CHEMICAL DEPAINT OF COMPOSITES:
Summary of Phases 1-3 Work and Final Phase 4 Work
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Outline

- Overview of Chemical Depaint of Composites
- Phase 1 Findings
- Phase 2 Findings
- Phase 3 Findings
- Phase 4 Work – Plans and Current Results

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Overview of Chemical Depaint of Composites

- **Funding provided by AFLCMC/ACO (Advanced Composites Office)**
  - **Funding under EPSE IV Contract – various task orders**
- **Understanding the effects of chemical depaint on composites was initiated by ACO in early 2019.**
  - **Phase 1 (2019-2020) focused on effects on pure resin materials.**
    - **RT Repair Epoxy (EA9396), 250°F Epoxy (7714A), 350°F Epoxy (977-3)**
  - **Phase 2 (2020-2022) focused on effects on lamina (unidirectional) composites**
    - **Studies on 250°F Epoxy (7714A), 350°F Epoxy (977-3) with carbon fiber.**
  - **Phase 3 (2022-2023) focused on quasi-isotropic fiber reinforced laminates with epoxy and an unreinforced high temperature resin (bismaleimide – BMI)**
    - **Studies on 250°F Epoxy (7714A), 350°F Epoxy (977-3) with carbon fiber, BMI (5250-4) pure resin.**
  - **Phase 4 (2023-2024) focuses on epoxy + fiberglass systems and a reinforced BMI.**
    - **Studies on two common repair epoxies (Hexcel 155, Hexcel 161) with 7781 fiberglass, and studying 5250-4 + carbon fiber (IM7 unidirectional).**

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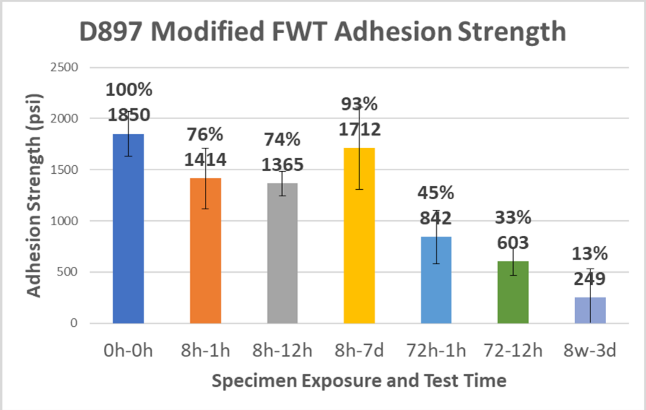
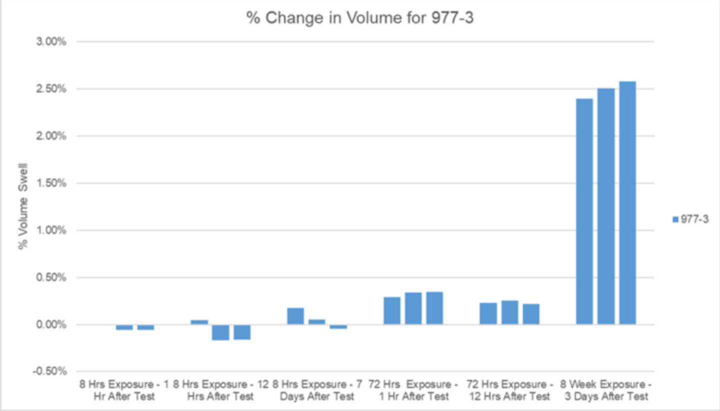
Phase 1 Findings

- Focused study on chemical stripper (B&B Tritech 9095) effects on pure resin materials.
  - Repair epoxy (room temperature cure, EA9396)
  - 250°F use temperature epoxy (7714A)
  - 350°F use temperature epoxy (977-3)
- Conduct mechanical and chemical analysis after chemical stripper exposure on resins with no carbon fiber reinforcement.
- Samples fully immersed in chemical stripper for various times.
  - 8 hour, 72 hour, 4-8 weeks

Phase 1 Findings

- B&B Tritech 9095 Chemical Stripper, while relatively benign for stripping metal substrates, has some effects on aerospace grade epoxies.
  - Significant degradation of thermal, chemical, and mechanical properties with EA9396 epoxy, even after 8 hour exposure.
  - Decomposition of EA9396 epoxy (chemical incompatibility) with 72 hour or longer exposure times. Severe decomposition at 8 weeks.
  - Some minor effects on thermal, chemical, and mechanical properties with 7714A and 977-3 epoxies after 8 hours, more notable effects after 72 hours. Severe degradation of properties with prolonged exposure (4-8 weeks).
  - Ranking of chemical stripper “resistance”:
    - 977-3 > 7714A > EA9396
- Phase 1 reports available through ACO.





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Phase 2 Test Plan Review

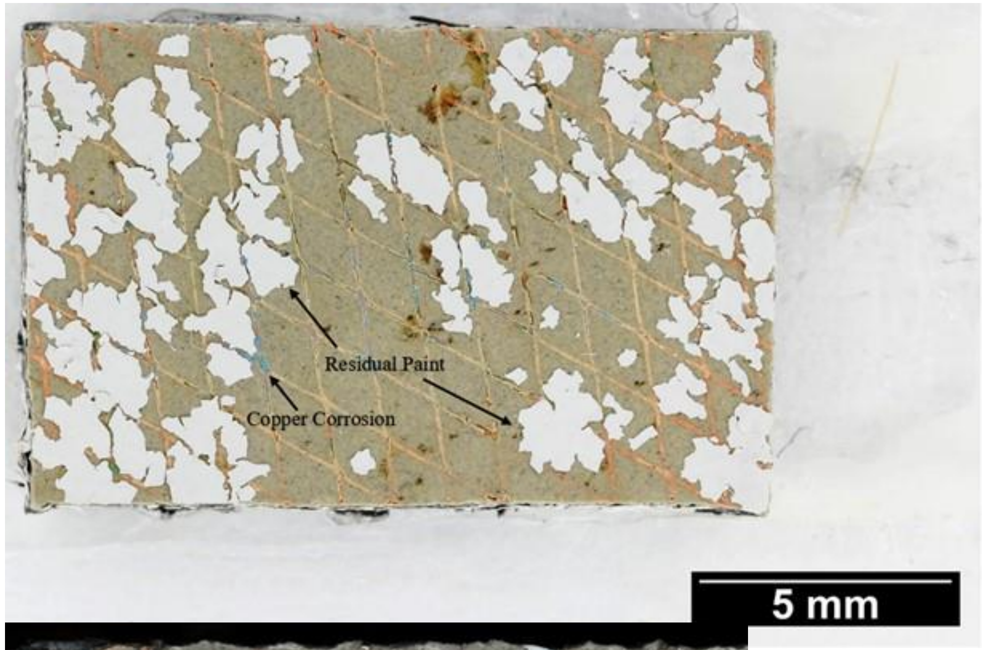
- Phase 1 results guided Phase 2 Test Plan development
- Focus on lamina samples (977-3/AS4, 7714A/T300)
- Chemical Stripper exposure times limited to 8 hour and 72 hour exposures, with exposures only on one side of the lamina (not complete immersion/exposure as done in Phase 1)
  - 8 Hour exposure, 16 hour rest, then test
  - 8 Hour exposure, 7 day rest, then test
  - 72 Hour exposure, 24 hour rest, then test
  - 72 Hour exposure, 7 day rest, then test
- Chemical stripper used was B&B Tritech 9095, same as Phase 1.
  - Active ingredients in chemical stripper: benzyl alcohol, hydrogen peroxide.

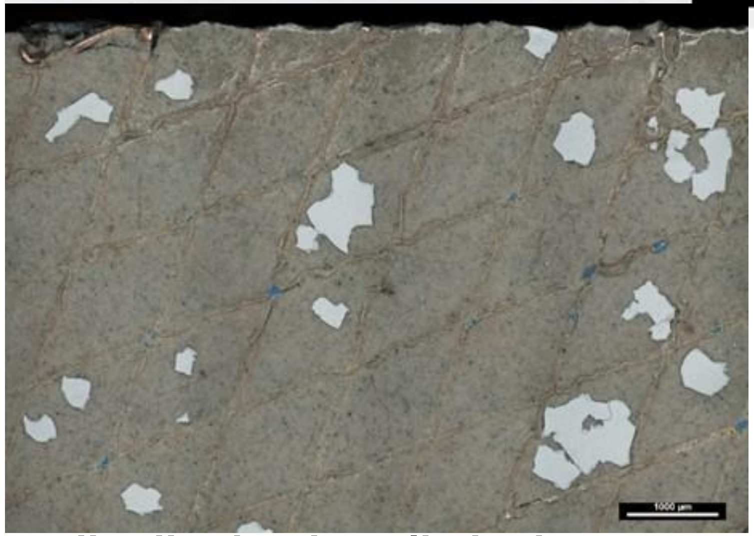
Phase 2 Findings

- The effects of the chemical stripper on epoxy + carbon fiber composite mechanical properties are less severe than the effects on unreinforced neat resin.
  - Results apply to both 977-3 and 7714A epoxy materials.
  - Mechanical properties either mostly unchanged vs. control, or slightly changed depending upon the test method.
    - Fatigue testing results inconclusive – more work needed.
  - Repair still seems possible after chemical depaint, but more work needed to validate this.
  - Thermal properties (glass transition temperature) do seem to take a permanent reduction in properties after chemical stripper exposure.
    - Property loss is more severe in 977-3 than in 7714A.

Phase 2 Findings

- While chemical stripper does absorb into the composite, <u>it can be removed with long drying times and temperature.</u>
  - Seems to restore / improve bonding after being forced out of a pure epoxy material.
  - Material is hard to get out of the composite.
- Real-world materials with multiple layers of paint and copper meshes (for lightning strike protection) show some interesting effects in the presence of the chemical stripper.
  - Paint scraper absorbs the chemical stripper.
  - The copper mesh corrodes when exposed to chemical stripper.





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Phase 3 Plans

- **Phase 3 test plan approved in February 2022 and was completed by May of 2023.**
- **Mechanical, Thermal, and Chemical Analysis Testing on carbon fiber laminate samples of epoxy, and on pure resin specimens of BMI.**
  - **977-3/AS4 and 7714A/T300 Epoxy + Carbon Fiber Laminates**
  - **5250-4 BMI Neat Resin**
- **Reports available through AFLCMC/EZPT-ACO.**
- **Builds off Phase 2 test plan, and studies effects of 8 and 72 hour exposures. 8 Week exposures for BMI resin. Effects of Hot/Wet testing on properties also studied.**
- **Chemical stripper used was B&B Tritech 9095, same as Phase 1.**
  - **Active ingredients in chemical stripper: benzyl alcohol, hydrogen peroxide.**

Phase 3 Conclusions

- Water results in greatest reduction in properties for composites.
- Water is present in chemical stripper, and chemical stripper does appear to "seal in" some of this water, making it difficult to get back out.
- For thick composites, little effect from the chemical stripper on properties. Minor reduction in use temperature (T_g) noted.
- BMI resin is quite resistant to both water and chemical stripper up to 72 hour exposures.
  - For 8 week exposures – some drops in properties noted.

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- **Phase 4 Work – Plans and Current Results**

Phase 4 Testing

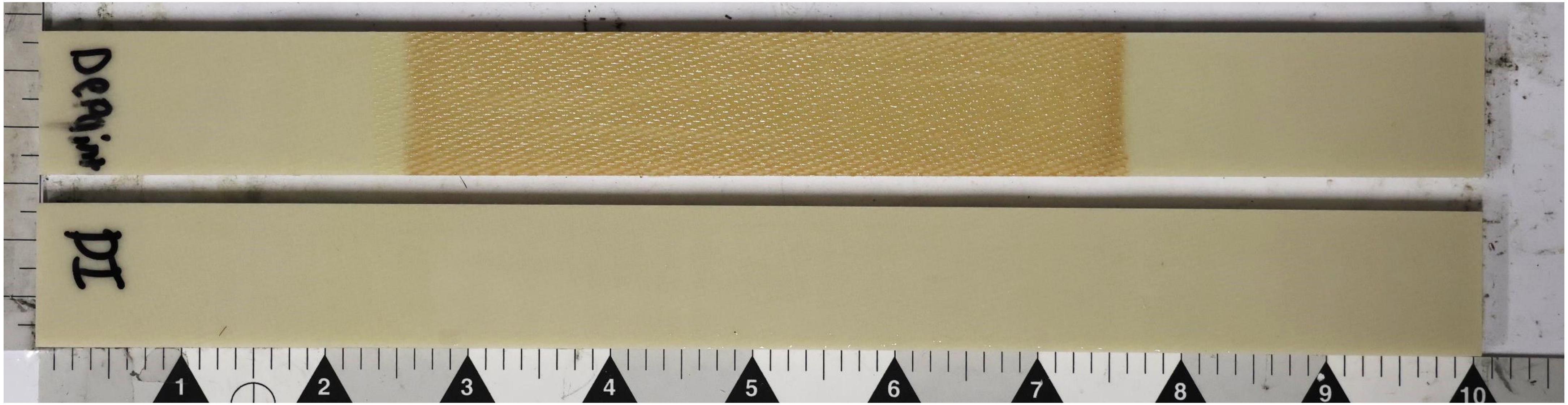
- Built off Phases 1-3, but focuses on:
  - Hexcel 155 epoxy / 7781 fiberglass
  - Hexcel 161 epoxy / 7781 fiberglass
  - 5250-4 BMI / IM7 carbon fiber
  - Hexcel 155 epoxy (to understand effects on resin only – helps to separate out effects of paint stripper on resin only – no fiber effects)
    - Unable to obtain Hexcel 161 epoxy without fiberglass (product is not sold)
- Same chemical stripper, same exposure times, same studies of hot-wet effects on properties.
- Goal is to verify that fiberglass doesn't show different / worse effects and that BMI + Carbon fiber shows ability to resist chemical paint stripper as it showed when in pure resin form.
- Two probe experiments: development of water detection technique when carbon fiber present, and use of femtosecond laser to see if it can dry the composite surface.

Phase 4 Results: Hexcel 155 Epoxy

- Currently Ongoing
  - Same test types as Phase 1 and used on the BMI neat resin testing in phase 3
    - Tension, 3pt flex, Compression, IZOD Impact, Shore D Hardness, Flatwise Adhesion, Dynamic Volume Swell, T_g via DMA, TGA, Infrared Spectroscopy
  - Results not available at this time
- Testing will provide insight to the behavior of the matrix separate from the fiber reinforcement
  - Comparable to a worst-case scenario
- Reports will be available through AFLCMC/EZPT-ACO when complete

Phase 4 Results: Hexcel 155 Epoxy Optical Images after Exposure

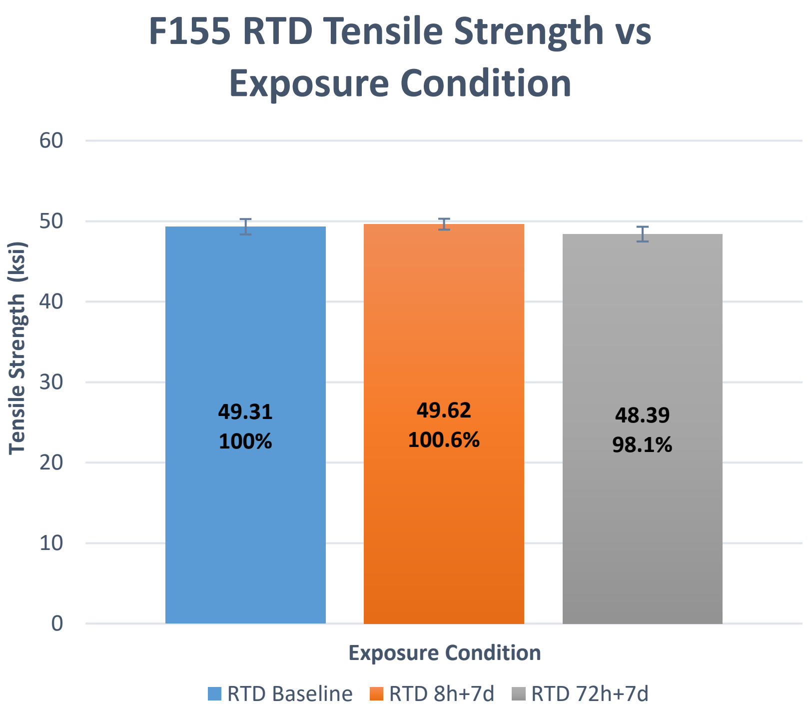
- During chemical exposures on fiber reinforced specimens, discoloration and a texture change of the material was noticed
  - Changes were much more rapid on humidity conditioned specimens
- One specimen was exposed to the chemical for an extended period, and one was exposed to deionized water for the same period of time
- Image below is after 17 days of exposure

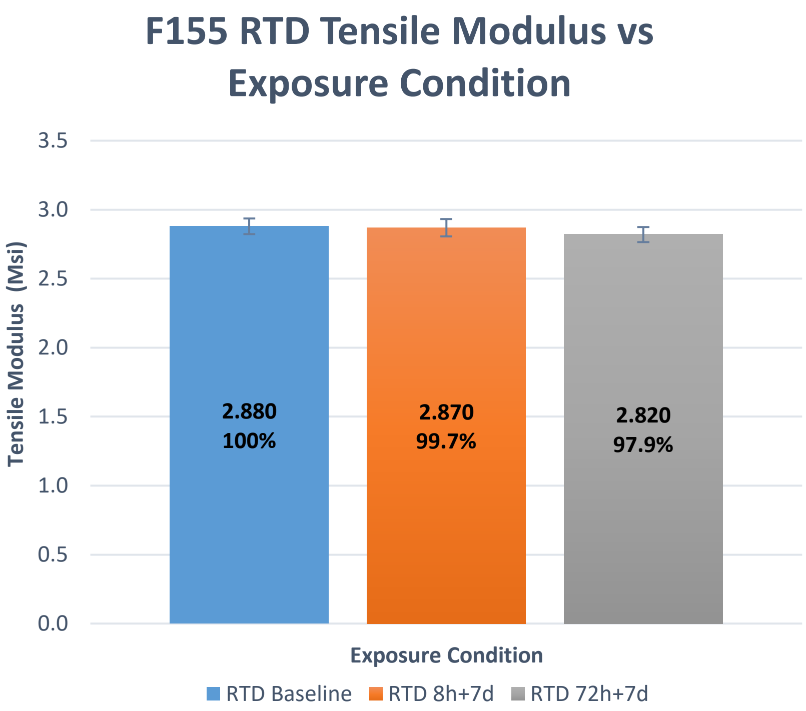


Phase 4 Results: Hexcel 155 + Fiberglass

- **Mechanical Testing**
  - Tension (RTD Only)
    - Strength and Modulus
  - In Plane Shear (RTD and ETW)
    - Shear Strength, Yield Strength and Shear Modulus
  - Open Hole Compression (RTD and ETW)
    - Open Hole Compression Strength
  - Flexure Fatigue (RTA)
    - Cycles to Failure and 3 different stress levels
  - Dynamic Mechanical Analysis (RTD and ETW)
    - T_g

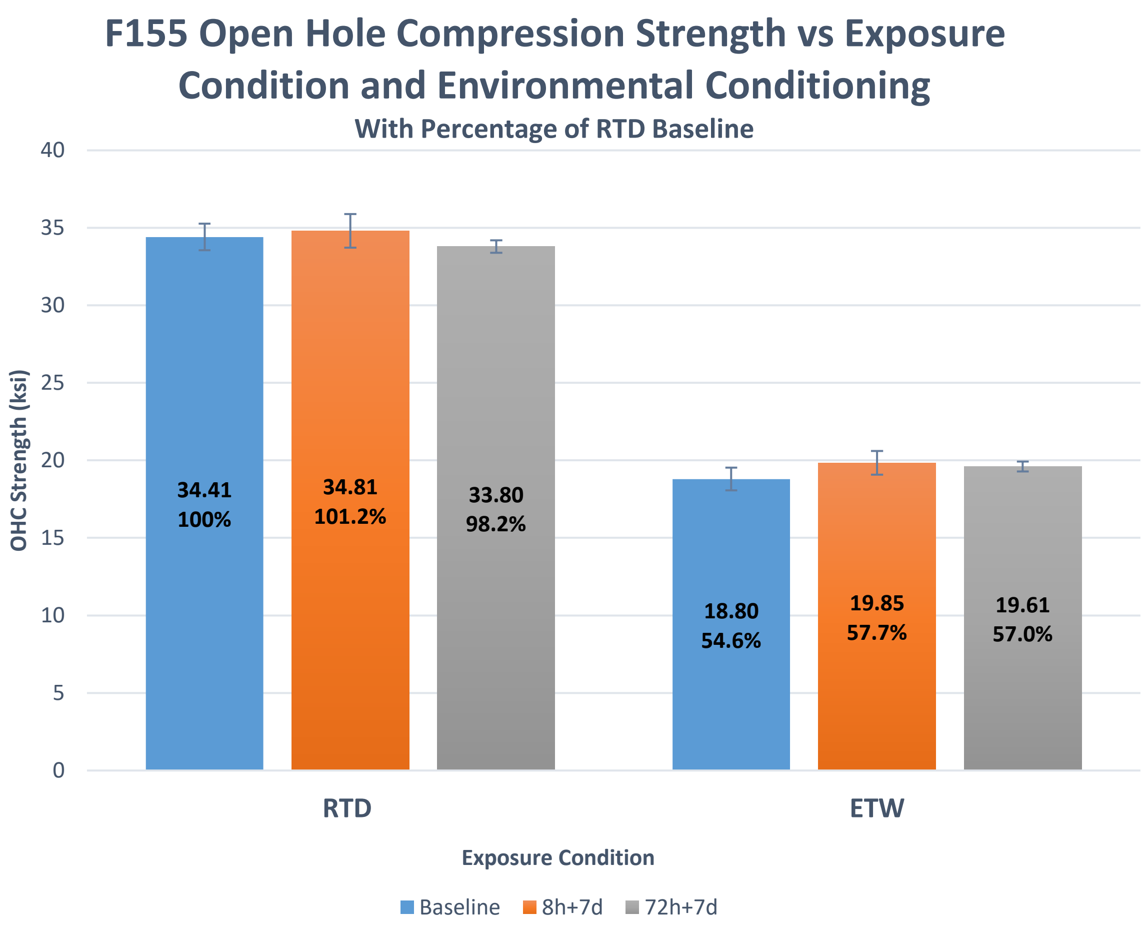
Phase 4 Results: Hexcel 155 Epoxy + Fiberglass Tensile Strength and Modulus (ASTM D3039)





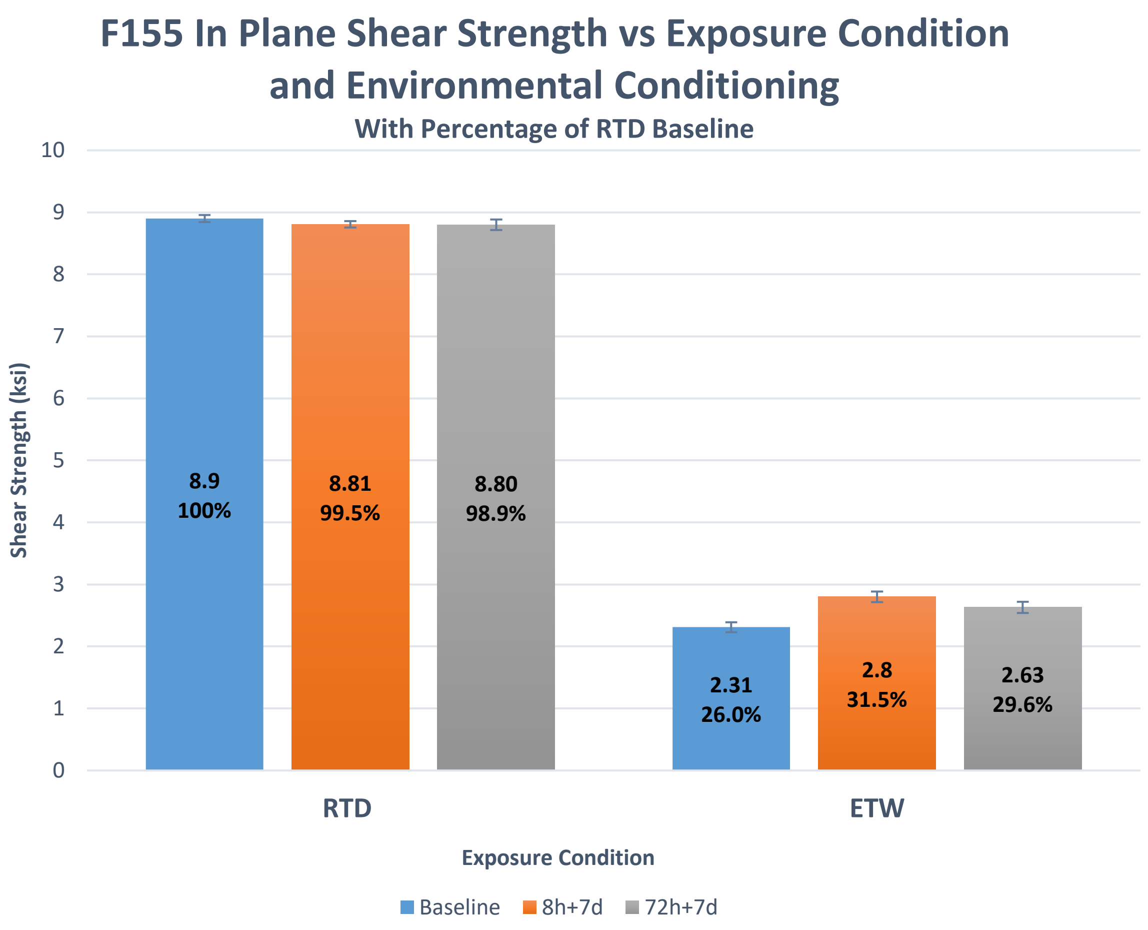
- **Insignificant Decreases to Tensile Strength and Modulus**
- **Tests were only conducted at room temp on dry specimens**

Phase 4 Results: Hexcel 155 Epoxy + Fiberglass Open Hole Compression (ASTM D6484)



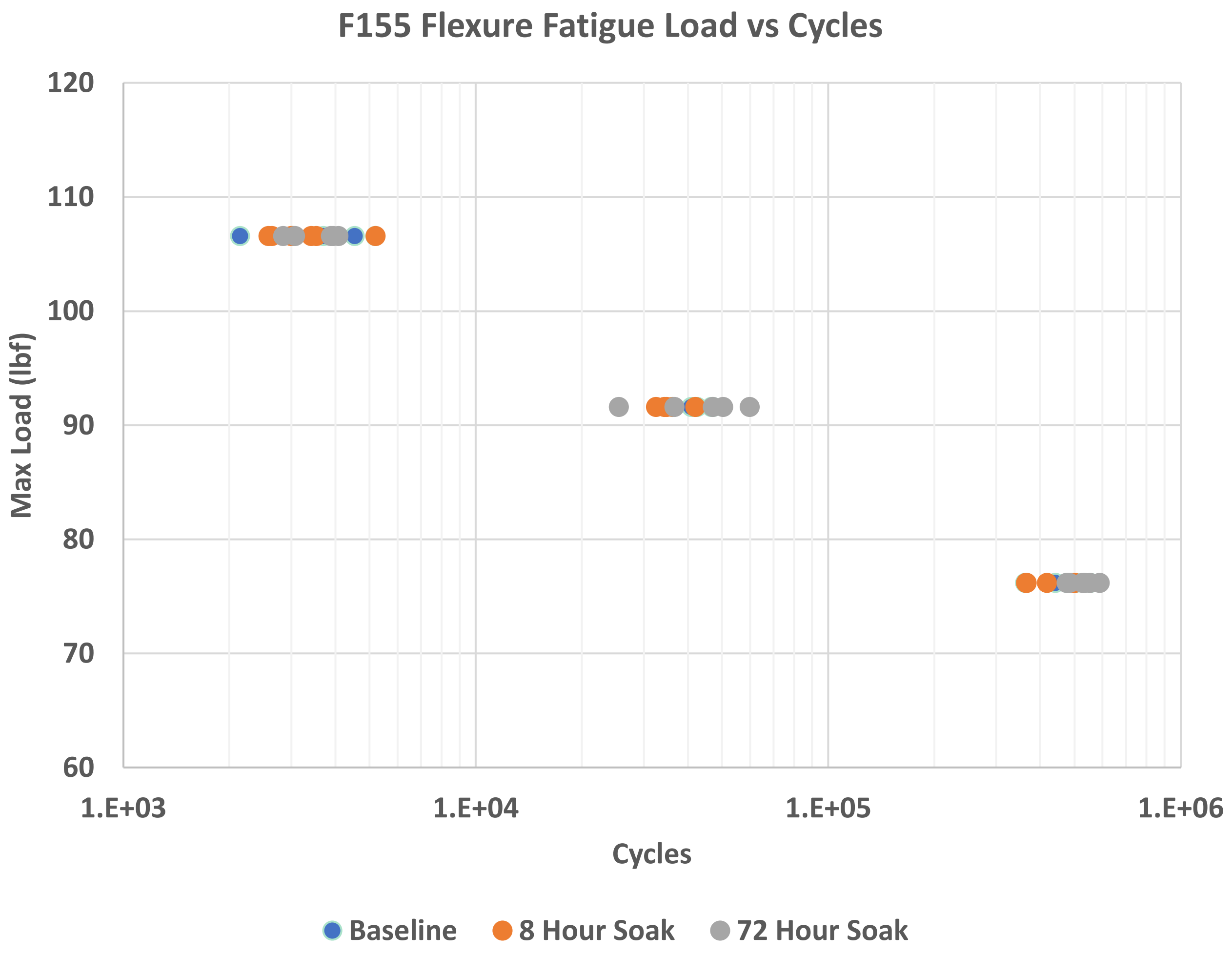
- **ETW specimens conditioned for 1200 hours at 160°F 85% RH before chemical exposure**
- **ETW specimens were tested at 145°F based on the baseline wet T_g results from DMA testing**
- **Insignificant mechanical change from chemical exposure**
- **Humidity conditioning + elevated test temp caused very large decrease to open hole compression properties**

Phase 4 Results: Hexcel 155 Epoxy + Fiberglass In Plane Shear (ASTM D3518)



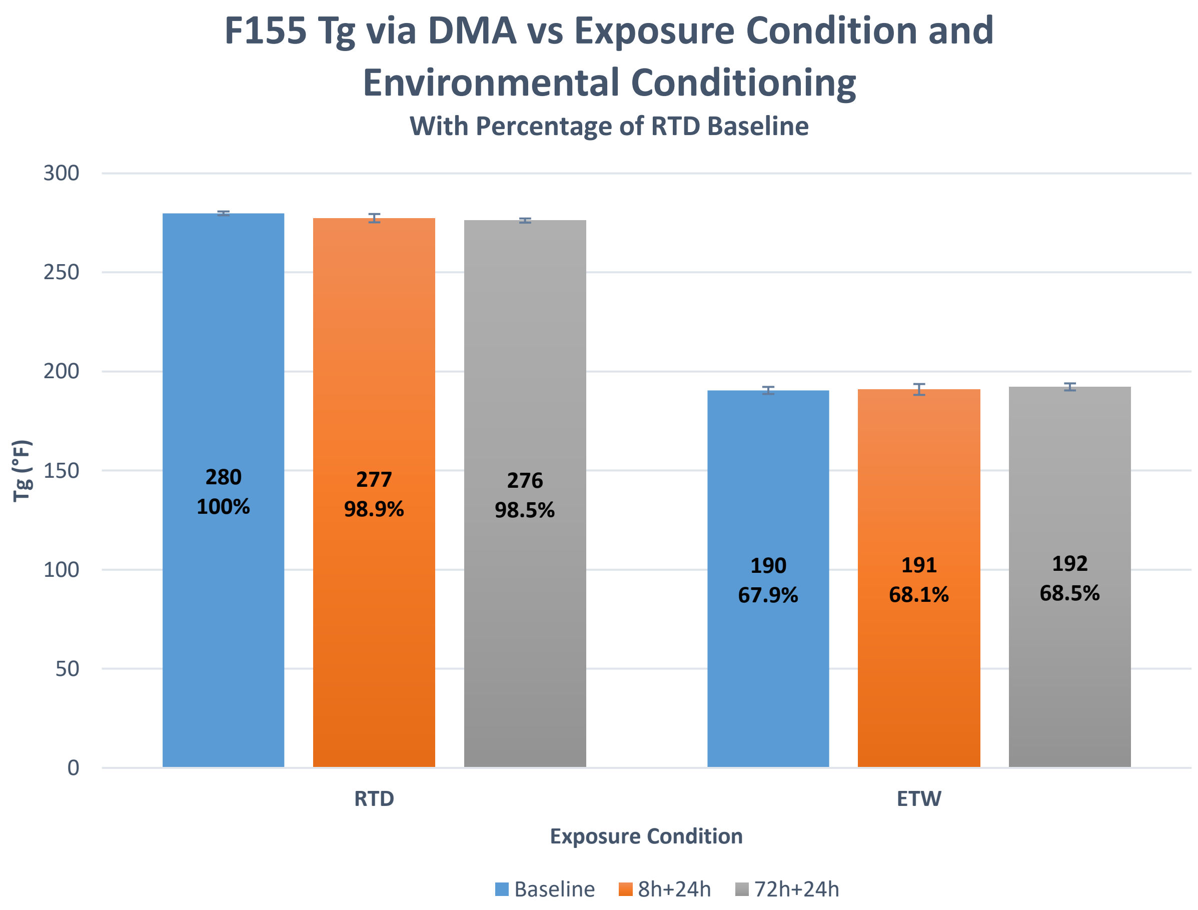
- ETW specimens conditioned for 1200 hours at 160°F 85% RH before chemical exposure
- ETW specimens were tested at 145°F based on the baseline wet T_g results from DMA testing
- Insignificant mechanical change from chemical exposure
  - Similar trends on Yield and Modulus
- Humidity conditioning + elevated test temp caused very large decrease to In Plane Shear properties

Phase 4 Results: Hexcel 155 Epoxy + Fiberglass Flexural Fatigue Testing



- **5 Specimens tested for each exposure condition and each load level**
- **No noticeable effect on fatigue life from the chemical stripper**

Phase 4 Results: Hexcel 155 Epoxy + Fiberglass Dynamic Mechanical Analysis (T_g)

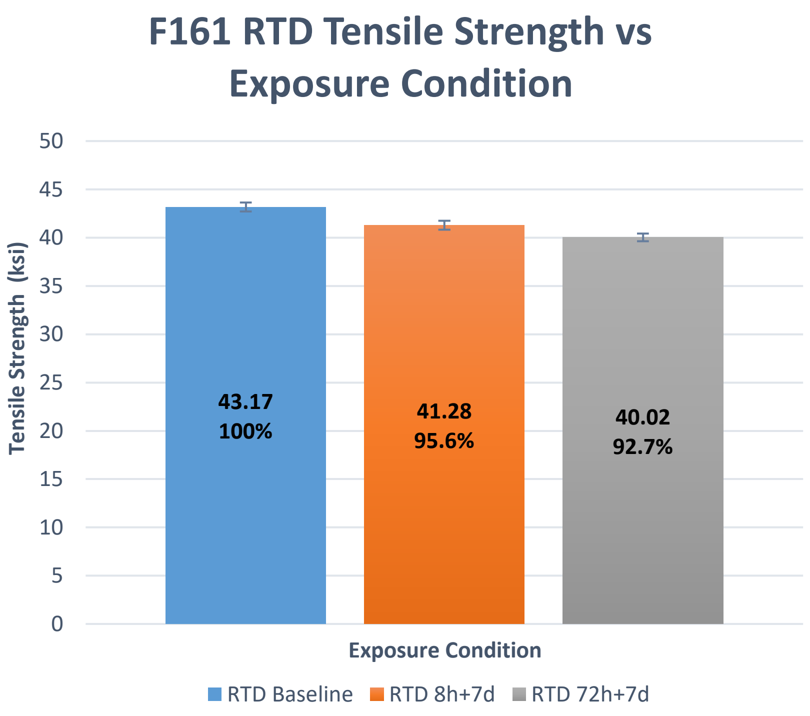


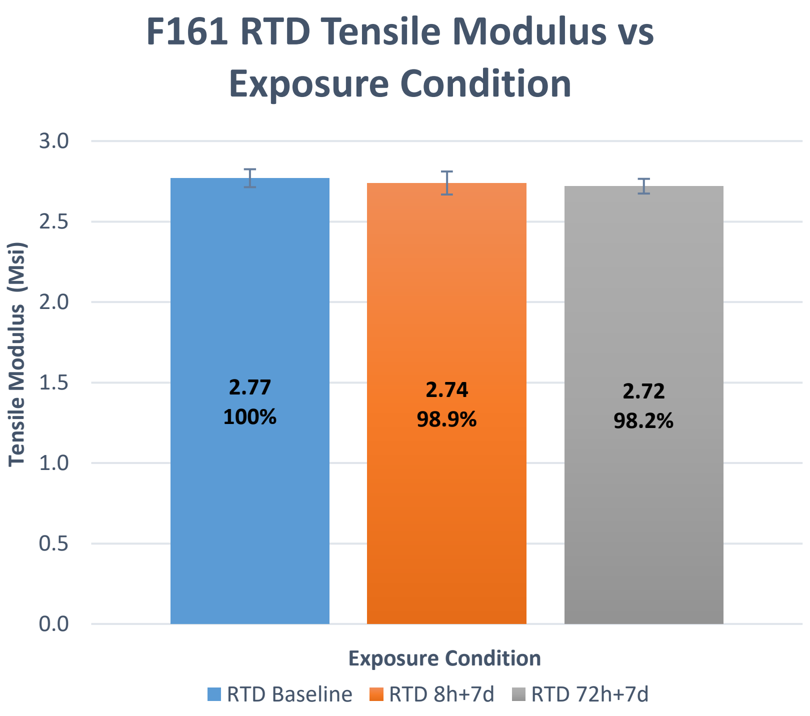
- **ETW specimens conditioned for 1200 hours at 160°F 85% RH before chemical exposure**
- **Insignificant change from chemical exposure in both RTD and ETW conditions**
- **Humidity conditioning caused large drop in T_g, any effect of chemical stripper is minimal compared to the drop between dry and wet T_g**

Phase 4 Results: Hexcel 161 + Fiberglass

- **Mechanical Testing**
  - Tension (RTD Only)
    - Strength and Modulus
  - In Plane Shear (RTD and ETW)
    - Shear Strength, Yield Strength and Shear Modulus
  - Open Hole Compression (RTD and ETW)
    - Open Hole Compression Strength
  - Flexure Fatigue (RTA) – Results not ready at this time
  - Dynamic Mechanical Analysis (RTD and ETW)
    - T_g

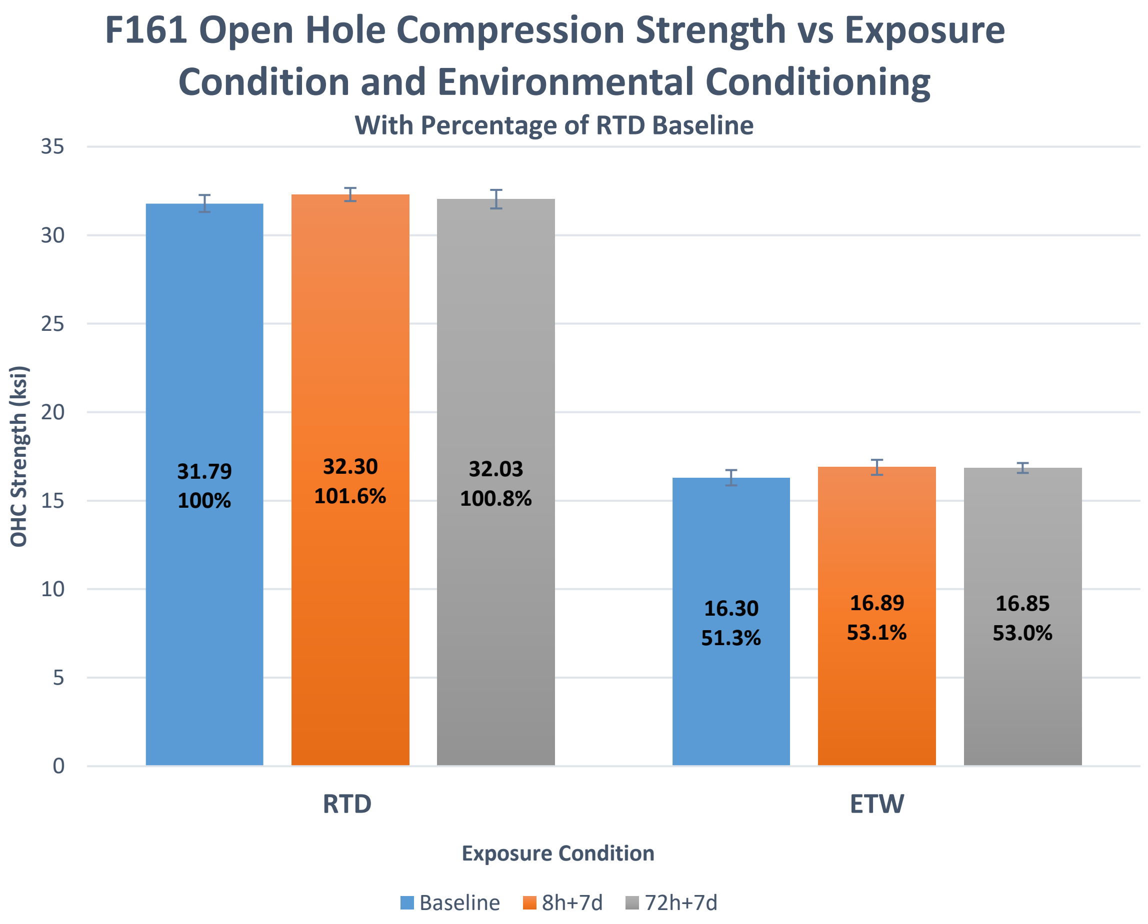
Phase 4 Results: Hexcel 161 Epoxy + Fiberglass Tensile Strength and Modulus (ASTM D3039)





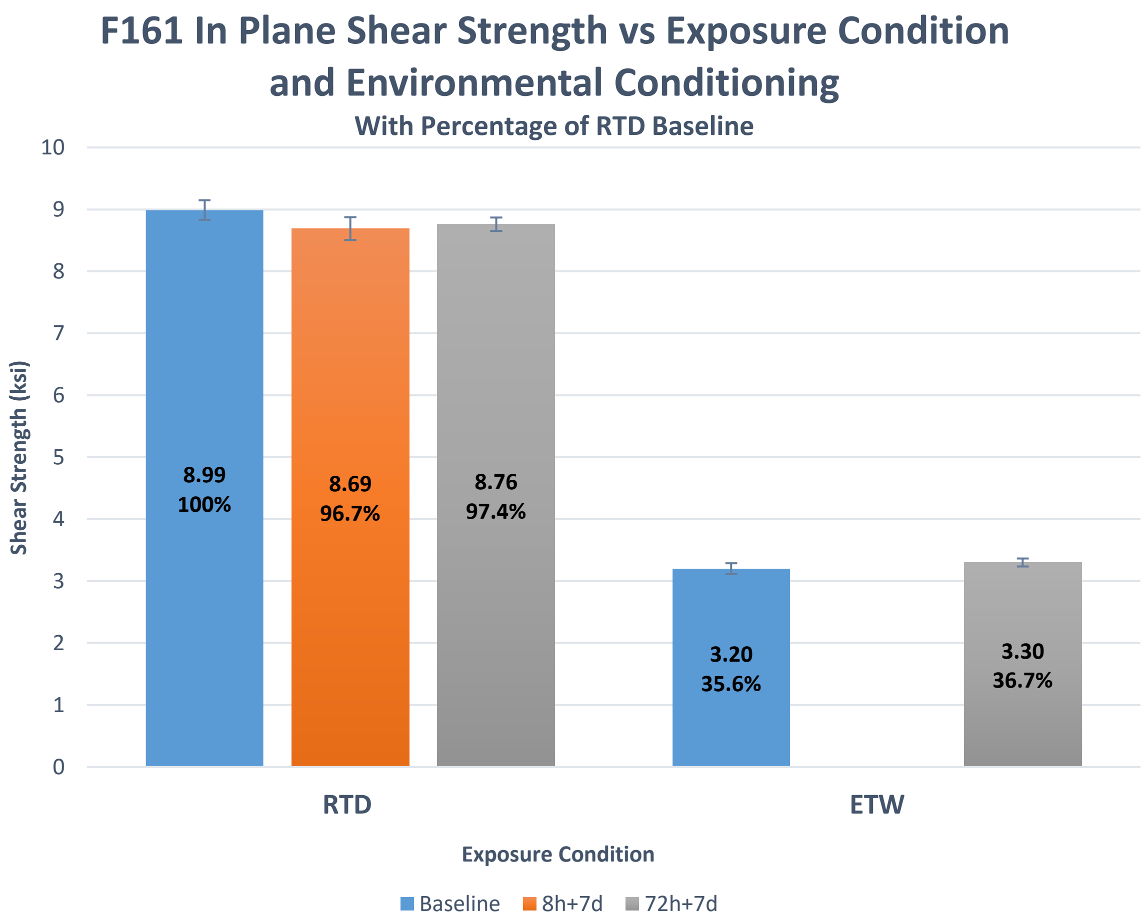
- **Small Decreases to Tensile Strength and Modulus**
- **Tests were only conducted at room temp on dry specimens**

Phase 4 Results: Hexcel 161 Epoxy + Fiberglass Open Hole Compression (ASTM D6484)



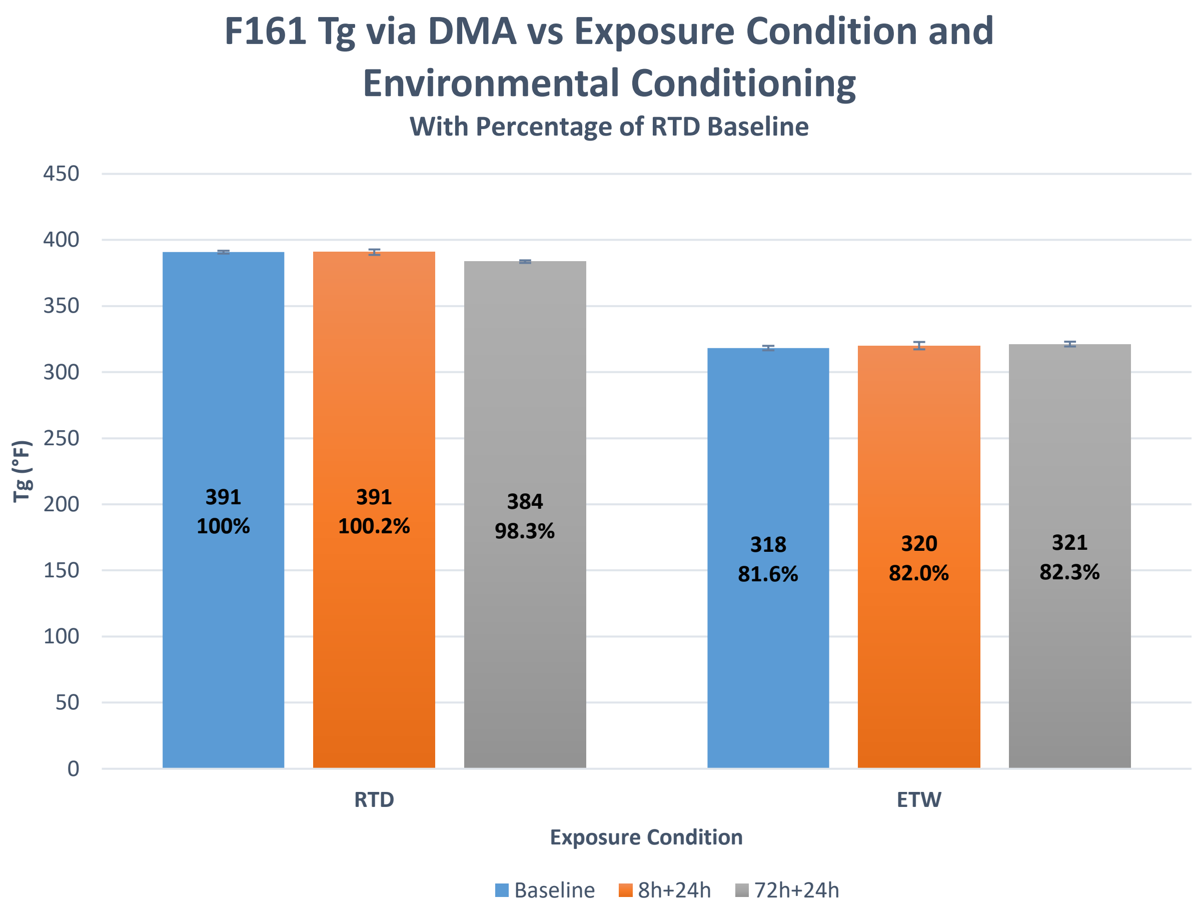
- ETW specimens conditioned for 1200 hours at 160°F 85% RH before chemical exposure
- ETW Specimens were tested at 275°F based on the baseline wet T_g results from DMA testing
- Insignificant change from chemical exposure
- Humidity conditioning + elevated test temp caused very large decrease to Open Hole Compression properties

Phase 4 Results: Hexcel 161 Epoxy + Fiberglass In Plane Shear (ASTM D3518)



- ETW specimens conditioned for 1200 hours at 160°F 85% RH before chemical exposure
- ETW Specimens were tested at 275°F based on the baseline wet T_g results from DMA testing
- Small change in properties from chemical exposure
  - Similar trends on Yield and Modulus
- Humidity conditioning + elevated test temp caused very large decrease to In Plane Shear properties

Phase 4 Results: Hexcel 161 Epoxy + Fiberglass Dynamic Mechanical Analysis (Tg)

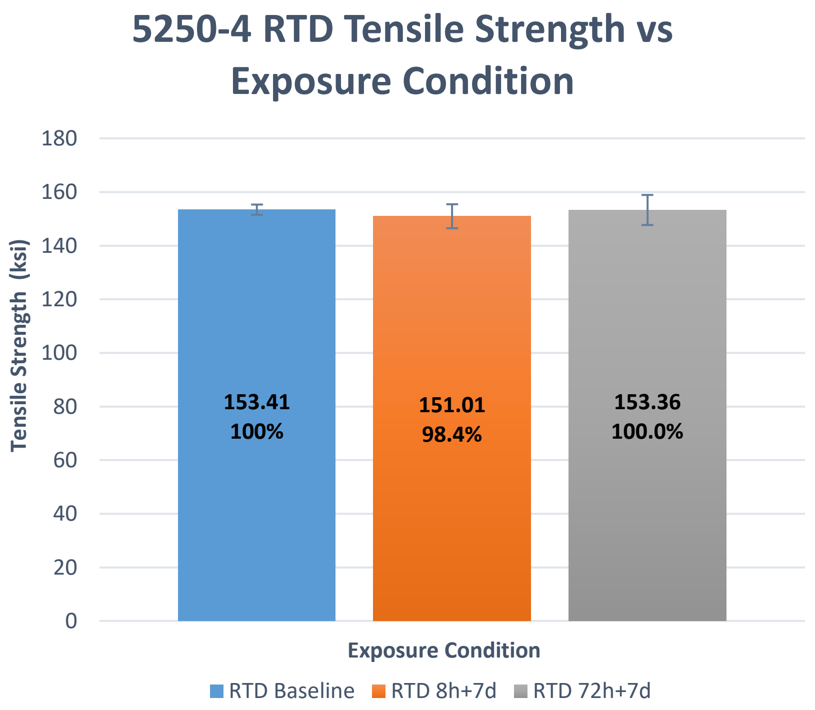


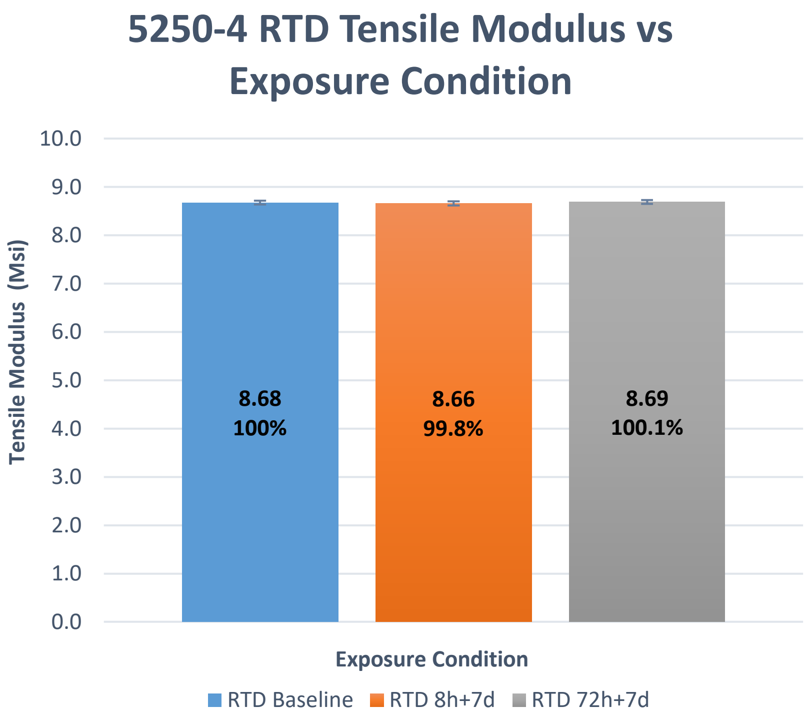
- **ETW specimens conditioned for 1200 hours at 160°F 85% RH before chemical exposure**
- **Insignificant change from chemical exposure in both RTD and ETW conditions**
- **Humidity conditioning caused large drop in T_g, any effect of chemical stripper is minimal compared to the drop between dry and wet T_g**

Phase 4 Results: 5250-4 + Carbon Fiber

- **Mechanical Testing**
  - Tension (RTD Only)
    - Strength and Modulus
  - In Plane Shear (RTD and ETW)
    - Shear Strength, Yield Strength and Shear Modulus
  - Open Hole Compression (RTD and ETW)
    - Open Hole Compression Strength
  - Flexure Fatigue (RTA) – Results not ready at this time
  - Dynamic Mechanical Analysis (RTD and ETW)
    - T_g

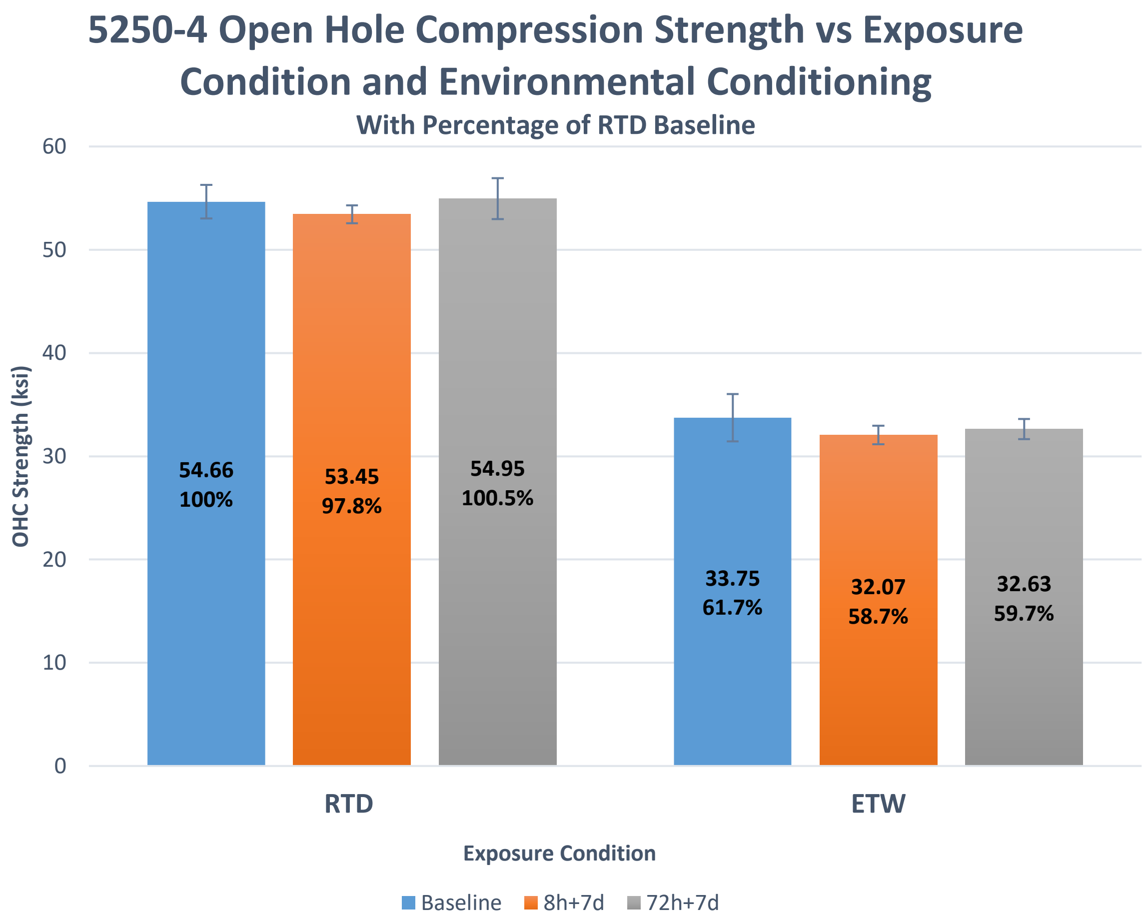
Phase 4 Results: 5250-4 + Carbon Fiber Tensile Strength and Modulus (ASTM D3039)





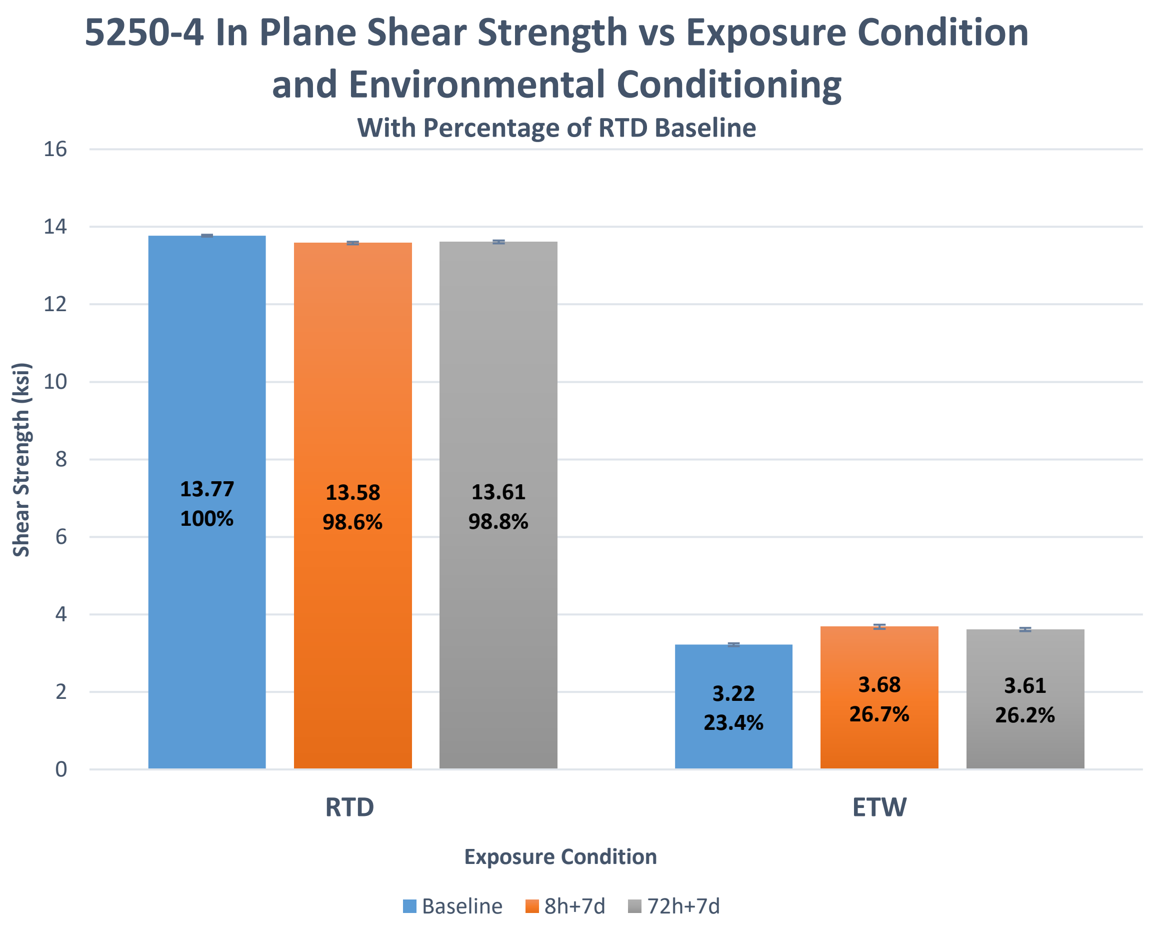
- **Insignificant Changes to Tensile Strength and Modulus**
- **Tests were only conducted at room temp on dry specimens**
- **No apparent effect from chemical exposure**

Phase 4 Results: 5250-4 + Carbon Fiber Open Hole Compression (ASTM D6484)



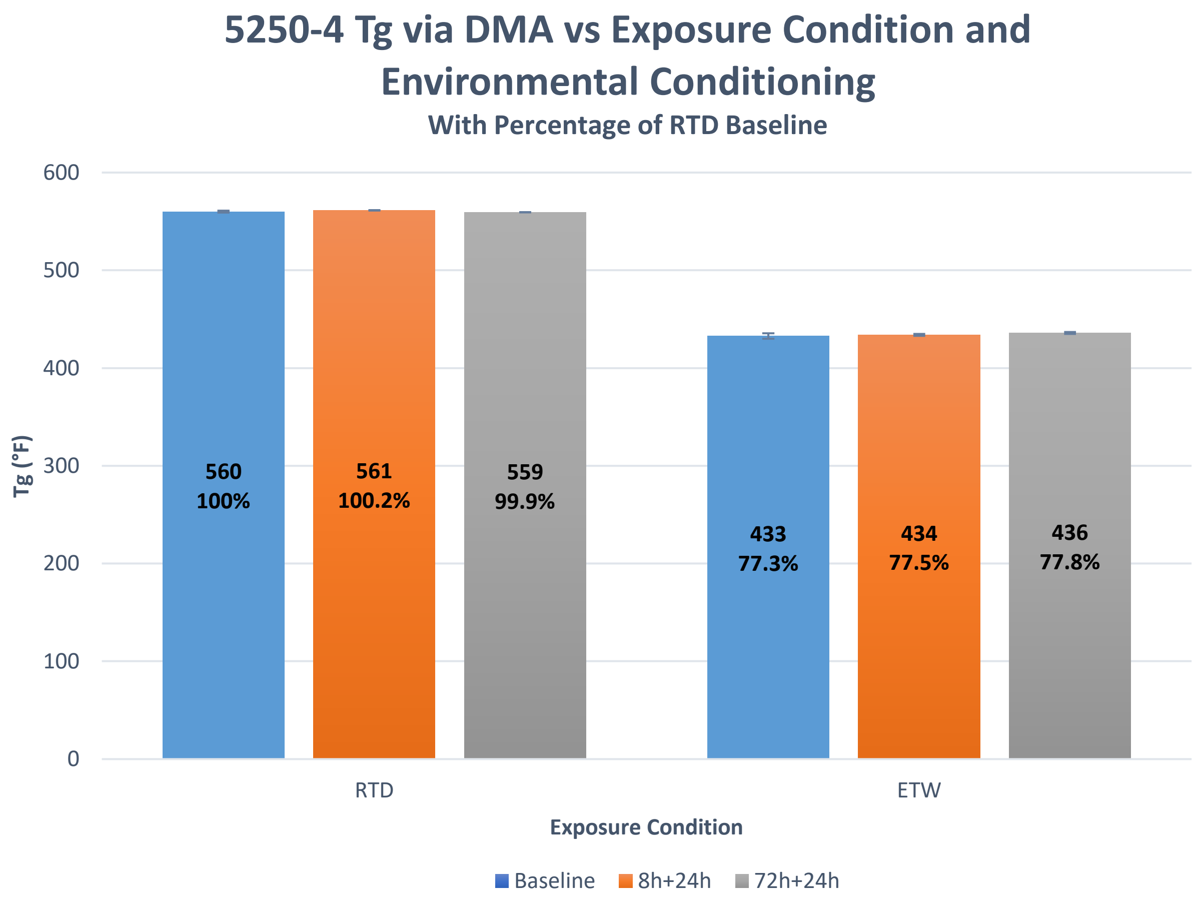
- **ETW specimens conditioned for 1200 hours at 160°F 85% RH before chemical exposure**
- **ETW Specimens were tested at 385°F based on the baseline wet T_g results from DMA testing**
- **Insignificant change from chemical exposure**
- **Humidity conditioning + elevated test temp caused very large decrease to Open Hole Compression properties**

Phase 4 Results: 5250-4 + Carbon Fiber In Plane Shear (ASTM D3518)



- ETW specimens conditioned for 1200 hours at 160°F 85% RH before chemical exposure
- ETW Specimens were tested at 385°F based on the baseline wet T_g results from DMA testing
- Insignificant mechanical change from chemical exposure
  - Similar trends on Yield and Modulus
- Humidity conditioning + elevated test temp caused very large decrease to In Plane Shear properties

Phase 4 Results: 5250-4 + Carbon Fiber Dynamic Mechanical Analysis (T_g)



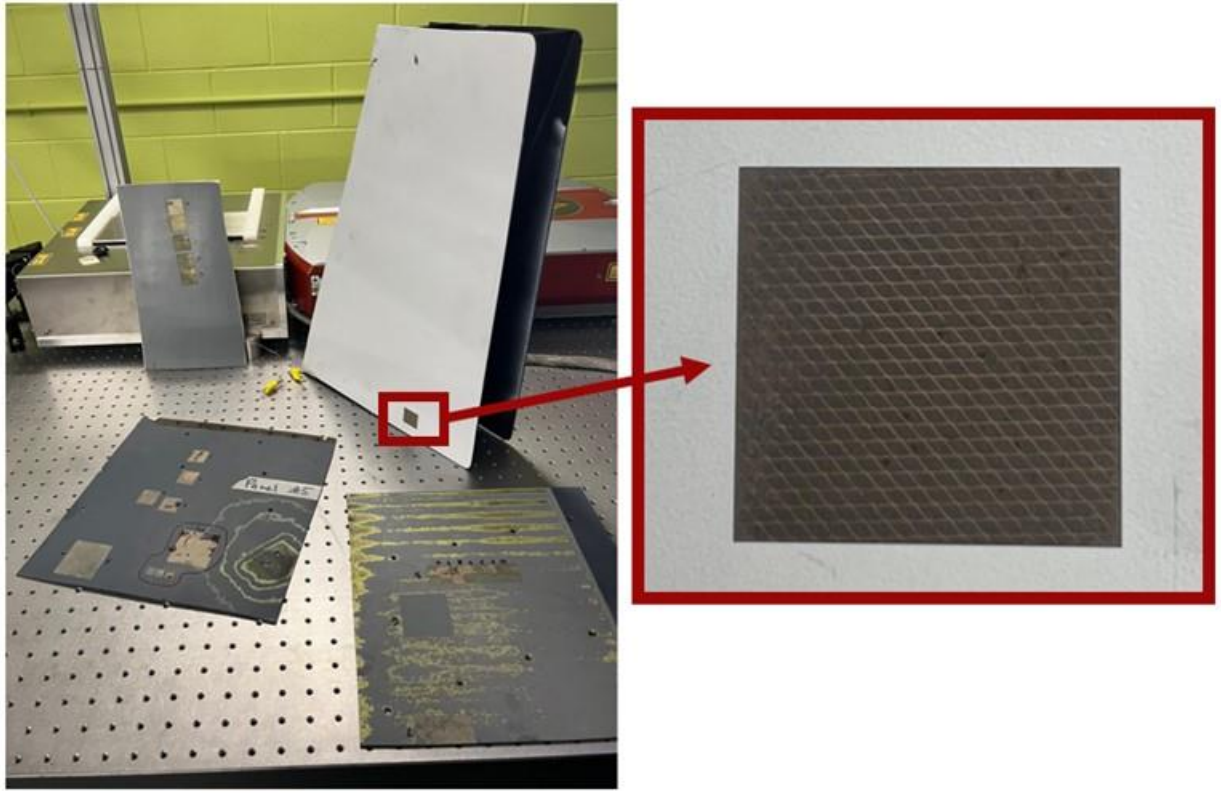
- **ETW specimens conditioned for 1200 hours at 160°F 85% RH before chemical exposure**
- **Insignificant change from chemical exposure in both RTD and ETW conditions**
- **Humidity conditioning caused large drop in T_g, any effect of chemical stripper is minimal compared to the drop between dry and wet T_g**

Phase 4: Probe Experiment #1 Results

- **Goal of Probe Experiment #1 in Phase 4 was to advance a near-infrared detection technique which was found in Phase 3 to be able to detect water absorbed into epoxy / BMI materials.**
- **Near-Infrared (NIR) was able to measure water absorption on the surface and past the surface of epoxy / BMI materials – but needed to be advanced for possible hand-held use for depot maintainers.**
- **Probe Experiment #1 would determine if this technique could detect water past carbon fiber and fiberglass, since both would interfere with the measurements.**
- **Work ongoing – progress will be fully reported in Phase 4 final reports.**

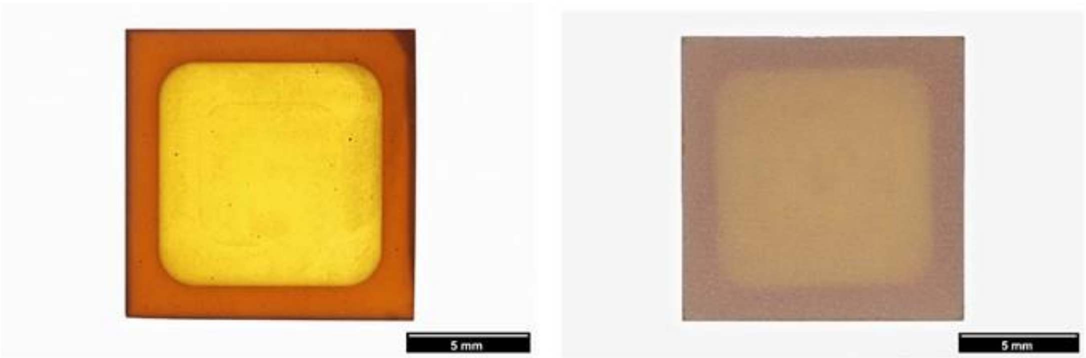
Phase 4: Probe Experiment #2 Results

- **Use of Femtosecond laser on surface of F16 part was able to successfully remove the paint without damaging the underlying copper mesh.**
  - **Chemical paint stripper caused the copper mesh to corrode and did not get all the paint off.**
  - **Thought was to see if femtosecond laser could drive out trapped moisture in a part exposed to chemical paint stripper.**

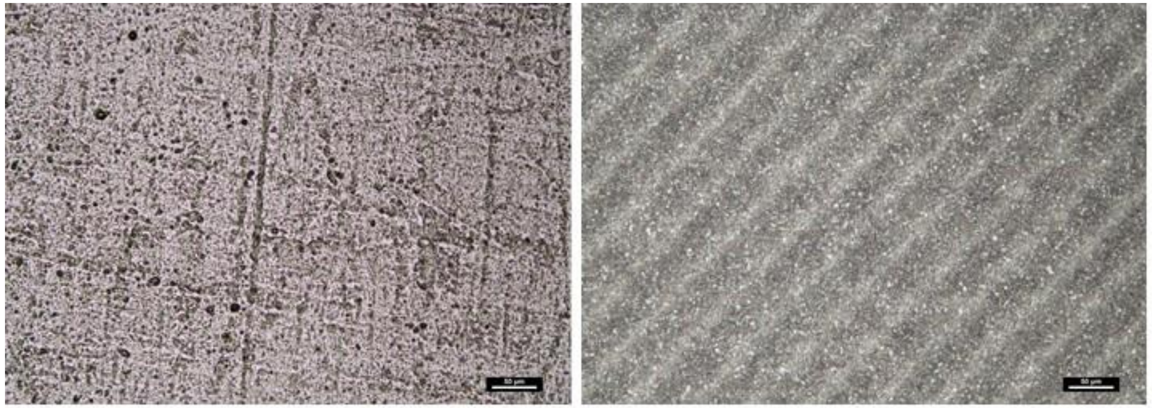


Phase 4: Probe Experiment #2 Results

- **Because the femtosecond laser is a surface focused treatment, the water absorbed in the bulk of the epoxy was not removed.**
- **Laser did show a uniform surface preparation of the epoxy, which suggests it may yield a slightly roughened surface for better paint adhesion.**



Before After



Before After

Phase 4 Conclusions

- F155 and F161 Epoxy and 5250-4 BMI shows susceptibility to water, with some notable drops in properties after humidity exposure.
- Chemical Paint Stripper does seem to cause some swelling and discoloration of the F155 Epoxy, but this is not seen with F161 Epoxy.
- 5250-4 BMI shows resistance to chemical paint stripper – no notable changes in properties seen with testing conducted so far. Moisture and Elevated Temperatures have a significant effect on properties.

Conclusions and Guidance

- **Very important to check for compatibility between epoxy/resin of interest and chemical paint stripper early on via simple prolonged exposure tests.**
- **T_g seems to be one of the most affected properties after chemical stripper exposure, so pay attention to this measurement.**
- **Water, once in the composite, greatly changes the properties of the material, and the chemical paint stripper appears to "seal" the material in the composite, making it difficult to remove.**
- **Final Reports from Phase 4 to be issued in May / June 2024, and ACO to issue official guidance on chemical paint stripper use for USAF aircraft composites.**

Questions?

- **Thank you for your attention.**