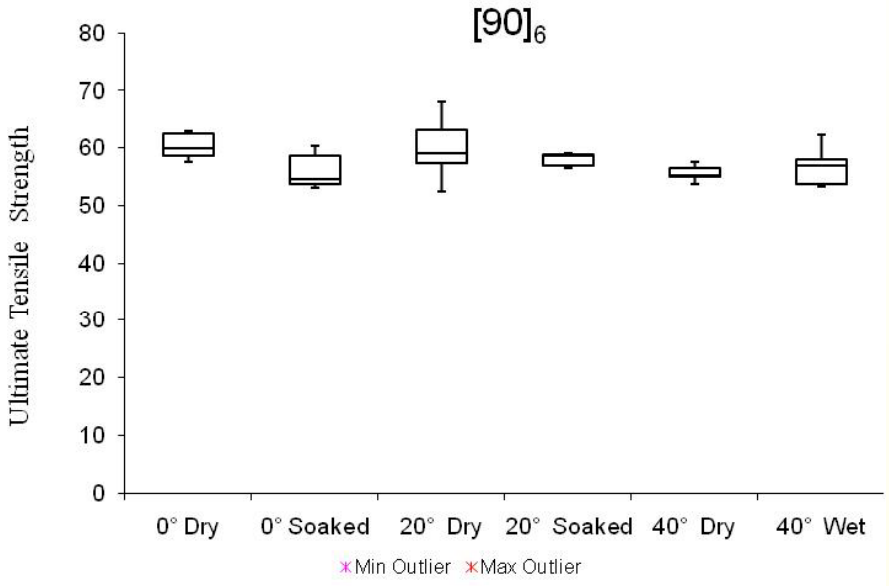


Tensile Mechanical Testing: transverse direction $[90]_6$

- Tensile strength is little affected as temperature and moisture increase
- Not fully saturated with 0.47 % Wt. Gain, $V_f = 0.57$
- Average values plotted with standard deviation
- Box plot showing median, quartiles, max/min and outliers

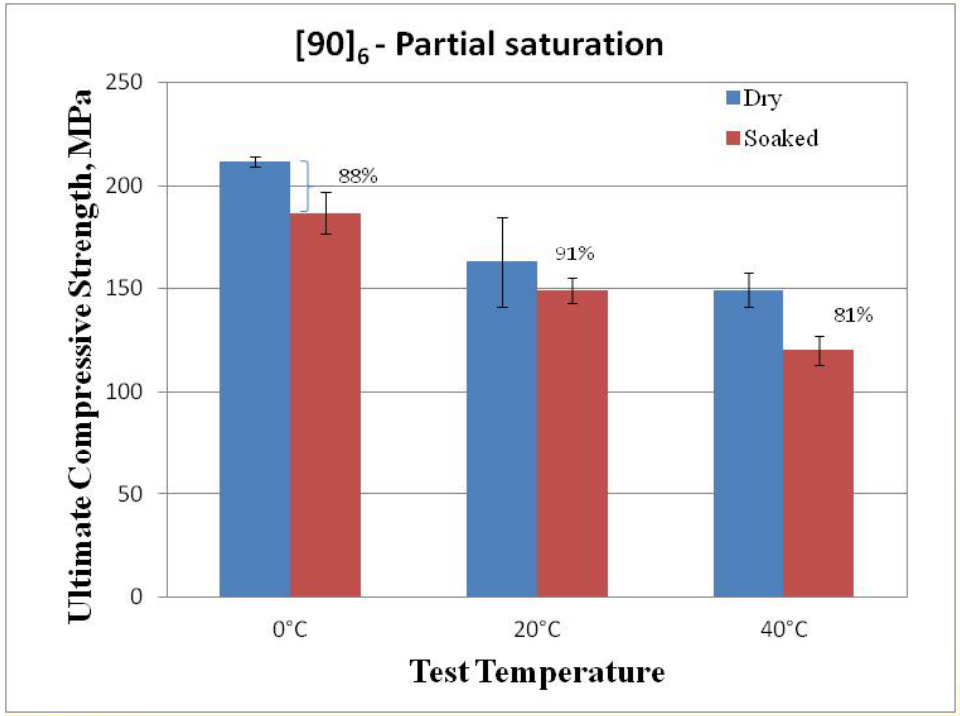


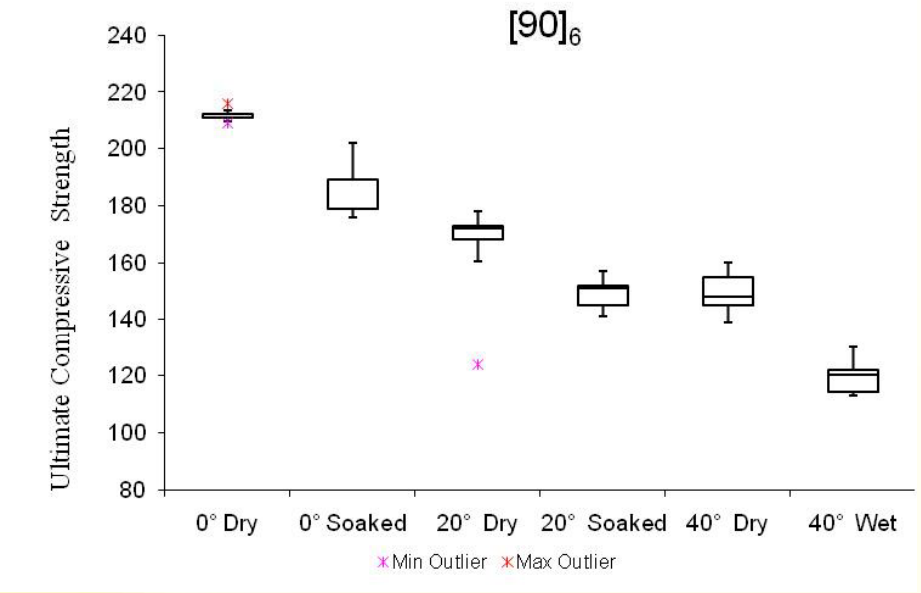




Compressive Mechanical Testing: transverse direction $[90]_6$

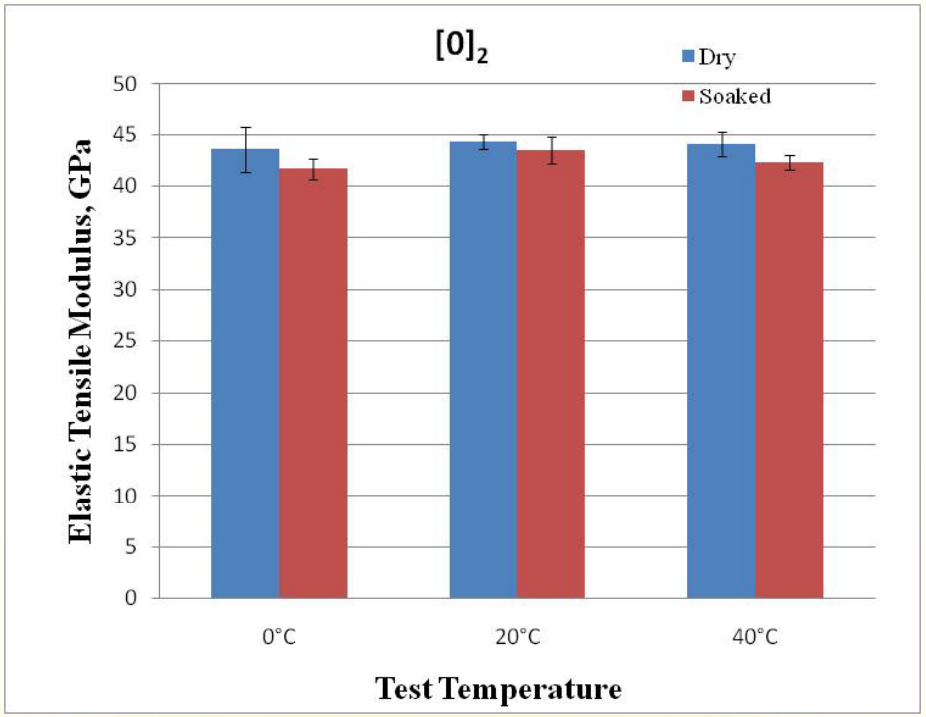
- decreased compression strength as temperature and moisture increase
- Not fully saturated with 0.47 % Wt. Gain, $V_f = 0.57$
  - Average values plotted with standard deviation
  - Box plot showing median, quartiles, max/min and outliers

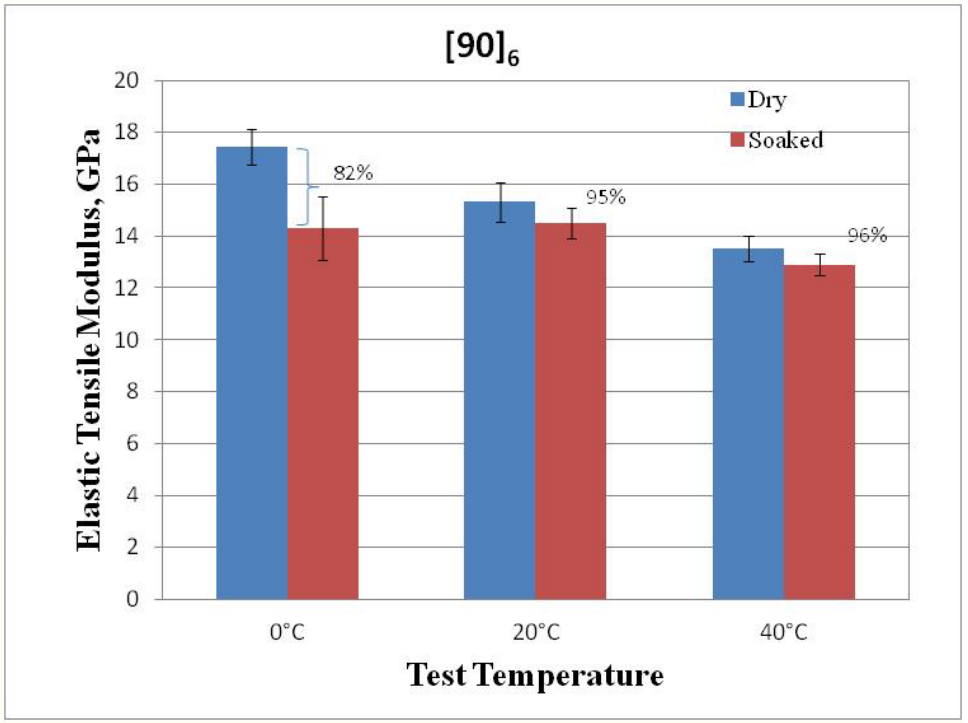




Mechanical Testing

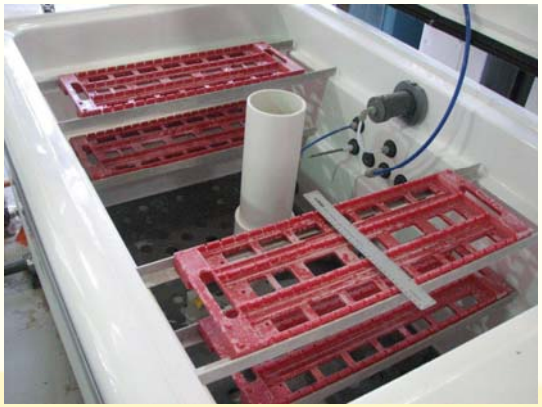
- elastic modulus (E) is little affected in the longitudinal direction but slightly decreases in the transverse direction.
- Not fully saturated with 0.47 % Wt. Gain, $V_f = 0.57$
- Average values plotted with standard deviation



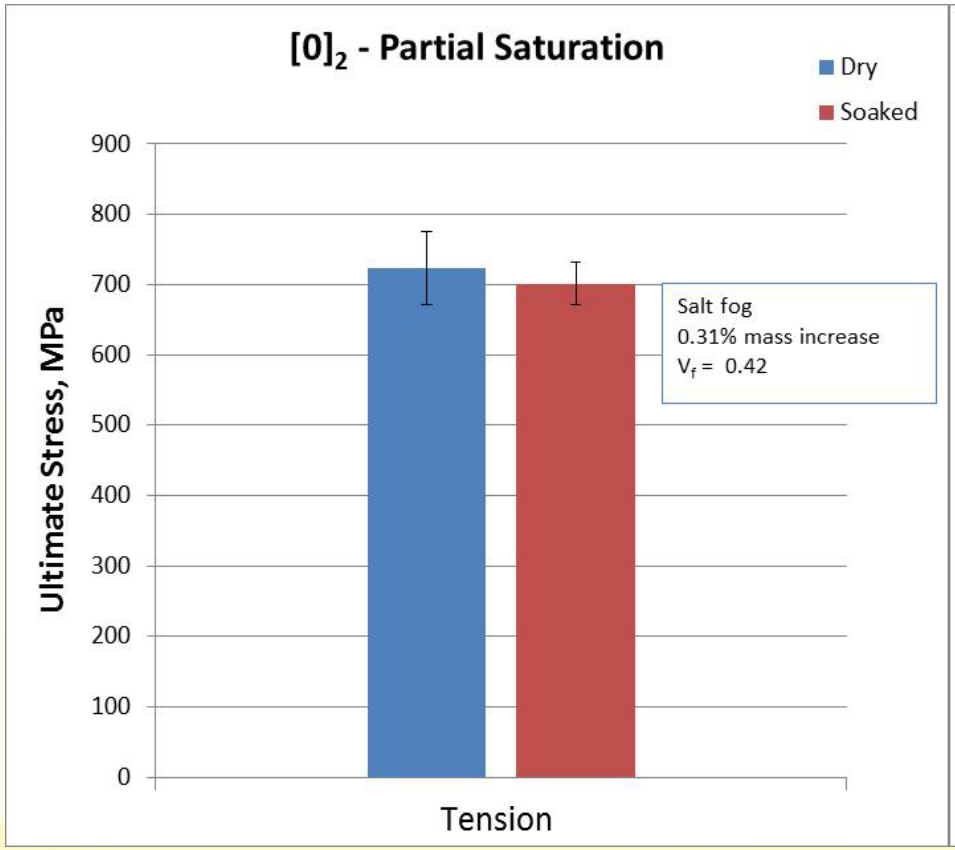


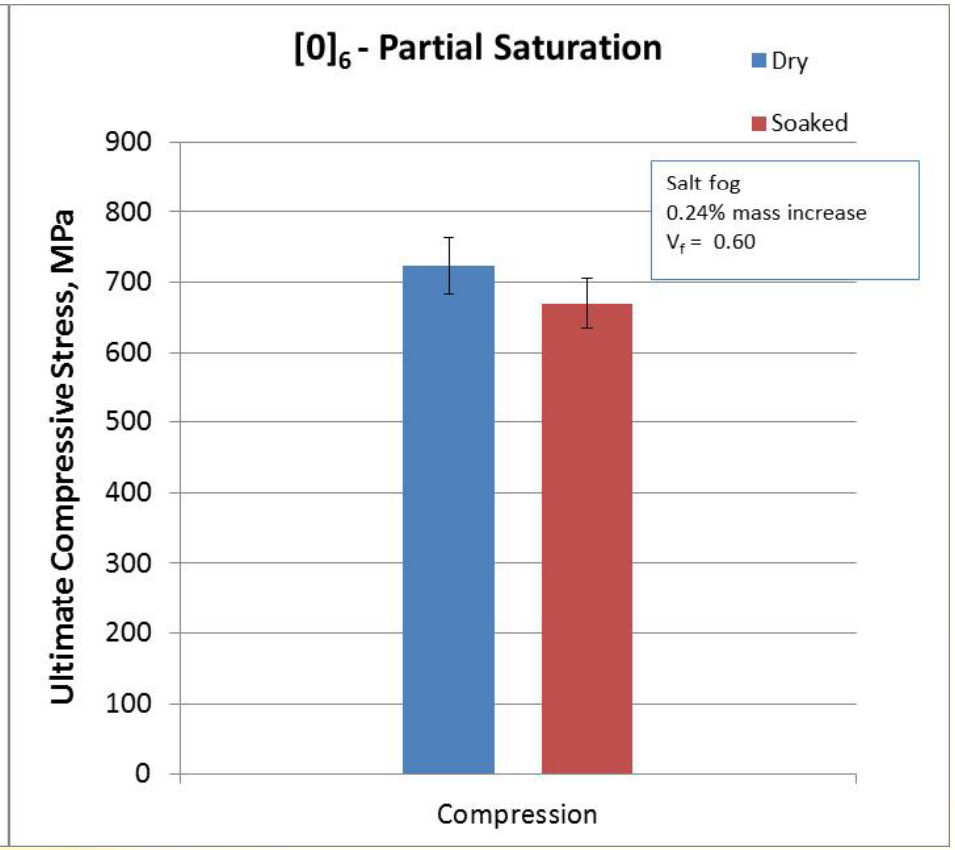
Salt Fog Immersion Tests

- Samples
  - Resin: Momentive RIMR135/RIMH1366; UD glass fabric: PPG-Devold L1200/G50-E07
  - Processing: Infusion, curing at 25°C/24 hrs followed by 80°C/12 hrs, fiber volume content 43% for $[0]_2$ 60% for $[0]_6$, $[90]_6$.
- Environmental Conditioning (at SNL, Todd Griffith)
  - Salt Fog Apparatus per ASTM standard B117-03
  - 4 week soak at 40°C
  - Final %'s only, and small at that!!!

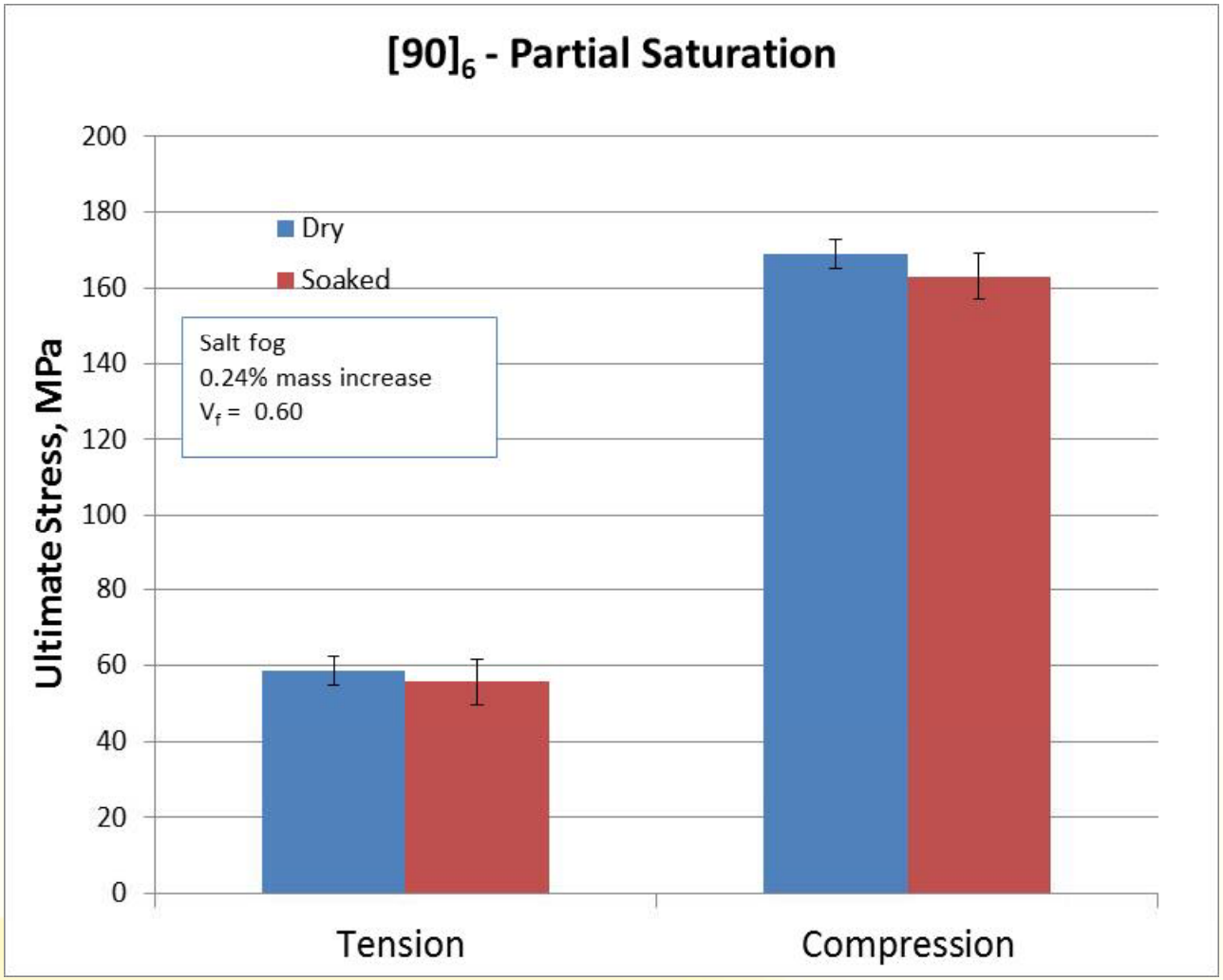


Salt Fog Results: Longitudinal





Salt Fog Results: Transverse



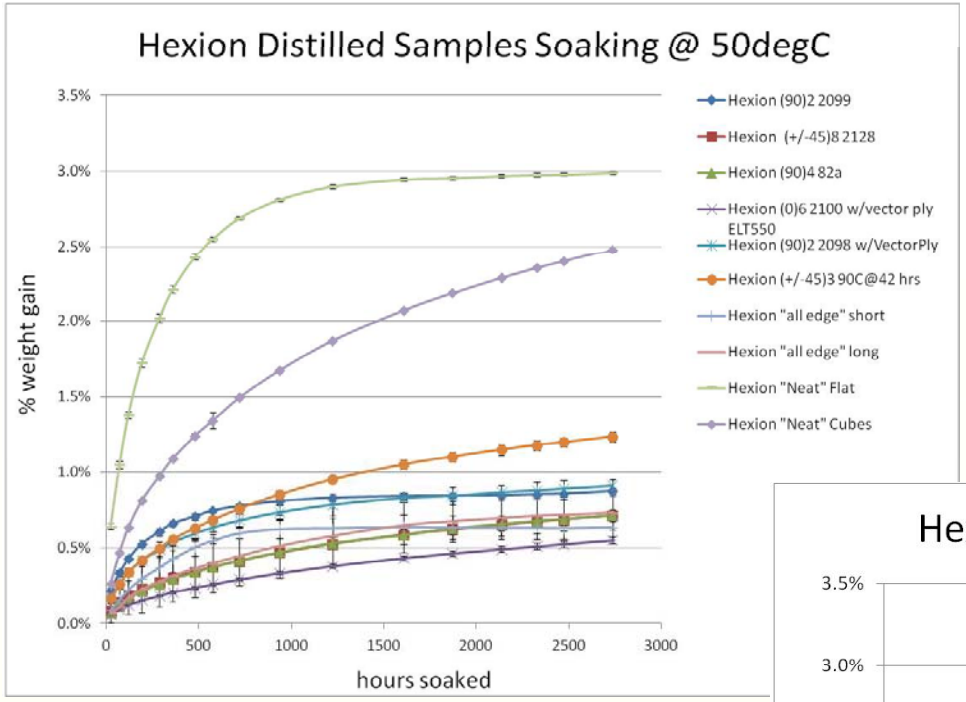
Latest Immersion Tests

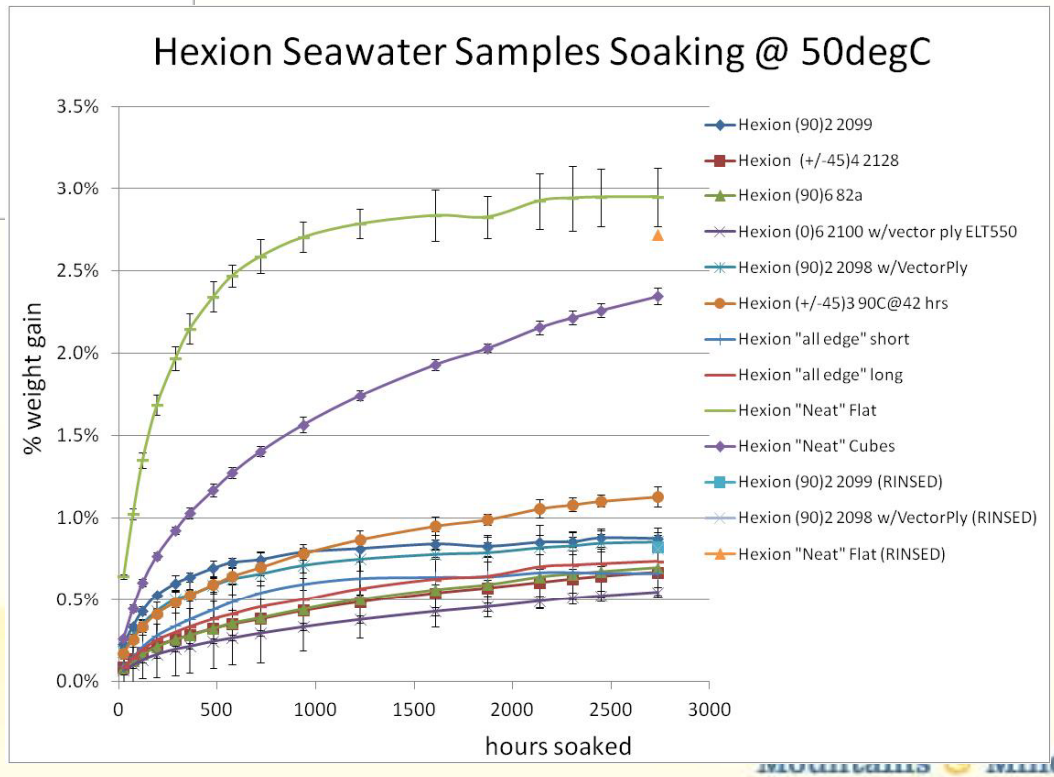
- Added multiple directions and neat material
  - Distilled water AND Saltwater Conditioned at 50°C
  - Goal to fully saturate material

  - Resin: Vinyl Ester Ashland 601; UD glass fabric PPG-Devold L1200/G50-E07 glass fabric
  - Processing: Infusion, curing at 25°C for 24 hrs followed by 70°C,for 12 hrs; Layups: $[0]_2$, $[0]_6$, $[90]_6$, $[\pm 45]_6$

  - Resin: Momentive RIMR135/RIMH1366; UD glass fabric: PPG-Devold L1200/G30-E07
  - Processing: Infusion, curing at 20°C for 24 hrs followed by 80°C for 12 hrs; Layups: $[0]_2$, $[0]_6$, $[90]_6$, $[\pm 45]_6$
- Testing
  - Quasi-static and fatigue at fully conditioned state.

Current Immersion Tests: epoxy





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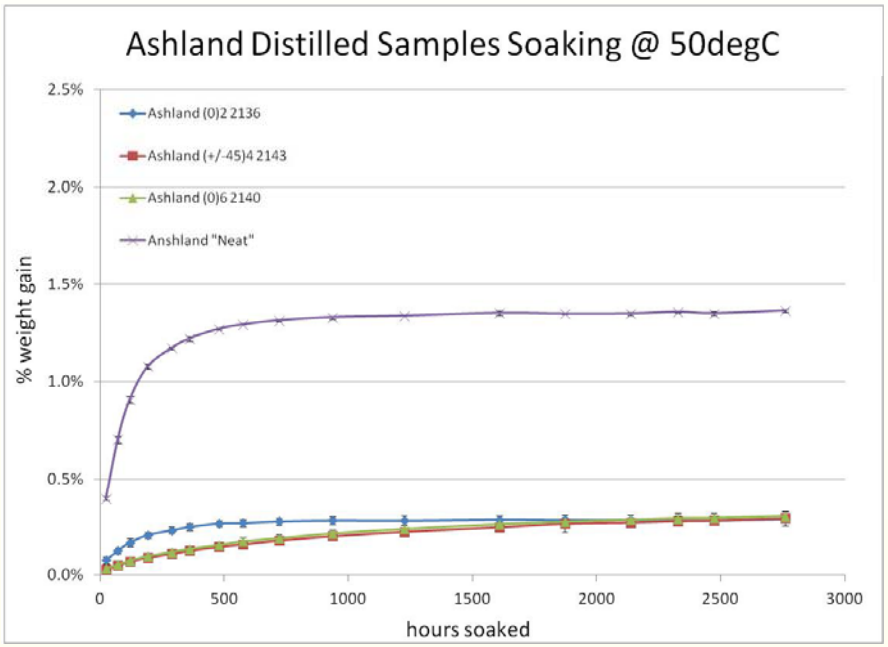
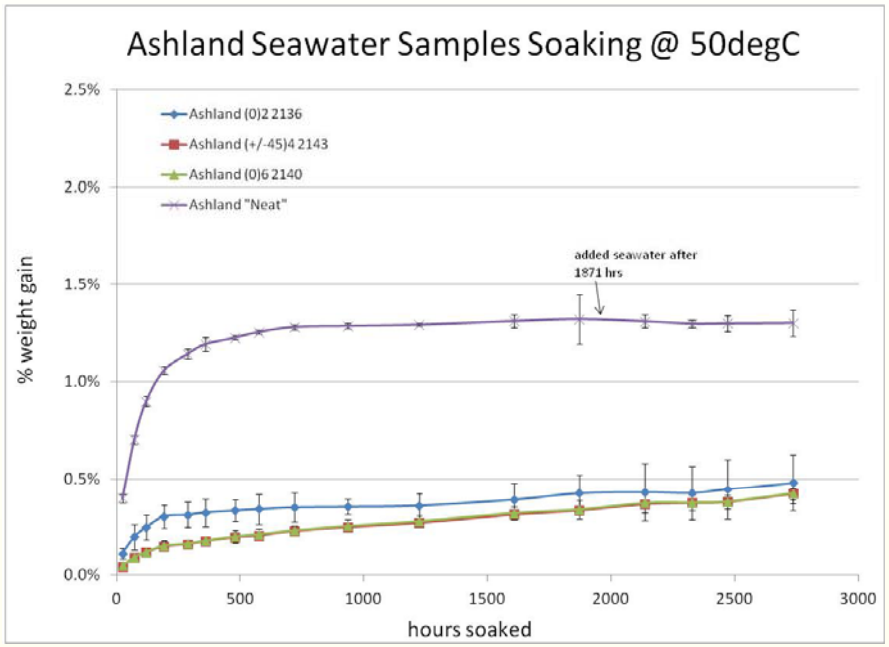
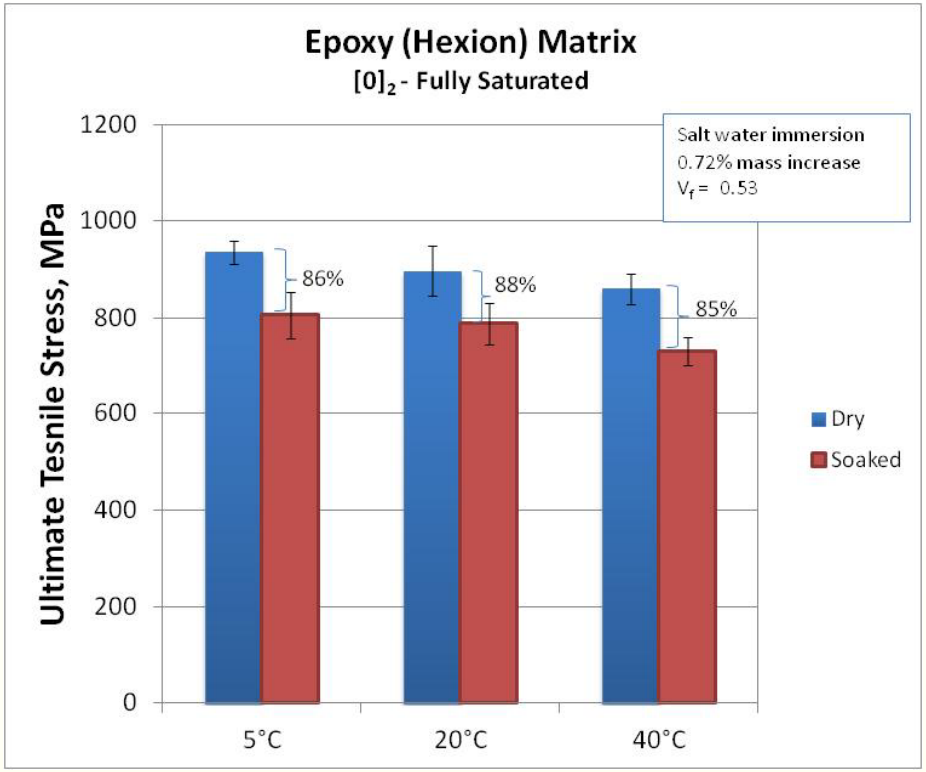
Current Immersion Tests: Vinyl-ester

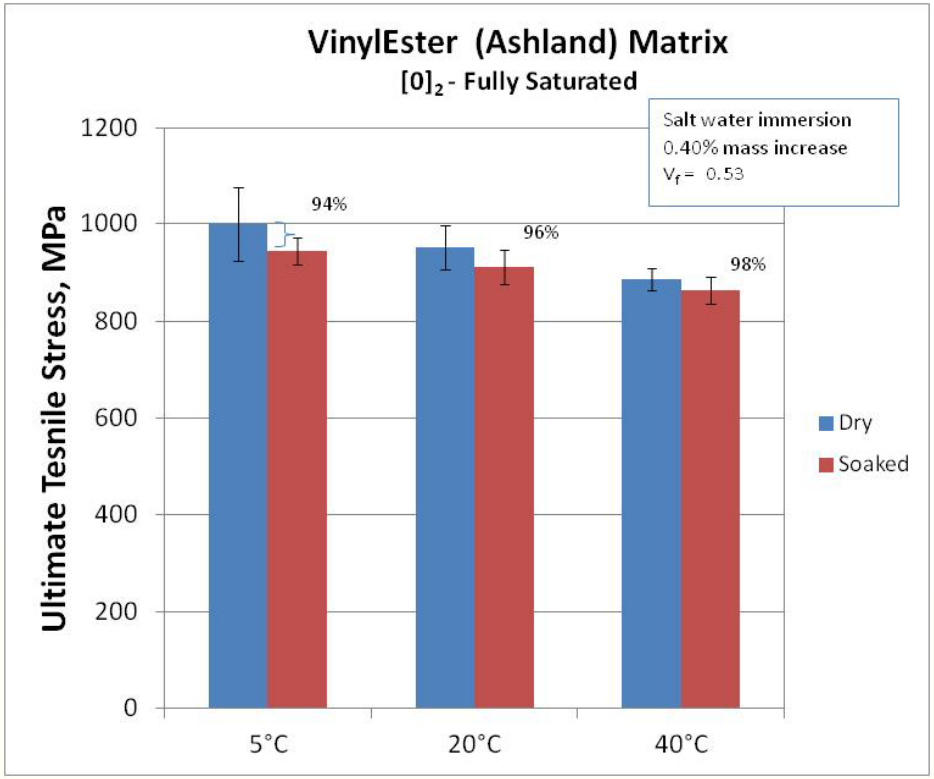
– Virtually no difference between water content, distilled vs seawater





• Tensile Mechanical Testing: longitudinal direction

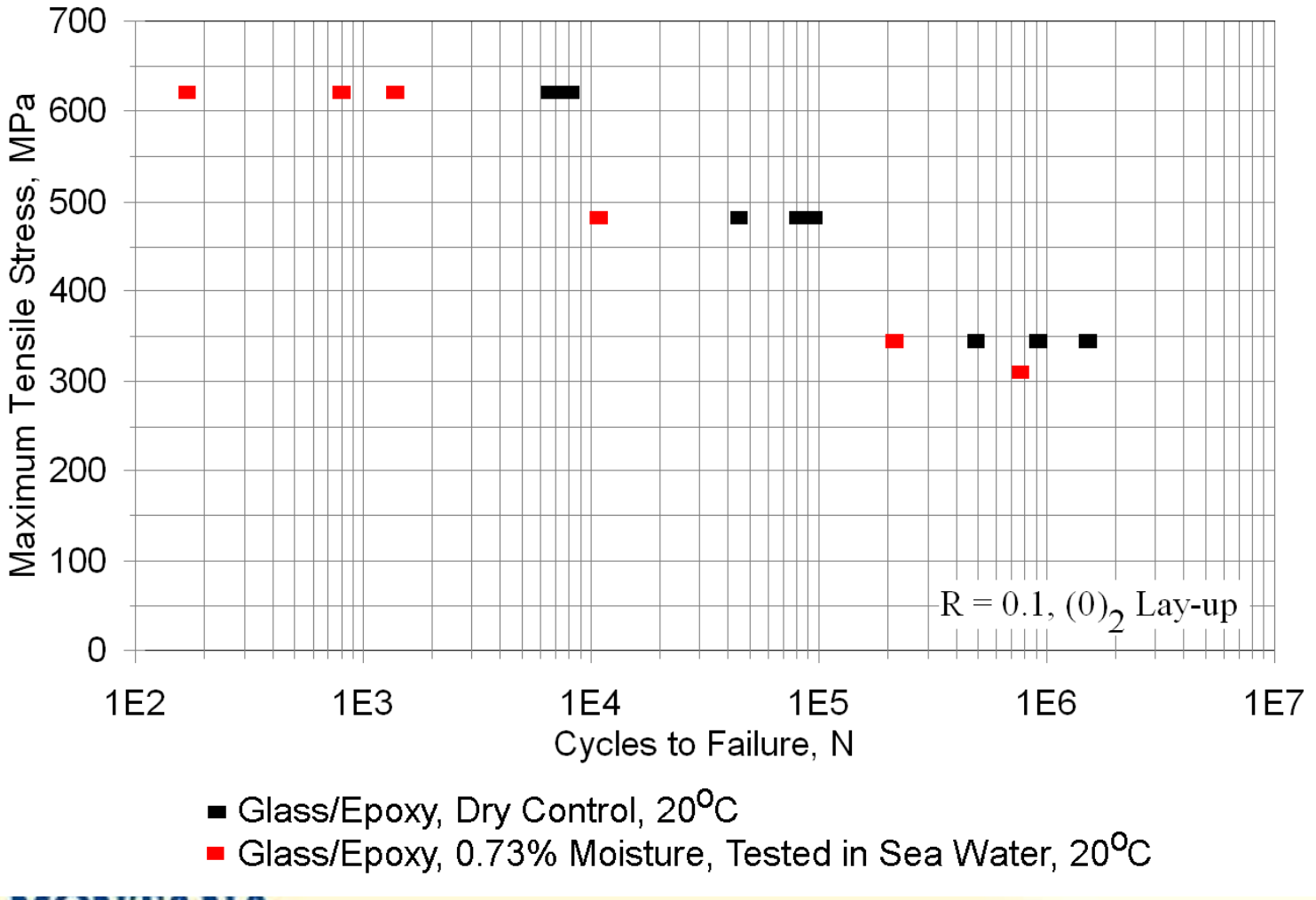




- reduced tensile strength with increasing moisture and temperature
- Fully saturated with 0.72 % Wt. Gain, $V_f = 0.54\text{-}0.55$

- Little reduction of tensile strength with increasing moisture
- Fully saturated with 0.40% Wt. Gain, $V_f = 0.53\text{-}0.55$

- Tensile Fatigue: longitudinal direction



Conclusions and Future Work

- Reduction seen in static tensile strength at full saturation
- Reduction seen in static compression strength at partial saturation
- Start including ….
  - Testing in Fatigue and off-axis directions (in progress!!)
  - Include adhesives and structural components (in progress!!)
  - Include coatings (under investigation)
  - Moisture induced expansion measurements (under investigation)
  - Recent agreements have been made between MSU and Hexcel and Gurit to test provided carbon and glass pre-preg laminates.
  - Material Transitions
- Goal to include (second order tasks)….
  - Modeling…. Need moisture content at location (In Progress)
  - Decrease in properties as a function of moisture content (not at equilibrium).
  - Looking at potential of NDT techniques, e.g. acoustic emission, to identify location of damage.