EFFECTS OF LONG-TERM DEEP-SEA IMMERSION ON THE IMPLOSION AND EXPLOSION OF HOLLOW COMPOSITE CYLINDERS

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EFFECTS OF LONG-TERM DEEP-SEA IMMERSION ON THE IMPLOSION AND EXPLOSION OF HOLLOW COMPOSITE CYLINDERS

BY

TYLER CHU

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF
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OF

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DEAN OF THE GRADUATE SCHOOL

UNIVERSITY OF RHODE ISLAND

2021
ABSTRACT

The effects of long-term exposure to a sea-floor depth ocean environment on the mechanical behavior of carbon-fiber / epoxy (CFE) continuous filament-wound composite tubes were investigated experimentally. Using a novel high pressure accelerated aging vessel and the Arrhenius relationship for water diffusion into a polymer matrix composite, the acceleration factor was determined to be approximately 21.5; that is one day of aging in lab at 70°C represents 21.5 days of real life service at 3°C. Specimens were aged for 0, 9, and 24 days which represent 0, 193.5, and 516 days of service. The specimens were sealed and half were subjected to external hydrostatic pressure until they imploded, and the other half were pressurized to 80% of the average implosion pressure previously determined and subjected to a strong impulse by means of a nearby explosive detonator. The behavior of the tube immediately before and during its collapse was recorded by high frequency pressure transducers and high speed digital stereo photography. In both the hydrostatic-initiated and explosive-initiated implosions, the composites did not significantly degrade nor change their properties with aging. These observations are in contrast to a previous study with nearly identical conditions except aged at atmospheric pressure. The results from this current study are supported by the conclusions of another study which studied CFE laminate aging under identical conditions. An additional set of experiments were performed using the same preparation methods and similarly sourced and dimensioned samples to investigate the impact of long-term deep-sea immersion on the composite’s ability to hold internal pressure. The specimens were sealed and internally pressurized until leak or burst, and the phenomenon recorded by the same camera and transducer setup. It was found that
the high pressure aging process did not significantly change the bursting pressure of the CFE tubes, but did significantly affect the fracture characteristics, which may have implications on non-bursting failure such as leaking.
ACKNOWLEDGMENTS

The author expresses his foremost gratitude to his parents, siblings, and friends for supporting him through this adventure. He also acknowledges appreciation for the support and guidance from his major professor, Dr. Arun Shukla. The author is thankful for the excellent faculty and peers in contributing to his knowledge of the engineering discipline. His peers at the DPML provided critical feedback, suggestions, and support. Several individuals deserve special mentions: Dillon Fontaine for his friendship and mentoring throughout the author’s graduate experience, Tim Pickard for his unfailing assistance and ceaseless eagerness to help, Nic Zirker for providing high quality model renders, and Dave Ferriera and Joe Gomez of the URI machine shop for their watchful eyes and machining advice. Finally, thanks are in order to Dr. Maria Medeiros and ONR grant #N00014-18-1-2641 for the opportunity to perform this research, and Dr. Jim LeBlanc for his external mentorship as the NUWC contact.
PREFACE

This thesis is written in the Manuscript format. Both chapters are intended to be submitted for publication. The works contained in these documents are intended to support and contribute to the understanding of carbon-fiber epoxy composites under extreme conditions for long durations.
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MANUSCRIPT 1

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Collapse Behavior of Carbon-Fiber Epoxy Cylinders Subjected to Long-term Seawater Exposure at Seafloor Depth Pressures

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ABSTRACT

This study investigates how extended submersion in high pressure seawater affects the implosion behavior of round carbon fiber epoxy tubes. Accelerated life cycling was performed by submerging samples in 3.5% saline solution at 70°C for 0, 9, and 24 days pressurized at 6000psi. Using the Arrhenius relationship, these times represent 0, 193.5, and 516 days of service. All experiments were conducted underwater in a large vessel designed to maintain pressure throughout the implosion while mimicking a free field environment. All samples were sealed with endcaps. Half of the samples were externally pressurized until collapse. The other half were pressurized to 80% of the average collapse pressure determined by the previous work and subjected to a sharp impulse by an explosive detonator placed nearby. The events were recorded with high frequency pressure transducers and two high speed cameras in a stereo configuration for 3D analysis. The samples showed little to no change in their collapse behavior as opposed to similar specimens aged in atmospheric pressure saline, which did show dramatic and negative change. This indicates that aging at these high-pressure conditions virtually negates degradation from moisture absorption.
1. Introduction

Composite materials have seen an increasing amount of use in vessels and structures due to their high strength to weight ratio. Particularly for marine environments, composites are valued for having greater corrosion resistance compared to common structural metals. Mouritz et al. [1] discuss the increasing use of fiber reinforced polymer composites in naval warships and submarines for both component and their housings, and the vessel’s superstructure. There is also an increasing interest in unmanned underwater vehicles (UUVs) and remote operated vehicles (ROVs). Because these vehicles can exclude the life support and safety/design factors required with an on-board human operator, they can be made lighter, more compact, and be made to perform under more strenuous conditions. These increased capabilities require the strength and light weight that composites bring.

There has been an increasing interest in forward deployed structures, where vehicles and/or payloads may be stored near or on the ocean bed for years before being called into service. Unmanned and remote vehicles are the perfect candidate for these purposes because they can be made to be much more difficult to detect, as well as able to being able to be placed in such extreme conditions without the need to care for a human. Due to the time scales and pressures present in such a venture, there is a need to understand how the composite’s properties will be affected, especially with regards to how the composite behaves when subjected to crushing external pressures or shock loading at near-surface conditions.

Water diffusion into resins and resin composites have been studied in pressures equivalent to shallow depths [2-5] and at pressures up to 50MPa, equivalent to 5000
meters deep [6-10]. Rice and Romatowski [11] studied the Arrhenius temperature dependence of water diffusion into polymers used as composite matrices for accelerated life cycling purposes, a technique that this study uses. Literature regarding the effects of pressure on diffusion are inconsistent and sensitive to the type of polymer in question, with some finding that pressure tends to increase saturation mass yet leave diffusivity unchanged [12], while others found that both saturation mass and time to saturation increased with pressure [13].

This study closely follows the work done by Javier, Matos, and Shukla [14] and by Fontaine, Leblanc, and Shukla [15], which explored hydrostatic and shock initiated implosions of carbon fiber epoxy (CFE) composite cylinders after prolonged exposure to unpressurized saline, and the behavior of vacuum infused CFE laminate sheets after prolonged exposure to high pressure saline respectively. However, to the author’s knowledge, no work has been done on exploring CFE cylinder implosions after prolonged exposure to a pressurized saline environment. This study uses a novel high-pressure aging facility housed in the University of Rhode Island’s Dynamic PhotoMechanics Laboratory to expose specimens to high pressure saline at elevated temperatures. Two high speed cameras and high frequency tourmaline pressure transducers are used to capture the implosion event. It was found that for the hydrostatic implosions, there was little difference in collapse pressure between each set of aging. For the shock-initiated implosions, it was similarly observed that there was little difference in the tube’s behavior during loading and collapse.

2. Materials and Methods
2.1. Specimen Details

The CFE composite tubes used in this study are from RockWest Composites (West Jordan, UT). These tubes have a nominal inner diameter of 1.375 inches and outer diameter of 1.5 inches (part number 35050-S). Specimens were cut to be 15 inches long with a wet abrasive saw. Ends were sealed with endcaps intruding 0.5 inches into the tube from each side, for a nominal unsupported length of 14 inches. Table 1 shows the layup details and resin of the tubes. The fibers used are standard modulus carbon fibers.

Table 1: Layup details of RockWest Composite's 35050-S

<table>
<thead>
<tr>
<th>RockWest 35050-S</th>
<th>Layup:</th>
</tr>
</thead>
<tbody>
<tr>
<td># Circuits:</td>
<td>Angle (°):</td>
</tr>
<tr>
<td>12</td>
<td>±15</td>
</tr>
<tr>
<td>0.45 Lead</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>±45</td>
</tr>
<tr>
<td>13</td>
<td>±15</td>
</tr>
<tr>
<td>Nominal Dimensions (mm):</td>
<td>38.1OD x 1.59Wall x 381L</td>
</tr>
<tr>
<td>Resin:</td>
<td>Epon 862/Lindride 6K</td>
</tr>
</tbody>
</table>

2.2. Calculation of Acceleration Factor

Previous research [6, 14] suggests that degradation of the composite is primarily due to water diffusion into the polymer matrix. With this assumption, aging can be accelerated by increasing the rate at which water is absorbed. Rice and Ramotowski
[11] developed a method based on the Arrhenius relationship of rate of diffusion to temperature. The Arrhenius relationship as it applies to the diffusivity, $D$, of water into the matrix is:

$$D = D_0 e^{-\frac{E_A}{RT}} \quad (1)$$

where $D_0$ is a constant, $R$ is the ideal gas constant, $T$ is absolute temperature, and $E_A$ is the activation energy. The activation energy is assumed to be a function of material and environment; both are held constant for this study, so it is assumed that the activation energy is constant as well. This relationship and assumption allow for calculating the reaction rate at any temperature once diffusivity is found.

Figure 1: Small pressure vessel used for aging samples for the diffusion study.

To calculate diffusivity, Crank’s [16] method to calculate $D$ from a range of temperatures is used. Three tests were performed with small samples cut from the same tube stock as the experimental samples. These samples were desiccated for at least 48 hours to ensure a moisture-free baseline, and then immersed in 3.5% saline pressurized to 6000psi in the small pressure vessel shown in Figure 1, heated to 18, 45, and 65°C.
These samples were periodically removed from their bath and their mass change recorded by a scale with a precision of $10^{-4}$ grams; it is assumed that any mass change is solely due to water absorption with no leaching or chemical reactions occurring between the composite and the water or the pressure vessel. Absorption curves of time to percent mass change were plotted for each test. It is reasonable to assume the diffusion is Fickian and one dimensional because each sample is thin, with a face area much greater than the edge area. Under these conditions, Crank calculated the diffusivity to be

$$D = 0.049 \times \frac{l^2}{t_{50}} \quad (2)$$

where $l$ is the tube’s wall thickness and $t_{50}$ is half the time it takes for the sample to reach full absorption. Based on the assumption that there is no chemical change or leaching by the composite, it is safe to assume that $t_{50}$ is independent of temperature within the same conditions. With the diffusivity at each of the three temperatures known, Equation 1 can be rewritten as:

$$\ln(D(T)) = \ln(D_0) - \left(\frac{E_A}{R}\right)\left(\frac{1}{T}\right) \quad (3)$$

A linear regression can be drawn when plotting inverse temperature against the natural log of diffusivity. The slope of this line is $-E_A/R$ which is assumed to be constant. Acceleration factor can be found by taking the ratio of diffusivity between the laboratory aging temperature, $T_2$, and the service temperature, $T_{ref}$ as follows:

$$AF = \frac{D_0 e^{-\frac{E_A}{RT_2}}}{D_0 e^{-\frac{E_A}{RT_{ref}}}} = e^{\left(\frac{E_A}{R}\right)\left(\frac{T_2-T_{ref}}{T_2-T_{ref}}\right)} \quad (4)$$

For this study, $T_{ref}$ is taken to be 3.0°C, the commonly accepted value for seawater in the abyssal plain.
2.3. High Pressure Aging Facility and Aging Methodology

A custom vessel, shown in Figure 2, made to handle high pressure, high temperature saline was designed and manufactured in-house at the Dynamic PhotoMechanics Laboratory. Specimens to be aged were first desiccated for at least 48 hours, then placed in the vessel with 3.5% saline, and pressurized to 6000psi. The pressurized vessel was then placed in a large water bath heated to 70°C. This temperature was chosen in order to stay below the glass transition temperature of the composite at 82°C. This process was followed for two batches of samples: one for 9 days and the other for 24 days.
2.4. Hardware and Software used for Capture and Analysis

A high contrast, random, speckled black dot pattern on a white background was painted on each sample by hand after the high pressure aging process described in Section 2.3. This pattern was recorded by two Photron SA1.1 high speed cameras at 30,000 frames per second in a stereo imaging setup. The dots were sized and spaced to fill approximately 5 pixels diameter when viewed by the cameras. These stereo images were analyzed in Correlated Solution’s VIC-3D 8 analysis software.

Figure 2: Modelled view of the aging vessel.
Pressure signals from the experiments were captured by sensors placed around the specimen; the exact positions of these sensors are detailed in Section 2.5. These sensors are PCB Piezotronic’s tourmaline underwater blast sensor, model 138A05. The data was analyzed by the AstroVIEW X software, by Astro-Med Inc.

2.5. Setup of Experimental Facility and Experimental Procedure

A large pressure vessel is housed in the Dynamic PhotoMechanics Laboratory. It is a hemispherical vessel, 2.1 meters diameter, and capable of holding 1800-2000 gallons and 1000psi. The sealed and speckled specimen is placed horizontally in the pressure vessel such that it is supported by an internal frame, visible to the camera setup. In the hydrostatic series, four pressure sensors are placed 2.25 inches from the surface of the specimen. Two are placed along the center of the specimen, 40 degrees apart. The other two are placed in the same shape halfway between the center and the edge of the unsupported length, or 3.5 inches from the center. Figure 3 illustrates the experimental setup for the hydrostatically initiated implosion series.
To perform the hydrostatic experiments, the vessel is sealed and the components are placed as described. Nitrogen is introduced at the top of the tank at a rate of 1-2 psi per second until the implosion occurs. The cameras and sensors are activated by a manual trigger, capturing data preceding and following the trigger by a certain amount of time. These are saved for analysis, along with the implosion pressure.

In the shock-initiated series, an RP-85 exploding-bridgewire detonator by Teledyne Defense Electronics is placed behind the specimen, with 5.25 inches between the tip of the explosive and the surface of the specimen. One sensor is placed 3.25 inches above the specimen. Figure 4 illustrates the experimental setup for the shock-initiated implosion series.
Figure 4: Experimental setup for shock-initiated implosion experiments

Figure 5: Larger, more detailed view of the experimental tank setup. Specimen and sensor placements for hydrostatic and undex implosions are shown together.
To perform the shock-initiated experiments, the vessel is sealed with the components placed as illustrated in Figure 4. Nitrogen is used to pressurize the tank up to 80% of the average implosion pressure determined by the hydrostatic implosion experiments. When this pressure is reached, the RP-85 is detonated. The events are captured and recorded using the same procedures as with the hydrostatic experiments.

3. Results and Discussion

3.1. Results of the Diffusion Study

For each test, each specimen was immersed in saline solution pressurized to 6000psi and maintained at the test temperature, and the resulting mass gain periodically plotted against time to create the absorption curves. The saturation mass gain was found to be 0.44%, reached by the 65°C sample after approximately 450 hours, making half the saturation mass gain to be 0.22%. The saturation mass gain is assumed to be constant for each test because the temperature was kept under the glass transition, and the test conditions were kept constant between each test. To use Equation 2, we require the time at which the mass gain reached 0.22%. For each test, a logarithmic regression was plotted with a strong fit: the coefficient of determination (r²) being over 0.91 for each test. From here, it is a simple matter to find t₅₀ with the regression equation. Using Equation 4, Tₑᶠ was set to 3°C and T₆ to 70°C resulting in an acceleration factor of 21.5. That is, every day spent aging at 70°C in laboratory conditions is assumed to be equivalent to 21.5 days in service at 3°C.

3.2. Results of Hydrostatic Implosion Testing
Implosion testing was conducted to determine whether the mechanical properties of filament wound CFE composites would change for unaged (virgin), 9-day, and 24-day aged cases. Table 2 is a summary of these tests.

**Table 2: A summary of the hydrostatically initiated implosions.**

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Collapse Pressure (psi)</th>
<th>Max Dynamic Pressure (psi)</th>
<th>Avg Collapse Pressure</th>
<th>% Change from Virgin</th>
</tr>
</thead>
<tbody>
<tr>
<td>Virgin 1</td>
<td>464</td>
<td>420</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Virgin 2</td>
<td>463</td>
<td>460</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Virgin 3</td>
<td>470</td>
<td>323</td>
<td>465.7</td>
<td>0</td>
</tr>
<tr>
<td>9-day 1</td>
<td>455</td>
<td>373</td>
<td></td>
<td></td>
</tr>
<tr>
<td>9-day 2</td>
<td>449</td>
<td>377</td>
<td></td>
<td></td>
</tr>
<tr>
<td>9-day 3</td>
<td>461</td>
<td>359</td>
<td>455</td>
<td>-2.3</td>
</tr>
<tr>
<td>24-day 1</td>
<td>494</td>
<td>285</td>
<td></td>
<td></td>
</tr>
<tr>
<td>24-day 2</td>
<td>498</td>
<td>396</td>
<td></td>
<td></td>
</tr>
<tr>
<td>24-day 3</td>
<td>486</td>
<td>402</td>
<td>492.7</td>
<td>5.8</td>
</tr>
</tbody>
</table>

Javier et al. [14] found that when saline aged in atmospheric pressure, the average collapse pressure for their similar CFE specimens dropped by over 20% and 23% for age times of 35 and 70 days respectively. They believe that this is due to the degradation of the in-plane shear modulus, which dropped by 28% and 33% for their 35 and 70 day aging respectively. While the time aged by Javier et al. is significantly longer than that done for this study, the composites in both experiments were brought to (nearly) saturation. If the only significant source of material degradation is water ingression, the difference in specimen behavior with regards to percent saturation between aging at different pressures are comparable.

Fontaine et al. [15] performed mechanical testing on CFE laminate sheets aged under identical high-pressure conditions using the same facilities as this study. Their results showed that quasi-static in-plane tensile and shear moduli and ultimate tensile
and shear strengths and strains were not significantly affected by the aging process. They propose that this is due to the construction of vacuum infused laminates, where a cross-section view shows alternating fiber-rich and matrix-rich bands. The high pressure is thought to close micro-voids at the fiber-matrix interface as well as compressing the matrix which limits the volume which water can occupy and forcing moisture to collect largely between plies. Koudela and Strait [17] developed a modified equation for determining the critical implosion pressure for round composite shells:

\[
P_{\text{crit, anis}} = \frac{0.85 \sqrt{E_1 E_2}^{2.5}}{(1-\nu_{12}\nu_{21})^{75} LR_0^5 R_0} + \frac{1}{1+\sqrt{E_1E_2}t^3} \]  

(5)

The physical dimensions remain constant over the aging period, leaving longitudinal and transverse elastic moduli, Poisson’s ratios, and shear modulus. Because the collapse pressures varied by less than 6%, it can be surmised that these variables have also changed by an insignificant value. This conclusion is supported by Fontaine et al., where there were insignificant changes to tensile and shear moduli. Therefore, while there are differences in construction between filament wound tubes and vacuum infused laminate sheets, extreme pressures cause similar absorption and degradation behaviors. This degradation is minimal and can be accounted for by minor differences between the manufacturer’s stocks.

The max dynamic pressure shown in Table 2 indicates the peak magnitude of the pressure pulse emitted by the implosion phenomenon. Peak pressure is directly related to the speed by which the walls of the imploding specimen arrest themselves, causing a “rebounding” shock into the water moving with the walls. There is no significant trend in the distribution of this peak pressure, however there is a large range in the peak pressure. This large variance is likely due to the chaotic nature of composite fracture,
where in some specimens parts of the walls may arrest themselves while other sections continue to move, and in other specimens the walls do not fracture as severely and come to a halt more uniformly. Therefore, it can be concluded that peak pressure is insignificantly related to how long a composite is aged.

Figures 6-8 each show a set of five images of the implosion process, with each figure showing a different aging period. Figure 9 shows these same images side by side for ease of comparison. These images show that the speed at which the implosion front propagates is very similar; each experiment takes approximately 0.8ms to fully implode regardless of the aging time. Implosion velocity was also investigated. This was calculated by differentiating center point radial deflection. It was found that the radial velocity spanned a large range, from approximately 16 m/s to 24 m/s with an average just over 20 m/s and standard deviation of just under 3.2 m/s This spread did not follow any trend with aging. A velocity plot of three specimens, one from each aging time, are shown in Figure 10. These results are in contrast with the results by Javier et al. where the radial velocity of their virgin samples was less than half of that of their 70-day samples. This difference further supports the hypothesis that aging under these high-pressure conditions do not appreciably degrade the CFE tubes.
Figure 6: Virgin specimen (A01) implosion. Images go from -0.2 to +0.2ms in 1ms increments. The middle image, t=0, is when first wall contact is made.
Figure 7: 9-day specimen (C01) implosion. Images go from -0.2 to +0.2ms in 1ms increments. The middle image, $t=0$, is when first wall contact is made.
Figure 8: 24-day specimen (B01) implosion. Images go from -0.2 to +0.2ms in 1ms increments. The middle image, t=0, is when first wall contact is made.

Figure 9: A side-by-side view of Figures 6-8.
Figure 10: Center point velocity of one specimen from each aging time.

Figure 11 shows the radial velocity graph by Javier et al., taken from their paper. They measured the radial velocities as 6.8, 11.5, and 14.1 m/s for their H0, H35, and H70 experiments respectively, showing a clear trend of radial velocity increasing with aging. Furthermore, their most aged series has over twice the velocity as their virgin specimens.
Figure 11: Radial velocity of the experiments by Javier et al., taken from their paper.

3.3. Results of Shock-Initiated Implosion Testing

Explosive shock-initiated implosion experiments were performed to investigate the performance of aged CFE tubes under shock loading. The aging times and conditions are the same as those done for hydrostatic implosions: 0, 9, and 24 days at 6000 psi, 70°C.

When the explosive is detonated, a shock wave impacts the specimen, causing radial deformation. This first shock by itself is not necessarily enough to cause instability and initiate the implosion. The gas bubble caused by the explosion will pulse, expanding to
a maximum radius defined by the explosive strength and surrounding pressure, then collapsing on itself and emanating a pressure pulse, and may then re-expand and repeat the process several more times. These bubble pulse pressure spikes combined with the pressure reflections from the pressure tank walls were strong enough to initiate the specimen’s implosion if sufficiently degraded. If the specimen was not aged enough, they would undergo collapse by means of accumulated damage from repeated loading and the oscillation behavior [14]. For each experiment performed, the specimen did not collapse with the initial shock; each one showed some number of oscillations before imploding. However, there was no trend in the number of oscillations or quality of damage with relation to aging time. Javier et al. noted cavitation bubbles forming on the surface of their specimens, which caused initial damage when they collapsed. In their experiments, each set of aging experienced different levels of damage from the cavitation collapse, but the damage was significant enough to affect the implosion process. No cavitation bubbles were observed for the experiments done for this work, likely due to the slightly increased explosive standoff distance from 4.25 to 5.25 inches. Therefore, the only cause of damage would be from pressure waves from the explosive, the resulting bubble pulses, and their reflections.

As mentioned previously, Javier et al. concluded that implosion could be driven by the reflected pressure pulses combined with the shocks from the bubble pulses, or by accumulated damage depending on how far the specimens were degraded. To ascertain how far the high-pressure specimens were degraded, the time at which collapse started was found and compared to the corresponding pressure plot. Figure 12 shows the pressure plot of one of the virgin specimens. The first spike just after 0ms is the shock
from detonation. The reflection time inside this experimental vessel is approximately 1.4ms, at which a small amount of pressure activity is seen. The large spike just after 2ms is the first and only bubble pulse. The bubble does oscillate and “breathe” after this first pulse but does not implode with the vigor and violence that can be described as any additional true pulses. Reviewing the DIC data, the specimen does not begin to implode until approximately 4.8ms after detonation. Looking around 5ms in Figure 10, it can be seen that there are no particularly noteworthy features in the pressure signal, implying that the specimen failed due to accumulated damage by repeated loading and oscillations. This trend remains true for each experiment, with a single bubble pulse just under 2ms after detonation and the specimen imploding or otherwise breaking several milliseconds after that.

![Pressure graph of a virgin specimen (UA02) plotted against time.](image)

Figure 12: Pressure graph of a virgin specimen (UA02) plotted against time.
The time between detonation and specimen implosion, as well as the number of oscillations the specimen underwent before implosion, were tracked and checked for trends. There was correlation between the aging times and the time to implode, nor the number of oscillations. The period of oscillations also remained constant between all experiments, approximately 3.3 to 3.4 ms.

Figure 13 shows an interesting mode of damage in one of the virgin experiments. Instead of imploding normally where the tube simply flattens, the damage here is that the tube folded into itself when oscillating. There is nothing in the pressure signal or bubble behavior that would suggest this outcome. It is possible that in the small variations in experimental setup, that there happened to be a perfect overlap in how the specimen deformed and where and when an incoming shock impacted it. This phenomenon is not seen in any other experiment.

![Figure 13: Specimen UA03, with an unusual implosion where the back is blown in. The specimen is photographed in its bag for protection and containment of the sharp fibers.](image)

Neither the deformation behavior nor the collapse behavior follows any trend with aging time. This lack of a trend supports the conclusion that the material properties concerned with implosion were not significantly degraded in high pressure aging.
4. Conclusions

CFE tubes were aged in 3.5% saline at 6000psi and made to implode through hydrostatic and shock-initiated means. The intent was to study the effects of saline exposure at high pressure on the implosion and collapse behaviors of these structures. High speed digital photography, 3D digital image correlation, and high frequency pressure transducers were used to record the implosion events. Additionally, the results derived from this study are compared to previous studies which investigated: the behavior of CFE tubes after saline weathering in atmospheric pressure, and the behavior of CFE laminates after weathering in saline at 6000psi. From this study and comparing to previous work, the following conclusions were reached:

- The acceleration factor representing the factor of real-life service time to in-lab aging is 21.5. That is, one day of aging in lab is equivalent to 21.5 days in real life service.
- Exposure to saline for extended periods at 6000psi does not significantly affect the CFE tube’s critical implosion pressure. These results are supported by the conclusions of Fontaine et al. who noted that the elastic moduli of CFEs did not change significantly under identical aging conditions.
  - Comparing results against Javier et al. also support this conclusion because the large differences in collapse pressure noted by Javier et al. are not observed in this work.
- As a consequence of their material properties not changing significantly, the specimens subjected to UNDEX loading also behaved similarly across aging times.
• It is important to note that the reason by which the composites do not degrade is only speculative. Further specialized studies are required to confidently assert the physics of this phenomenon. Nonetheless, the CFEs have been discovered to negligibly change their behavior under external hydrostatic and UNDEX loading conditions when aged in 3.5% saline at 6000psi.
REFERENCES


MANUSCRIPT 2

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Effects of Long-term Seawater Exposure at Seafloor Depth Pressures on Carbon Fiber Epoxy Cylinders Behavior When Internally Pressurized

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ABSTRACT

This study investigates how extended submersion in high pressure seawater affects the behavior of carbon fiber epoxy tubes when internally pressurized. Accelerated life cycling was performed by submerging samples in 3.5% saline solution at 70°C for 0, 9, and 24 days pressurized at 6000psi. Using the Arrhenius equation, these times represent 0, 193.5, and 516 days of service. The events were recorded with two high speed cameras in a stereo configuration for 3D analysis. Results show that aging does not significantly affect the bursting pressure of these specimens, but does significantly affect the fracture propagation. This has implications on non-bursting modes of failure, such as leaking. It is suspected that this degradation mechanism is due to moisture absorption in the polymer matrix between layers of filament windings.
1. Introduction

Composite materials have seen an increasing amount of use in vessels and structures due to their high strength to weight ratio. Particularly for marine environments, composites are valued for having greater corrosion resistance compared to common structural metals. Mouritz et al. [1] discuss the increasing use of fiber reinforced polymer composites in naval warships and submarines for both component and their housings, and the vessel’s superstructure. There is also an increasing interest in unmanned underwater vehicles (UUVs) and remote operated vehicles (ROVs). Because these vehicles can exclude the life support and safety/design factors required with an on-board human operator, they can be made lighter, more compact, and be made to perform under more strenuous conditions. These increased capabilities require the strength and light weight that composites bring.

There has been an increasing interest in forward deployed structures, where vehicles and/or payloads may be stored near or on the ocean bed for years before being called into service. Unmanned and remote vehicles are the perfect candidate for these purposes because they can be made to be much more difficult to detect, as well as able to being able to be placed in such extreme conditions without the need to care for a human. Due to the time scales and pressures present in such a venture, there is a need to understand how the composite’s properties will be affected; a particularly important aspect to investigate is how these composites behave when internally pressurized, for example when used as storage tanks for hydrogen fuel cells or other high pressure gasses.
Water diffusion into resins and resin composites have been studied in pressures equivalent to shallow depths [2-5] and at pressures up to 50MPa, equivalent to 5000 meters deep [6-10]. Rice and Romatowski [11] studied the Arrhenius temperature dependence of water diffusion into polymers used as composite matrices for accelerated life cycling purposes, a technique that this study uses. Literature regarding the effects of pressure on diffusion are inconsistent and sensitive to the type of polymer in question, with some finding that pressure tends to increase saturation mass yet leave diffusivity unchanged [12], while others found that both saturation mass and time to saturation increased with pressure [13].

This study closely follows the work done by Fontaine, Leblanc, and Shukla [14], which explored the behavior of vacuum infused CFE laminate sheets after prolonged exposure to high pressure saline. To the author’s knowledge, no work has been done on exploring CFE cylinder explosions after prolonged exposure to a pressurized saline environment. This study uses a novel high-pressure aging facility housed in the University of Rhode Island’s Dynamic PhotoMechanics Laboratory to expose specimens to high pressure saline at elevated temperatures. The specimens were affixed in an open-top tank filled with water, and manually pressurized with water. A digital and analog pressure gauge read the line pressure to determine the approximate bursting pressure. Two high speed cameras are used to capture the swelling and explosion. It was found that the aging process degraded the composites, reducing their ability to hold pressure. It is likely that this mechanism of degradation is by means of moisture ingress into the polymer bands between layers of filament windings.
2. Materials and Methods

2.1. Specimen Details

The CFE composite tubes used in this study are from RockWest Composites (West Jordan, UT). These tubes have a nominal inner diameter of 1.375 inches and outer diameter of 1.5 inches (part number 35050-S). Specimens were cut to be 15 inches long with a wet abrasive saw. Ends were sealed with endcaps intruding 0.5 inches into the tube from each side, for a nominal unsupported length of 14 inches. Table 1 shows the layup details and resin of the tubes. The fibers used are standard modulus carbon fibers.

Table 1: Layup details of RockWest Composite's 35050-S

<table>
<thead>
<tr>
<th>RockWest 35050-S</th>
<th>Layup:</th>
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<tr>
<td></td>
<td># Circuits:</td>
</tr>
<tr>
<td></td>
<td>12</td>
</tr>
<tr>
<td></td>
<td>0.45 Lead</td>
</tr>
<tr>
<td></td>
<td>8</td>
</tr>
<tr>
<td></td>
<td>13</td>
</tr>
<tr>
<td>Nominal Dimensions (mm):</td>
<td>38.1OD x 1.59Wall x 381L</td>
</tr>
<tr>
<td>Resin:</td>
<td>Epon 862/Lindride 6K</td>
</tr>
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</table>

2.2. Calculation of Acceleration Factor

Previous research [6, 14] suggests that degradation of the composite is primarily due to water diffusion into the polymer matrix. Under this assumption, aging can be
accelerated by increasing the rate at which water is absorbed. Rice and Ramotowski [11] developed a method based on the Arrhenius relationship of rate of diffusion to temperature. The Arrhenius relationship as it applies to the diffusivity, $D$, of water into the matrix is:

$$D = D_0 e^{-\frac{E_A}{RT}}$$  \hspace{1cm} (1)$$

where $D_0$ is a constant, $R$ is the ideal gas constant, $T$ is absolute temperature, and $E_A$ is the activation energy. The activation energy is assumed to be a function of material and environment; both are held constant for this study, so it is assumed that the activation energy is constant as well. This relationship and assumption allows for calculating the reaction rate at any temperature once diffusivity is found.

Figure 1: Small pressure vessel used for aging samples for the diffusion study.

To calculate diffusivity, Crank’s [16] method to calculate $D$ from a range of temperatures is used. Three tests were performed with small samples cut from the same tube stock as the experimental samples. These samples were desiccated for at least 48 hours to ensure a moisture-free baseline, and then immersed in 3.5% saline pressurized
to 6000psi in the small pressure vessel shown in Figure 1, heated to 18, 45, and 65°C. These samples were periodically removed from their bath and their mass change recorded by a scale with a precision of $10^{-4}$ grams; it is assumed that any mass change is solely due to water absorption with no leaching or chemical reactions occurring between the composite and the water or the pressure vessel. Absorption curves of time to percent mass change were plotted for each test. It is reasonable to assume the diffusion is Fickian and one dimensional because each sample is thin, with a face area much greater than the edge area. Under these conditions, Crank calculated the diffusivity to be

$$D = 0.049 \times \frac{l^2}{t_{50}}$$

(2)

where $l$ is the tube’s wall thickness and $t_{50}$ is half the time it takes for the sample to reach full absorption. Based on the assumption that there is no chemical change or leeching by the composite, it is safe to assume that $t_{50}$ is independent of temperature within the same conditions. With the diffusivity at each of the three temperatures known, Equation 1 can be rewritten as:

$$\ln(D(T')) = \ln(D_0) - \left(\frac{E_A}{R} \right) \left(\frac{1}{T'} \right)$$

(3)

A linear regression can be drawn when plotting inverse temperature against the natural log of diffusivity. The slope of this line is $-E_A/R$ which is assumed to be constant. Acceleration factor can be found by taking the ratio of diffusivity between the laboratory aging temperature, $T_2$, and the service temperature, $T_{ref}$ as follows:

$$AF = \frac{D_{0e} e^\left(\frac{E_A}{RT_2} \right)}{D_{0e} e^\left(\frac{E_A}{RT_{ref}} \right)} = e^\left(\frac{E_A}{R} \right) \left(\frac{T_2-T_{ref}}{T_{2}^{\ast}T_{ref}^{\ast}} \right)$$

(4)
For this study, $T_{\text{ref}}$ is taken to be 3.0°C, the commonly accepted value for seawater in the abyssal plain.

2.3. High Pressure Aging Facility and Aging Methodology

A custom vessel, shown in Figure 2, made to handle high pressure, high temperature saline was designed and manufactured in-house at the Dynamic PhotoMechanics Laboratory. Specimens to be aged were first desiccated for at least 48 hours, then placed in the vessel with 3.5% saline, and pressurized to 6000psi. The pressurized vessel was then placed in a large water bath heated to 70°C. This temperature was chosen in order to stay below the glass transition temperature of the composite at 82°C. This process was followed for two batches of samples: one for 9 days and the other for 24 days.

In the process of pressurizing the 24-day specimens, the pressure vessel became damaged and lost pressure on the 9th day. The specimens were extracted from the vessel and placed in a refrigerated saline bath to prevent the specimens from drying out and stall the absorption process. The specimens remained in this bath for 11 days while parts were ordered and repairs performed. Once repairs were completed, the specimens were replaced in the vessel and repressurized for the remaining 15 days. This duration out of pressure is assumed to have a negligible impact on the moisture absorbed due to the chilled temperature.
2.4. Hardware and Software used for Capture and Analysis

A high contrast, random, speckled black dot pattern on a white background was painted on each sample by hand after the high pressure aging process described in Section 2.3. This pattern was recorded by two Photron FASTCAM Nova S-12 high speed cameras at 250 frames per second in a stereo imaging setup. The dots were sized and spaced to fill approximately 5 pixels diameter when viewed by the cameras. These stereo images were analyzed in Correlated Solution’s VIC-3D 8 analysis software.
2.5. Setup of Experimental Facility and Experimental Procedure

A square, open top, steel tank is housed in the Dynamic PhotoMechanics Laboratory. It is approximately 4ft a side, with angled viewports on one side allowing two cameras to be arranged in a stereo imaging configuration. The tube specimens were sealed with external endcaps and O-rings, held together by three steel rods. One of these caps had a port allowing for pressurized water to enter. Figure 3 shows a basic illustration demonstrating how the specimen fits into its endcaps. Figure 4 shows an approximation of the setup, illustrating the general locations of viewports in the square tank, the position of the cameras, the means of pressurization, and the specimen in its fixture.

![Figure 3: Cross section of specimen with endcaps, not showing speckles, steel rods, or pressure connector. Pressure inlet is on the left endcap.](image)

This specimen assembly was fixed in a rigid frame inside the tank such that the speckle pattern described in Section 2.4 was visible to the cameras. A manual hand
pump was used to flow pressurized water into the specimen until the specimen burst or the pressure dropped significantly. An analog and digital pressure gauge were linked to the pump to determine this change in pressure. The digital gauge was recorded to determine the max pressure experienced by the specimen before pressure loss or burst, accurate to +/- 1%.

![Tank Setup Illustration](image)

*Figure 4: An illustration of the square tank setup used. Location of the cameras (tripod mounts not shown), specimen in fixture, and pump system are shown. Viewports are sealed with clear polycarbonate.*

3. Results and Discussion

3.1. Results of the Diffusion Study

For each test, each specimen was immersed in saline solution pressurized to 6000psi and maintained at the test temperature, and the mass gain periodically plotted against time to create the absorption curves. The saturation mass gain was found to be 0.44%,
reached by the 65°C sample after approximately 450 hours, making half the saturation mass gain to be 0.22%. The saturation mass gain is assumed to be constant for each test because the temperature was kept under the glass transition, and the test conditions kept constant between each test. To use Equation 2, we require the time at which the mass gain reached 0.22%. For each test, a logarithmic regression was plotted with a strong fit: the coefficient of determination ($r^2$) being over 0.91 for each test. From here, it is a simple matter to find $t_{50}$ with the regression equation. Using Equation 4, $T_{\text{ref}}$ was set to 3°C and $T_2$ to 70°C resulting in an acceleration factor of 21.5. That is, every day aging at 70°C in laboratory conditions is assumed to be equivalent to 21.5 days in service at 3°C.

3.2. Results of Internal Pressurization Tests

Table 2 shows the bursting or failure pressure of each experiment.

Table 2: Summary of experiments. This table shows the approximate pressure each tube exploded or lost pressure in pounds per square inch (psi).

<table>
<thead>
<tr>
<th>Tube Stock</th>
<th>Virgin</th>
<th>9-day</th>
<th>24-day</th>
</tr>
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<tbody>
<tr>
<td>A</td>
<td>2700</td>
<td>2100</td>
<td>2600</td>
</tr>
<tr>
<td>B</td>
<td>2600</td>
<td>2300</td>
<td>2500</td>
</tr>
<tr>
<td>C</td>
<td>2400</td>
<td>2650</td>
<td>1800</td>
</tr>
<tr>
<td>Average</td>
<td>2567</td>
<td>2350</td>
<td>2300</td>
</tr>
</tbody>
</table>

Two specimens, 9-day tube A and 24-day tube C, leaked before bursting. Small fractures developed through the tube thickness and allowed water to leak through. Besides these two experiments, the other failure pressures are largely similar. However,
the characteristics and extent of the fractures noticeably change with aging. The virgin specimens show almost exclusively a clean spiraling fracture, appearing to be a delamination of fiber layers. As specimens are aged, the fracture line becomes increasingly irregular and discontinuous, with damage evolving to include fibers breaking and tearing.

*Figure 5: Close-up of virgin tube A, showing spiral fracture and delamination instead of through cracking.*

The fractures in the virgin specimens all occur similarly, Figure 5 shows an example image of the damage. The spiral fracture down the length of the tube is likely following some of the filament winding pattern.

There is significant variation between the 9-day series’ damage. Tube A lost pressure before burst, indicating there were small, discontinuous fractures that don’t create visible damage. Tube B, shown in Figure 6, has nonlinear fractures that branch and split. Additionally, there is significant fiber tearing at the surface whereas the virgin specimen showed almost exclusively delamination at the surface. Figure 7 shows damage on tube C superficially similar to the virgin specimens: a constant nearly unbroken spiraling fracture, delamination, and little fiber tearing. However, the delamination seen is more extensive than that seen in the virgin specimens.
Figure 6: Close-up of two sections of 9-day tube B. Both images show fiber tearing and branching or zigzagging fracture.

Figure 7: Close-up of 9-day tube C. Delamination appears to be the majority of the damage, very similar to the virgin specimens.

The damage in the 24-day specimens appear as more exaggerated versions of the damage seen in the 9-day specimens. Tube C leaked before bursting, in a similar manner to 9-day tube A. The images shown in Figure 8 show the fracture surface of 24-day tube A. In addition to the delamination damage, best seen on the left side of the first and last pictures of Figure 8, there is extensive fiber tearing and discontinuous fractures. In the middle picture, the fracture is seen to propagate circumferentially, with the damage appearing to be an even mix of delamination and fiber tearing. The damage in 24-day tube B is very similar.
Fontaine et al. [14] noted that when CFE specimens were aged under high pressure saline, their in-plane properties remained relatively unchanged, whereas the flexural modulus was degraded. In the layups he used, the flexural modulus is primarily controlled by interlaminar properties which are suspected to have degraded. It is suspected that the reason only the out-of-plane properties are degraded is that there is a larger concentration of matrix imperfections between layers of fibers, and the imperfections present between fibers are likely to be smaller on average. Under high pressure, these smaller voids are more easily compressed such that moisture uptake is
much more difficult, whereas the higher concentration of imperfections and voids between fibers can still absorb moisture.

This hypothesis is consistent with the results presented here. Fracture in fiber reinforced composites are difficult to predict; they are anisotropic, nonhomogeneous, and they may have small manufacturing defects that are not detectable or significant for common use but become important when loaded to their extreme limits. Furthermore, their failure limits change from batch to batch. If the polymer matrix were to fail in small and independent microfractures, it can create a leaking scenario as seen in 9-day tube A and 24-day tube C where there is no apparent damage but the composite is compromised. However, if the fractures in the matrix grow into one larger fracture, the parts can suddenly “snap” apart and impart a strong impulse on the fibers bridging the fracture, causing them to tear as well. This is likely the case seen in the 9-day tube B specimen for example. Furthermore, if the composite is sufficiently degraded, the damage can become increasingly erratic, with messier fracture lines as seen in the 24-day specimens.

Images from the high-speed photography were analyzed and checked using DIC techniques for how the specimens swelled and changed radius when pressurized. However, these results are inconclusive. CFE composites are brittle, exhibiting very little elastic or plastic deformation. The small expansions detected by the DIC are less than a 4% change: at most slightly under 1mm of radial expansion.
Examining Figure 9, there is no indication as to where or what direction the fracture seen in Figure 10 will appear. For some specimens, DIC was able to detect small diagonal deformations seeming to follow the filament winding pattern as seen in Figure 11 where the color contours can clearly be seen moving diagonally upwards right. However, these contours were seen to have no relation to the actual fractures that occurred, as in the same specimen shown in Figure 12 with a three-way fracture occurred, with two of the branches seeming to be almost parallel and perpendicular to the contour and the third at a supplementary angle to the crack parallel to the contour. When combining the results of the DIC analysis with the conclusions drawn from examining the fracture surface, it can be concluded that deformations throughout the pressurization process are not a reliable method for determining the fracture location or fracture pressure of these CFE composite tubes. Small, undetected defects combined
with the brittle nature can cause fractures to propagate in directions DIC analysis cannot predict.

Figure 11: 9-day specimen 2 (tube B2) one frame before bursting. The max and min dR (change in radius) are -.066 and .422mm.

Figure 12: 9-day specimen 2 (tube B2) one frame after fracture.

Figure 13: 24-day specimen 1 (tube A1) one frame before bursting. The max and min dR (change in radius) are .432 and .036mm.

Figure 14: 24-day specimen 1 (tube A1) one frame after fracture.

The swelling of each experiment was also investigated to determine if aging had an effect on how the composite bulges when pressurized. No trend was found in the magnitude of swelling: besides the specimens that leaked and virgin tube B, all
specimens showed a radial deformation of approximately .4 to .5mm the instant before fracturing.

4. Conclusions

CFE tubes were aged in 3.5% saline at 6000psi and pressurized internally until they lost pressure by leaking or bursting. The intent was to study the effects of saline exposure at high pressure on how these materials can hold internal pressure and their mechanism of failure. High speed digital photography and 3D digital image correlation were used to record the pressurization process. Additionally, the results derived from this study are compared to previous studies which investigated: the behavior of CFE tubes after saline weathering in atmospheric pressure, and the behavior of CFE laminates after weathering in saline at 6000psi. From this study and comparing to previous work, the following conclusions were made:

- The acceleration factor representing the factor of real-life service time to in-lab aging is 21.5. That is, one day of aging in lab is equivalent to 21.5 days in real life service.
- Exposure to saline for extended periods at 6000psi may affect the CFE tube’s ability to hold internal pressure. These results are supported by the conclusions of Fontaine et al. where the out-of-plane properties are degraded by this exposure, suggesting that degradation occurs largely in the matrix, especially in between fiber bands.
- While the fracture pressure of aged specimens does not appear to significantly decrease, failure by leaking before burst may occur at increasingly lower
pressures. Furthermore, the characteristics and extent of the fractures become more irregular and apparent across the surface of the specimen as aging progresses.

- The brittleness and anisotropic properties of composite tubes make it impossible to determine the location and direction of fracture when internally pressurized.

- It is important to note that the reason by which the composites do not degrade is only speculative. Further specialized studies are required to confidently assert the physics of this phenomenon.
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APPENDICES

Appendix 1: The Evolution of Work done for Manuscript 2

This appendix is intended to be a brief discussion on the evolution of the process performed for Manuscript 2.

The intent of this research is to investigate how composite overwrapped pressure vessels (COPVs) would degrade and behave when exposed to saline for long durations. Originally, the method of experimentation was to manufacture or obtain glass fiber vinyl ester composites overwrapping a thin aluminum shell. These pressure vessels were to be pressurized with helium until rupture. The aging process would have been performed under atmospheric pressure as opposed to under the high pressures described in these manuscripts.

The primary pitfall in this direction was that the energy released in this explosion would be massive, akin to several grams of high explosive depending on the size of the specimen. The thinnest available aluminum tubes in the weakest alloys commercially available by themselves required pressures over 600psi to burst. The ultimate tensile strength of 3003-H14 aluminum is approximately 22ksi. Using the hoop stress equation with thin wall assumptions, a .035 inch thick tube of 2.5 inches diameter would burst at 616psi. If this tube were 5 inches tall it would have approximately 6.3kJ of energy if the gas were to expand isothermally, equivalent to approximately 1.5g of TNT. It was predicted that any amount of composite overwrap would push the burst pressure to over 1000psi dramatically increasing the energy, presenting a potential danger to laboratory equipment and the researchers.
Furthermore, the pressurized gas available in the laboratory can only go to 2000psi. As can be seen by the failure pressures noted in Table 2 of Manuscript 2, all pressures are over 2000psi for unlined carbon fiber tubes.

It was then suggested that extremely thin aluminum tubes be used, and overwrapped with a single layer of filament winding (the filament winds once up the specimen, and once more back down for a complete circuit). Standard unassembled beverage cans .004 inches thick, made of 3003/3004 aluminum were obtained for this purpose. These cans had their tapered tops and bottoms cut off to become cylinders. These cylinders along with a custom rigid mandrel were sent to Nor’Easter Yachts in Milford, CT for custom filament winding. This winding was satisfactorily done, and the specimens were internally pressurized with nitrogen. These specimens failed in a way inconsistent with what a COPV was expected to display. The single layer of wrapping was relatively flexible, able to be deformed by hand pressure. The composite separated and delaminated easily under the strain (Figure A1), but the fibers themselves were largely undamaged; instead of bursting the liner and composite overwrap, the nitrogen instead simply broke through the liner and seeped through the separating filaments (Figure A2). In one specimen, neither the liner nor composite tore and separated. Instead. The liner developed many parallel, short (approximately 3mm long) fractures barely visible to the naked eye. Nitrogen seeped through these small fractures and through the vinyl ester matrix which had also developed similar microcracks under the expansion strain (Figure A3). It was deemed unwise to wind more layers of composite for fear that it would increase the bursting pressure too much.
Figure A1: A filament wrapped can whose fibers have severely separated.

Figure A2: A filament wrapped can with very minimal overwrap damage. The matrix simply cracked and distorted the fibers without tearing.
After reviewing the risks and failures described, a decision was made to perform the experiment described in Manuscript 2 with the same material as was used in Manuscript 1, aged under the same conditions. Additionally, the specimens would be pressurized with water to prevent the violent explosion that would follow a highly energetic air-powered burst. This experimental design also takes advantage of the unique high-pressure weathering tank available in the Dynamic PhotoMechanics Laboratory. Instead of two unrelated experiments, more correlations and conclusions can be drawn from investigating the same material given different loads.


