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**Effects of Water Saturation and Low Temperature Coupling on the Mechanical Behavior
of Carbon and E-Glass Epoxy Laminates**

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Effects of Water Saturation and Low Temperature Coupling on the Mechanical Behavior of Carbon and E-Glass Epoxy Laminates

ABSTRACT

An experimentally based study has been conducted to quantify the effects of coupled water saturation and low temperatures on the quasi-static and dynamic mechanical behavior of E-Glass and Carbon Epoxy laminates. The relative performance of the materials as a function of water saturation and decreasing temperature was characterized through detailed experiments, specifically in-plane (tensile / compressive) and shear material properties, static and dynamic Mode-I fracture, and impact / flexure after impact strength. In the investigation temperatures from Room Temperature (20°C) down to arctic seawater and extreme ocean depth conditions (-2°C) were evaluated. The materials utilized in the study, Carbon/Epoxy and E-glass/Epoxy, are chosen due to their primary interest to the underwater vehicle and marine industry communities. The results of the quasi-static and dynamic material experiments show that all properties are affected by both water saturation and decreasing temperature, although the trends are specific to the property under consideration.

1. INTRODUCTION

Advanced composite materials exhibit desirable characteristics which make them viable candidates for utilization in harsh marine environments. These materials produce components that have high strength to weight ratios, lighter overall weight, superior resistance to corrosion, and overall reductions in required maintenance. However, there are also unique considerations

that must be taken when using these materials in the design of a structure. These include reduced resistance to impact damage and cyclic loading conditions, post damage residual strengths, complex failure and damage mechanisms, repair difficulty, and stringent certification processes. The understanding of the performance of these materials when operating in extreme marine environments is key in ensuring a structurally adequate design that will retain robustness throughout the intended life cycle. The focus of this paper is on the conditions found in arctic environments, deep ocean depths, and applications with long seawater exposure times such as drilling operations. The conditions associated with these environments include near freezing seawater temperatures and water ingress into the structural laminates. The objective of the research is to quantify the mechanical and damage behavior changes of these advanced material systems due to water ingress, and decreasing temperature down to the lowest expected water temperatures of the arctic / deep ocean environments. The specific quantities of interest presented in the study consist of those that potentially affect operating capabilities of submerged components/structures arising from low temperature/water ingress exposures, namely material strength (tensile, shear, compression), damage resistance (fracture toughness, crack propagation/stability), and impact loading resistance.

The understanding of the complex mechanical and fracture characteristics of advanced composite materials is generally much more limited than the knowledge associated with historical metallic materials. This limited understanding can be compounded by the introduction of use in long term submergence and low temperature conditions. The use of polymer based materials in cold and arctic temperature environments results in higher stiffnesses but oftentimes

lower toughness properties which may lead to the onset of unique failure mechanisms. When considering the design of structures and vehicles comprised of composite materials to be operated in a cold water environment there is a certain need to understand the unique relationships between lamina stresses and distributions, the mechanics of the interlaminar ply interfaces, water absorption behavior, and fracture properties. In low temperature cases where impact type loading, such as wave slap or collisions, is a concern there is a greater desire to improve the fracture toughness of the materials and associated laminates. These material choices and characterizations are typically made and driven by the results of detailed laboratory testing in a controlled environment. Future advancements in the knowledgebase of the effects of arctic ocean type temperatures and long term water saturation on the failure characteristics of advanced composite materials will further the expanded use of these material in marine applications.

Prior investigations of arctic environment temperatures on composite materials have primarily been conducted on dry laminates at temperatures down to the cryogenic regime. Walsh et al. [1] evaluated the effects of temperatures down to 4K on the tensile, compression, and shear characteristics of an E-Glass/Epoxy laminate and reported increasing strength and stiffness with decreasing temperatures. Kim et al. [2] evaluated the cryogenic temperature effects on a T700 Carbon/Epoxy laminate, including thermo-mechanical cycling, and found slight increases in material stiffness with corresponding decreases in material strength with decreasing temperatures down to -150°C. The influence of low temperatures on the dynamic behavior of composite materials have been studied by several researchers. Sanchez-Saez et al. [3] investigated the effects of temperatures down to -150°C on the static and dynamic flexural behavior with a three-point bend configuration. Consistent with prior studies, the strength and energy capacity decreased with decreased temperature with the flexural modulus being relatively unaffected.

Ricciardi et al. [4] considered the effects of temperatures down to -50°C on the interlaminar shear strength and the flexure under impact performance of a woven basalt fiber based laminate. The study showed increased stiffness of the laminates but also a corresponding increase in delamination damage with decreasing temperature. Garcia-Gonzalez et al. [5] performed low velocity impact experiments on short carbon fiber reinforced PEEK laminates at temperatures ranging from 25°C to -75°C . The study identified a ductile-to-brittle transition in the material damage and a quantifiable decrease in energy absorption at the lower temperatures.

An investigation conducted by Kichhannagari [6] highlighted that at low temperatures there is a larger level of material micro-cracking for specimens subjected to both uniaxial and biaxial loading conditions as compared to ambient temperature conditions. This was determined to result from matric shrinkage and resulting residual laminar shear stresses due to thermal contraction. The presence of these interlaminar microcracks was found by several researchers [7, 8, 9] to increase material permeability which results in path initiation for absorption of moisture and water with associated material swelling. When subjected to multiple cycles of freezing and thawing in a saturated condition, this swelling effect can serve as the source of stresses that are environmentally induced. Coalescence of these microcracks into meso- and macroscale groupings has the pronounced effect of reducing the materials fracture toughness and associated damage performance. A full working knowledge of the freeze and thaw cycle effect is required to support the design and construction of composite structures in low temperature environments. The consideration of fracture toughness performance is of greater concern when impact type loading is expected to be imparted to a composite structure, particularly in low temperature ocean environments. These types of loading events are well suited for evaluation in a controlled laboratory environment through drop tower impact experiments. Results of these

tests may be further utilized for material selection optimization by the understanding of the rate dependent fracture toughness characteristics.

2. MATERIALS

The materials utilized in the current study are consistent with those evaluated in [10] to allow for for meaningful comparisons. The fabrics are balanced in the warp and weft directions and are of a greige (untreated) condition. The E-Glass and Carbon fabrics have respective areal weights of 190 g/m^2 (5.61 oz/yd²) and 199 g/m^2 (5.88 oz/yd²). All laminated panels utilized ProSet 125/226 laminating epoxy and were manufactured in a press with curing conducted at 82°C (180°F) under 140 kN (14 tons) of load. Panels were manufactured in plate thicknesses of 2.54 and 5.08 mm (0.1 and 0.2 in.) to support the specifications of the pertinent test standards.

Figure 1 - Plain Weave Fabric Architecture (Courtesy of JPS Composites Technical Reference Handbook [11])

3. WATER SATURATION METHODOLOGY

The investigation into the effects of water ingress on the mechanical behaviors of FRP composites required that the test specimens were immersed in a water bath until fully saturated. This water-saturated condition mimics the effects of long-term water submersion for a period of months. The specimens were placed in a bath of distilled water over a period of 4 to 6 months

until they reached saturation. All specimen surfaces were in contact with water during submersion in the water bath. To accelerate the soaking time, the water baths were placed into ovens at 45°C. The temperature of 45°C was chosen since it is below the FRP composite's epoxy glass transition temperature of 80°C previously determined experimentally in [10]. This lower temperature will ensure that the epoxy does not experience physical changes during soaking. To determine when the specimens became saturated, the mass of representative samples from each specimen type was monitored throughout the soak until the change in mass approached a plateau dictated by Fickian diffusion analytical modeling. The samples were held in the water baths until each mechanical test was performed.

4. MECHANICAL CHARACTERIZATION EXPERIMENTAL APPROACH

The experimental methods utilized in the current study to evaluate the effects of water saturation and low temperatures on the Carbon and E-Glass laminates are consistent with those previously utilized by the authors in [10]. The full details of the mechanical characterization test methods are contained in the previous publication and are summarized in the following section for brevity.

4.1 Tension / Compression / Short Beam Shear

The effect of coupled water saturation and low temperatures on the stiffness and strength properties of the respective laminates was characterized through controlled laboratory testing consisting of tension, compression, and short beam shear. These material tests were performed in accordance with ASTM D638 (Tension) [12], ASTM D3410 (Compression) [13], and ASTM D2344 (Short Beam) [14]. Each respective material characterization test (stiffness and strength) was performed at temperatures of 20°C, 5°C, and -2°C. An Instron® machine operated in

displacement-controlled loading was used in each of the test series and for the cases on low temperatures, an environmental chamber utilizing liquid nitrogen as the cooling source was employed. Prior to the respective mechanical testing all specimens were “Thermally Soaked” for a minimum of one hour in the cooling chamber to bring the specimens to the desired temperature. The details of individual test configurations is shown in Figure 2.

Figure 2 – Tensile, Compressive, and Short Beam Shear Test Setups

4.2 Static Mode I Fracture

The critical strain energy rate, G_{IC} , dependence on temperature and water ingression was evaluated through Mode-I fracture tests conducted at a quasi-static load rate. Testing of each laminate conducted at temperature values of 20°C, 5°C, and -2°C. The double cantilever beam specimens with a geometry of 22.80 cm long by 2.54 cm wide (9 in. by 1 in.) were used in all cases. A precrack was induced in the DCB specimens in the form of a 7.62 cm (3 in.) Teflon insert positioned at the mid-plane of the laminate. The real time crack length and crack growth stability were measured through the use of fracture gages (Vishay TK-09-CPC03-003/DP)

bonded to the specimen edges ahead of the crack front. The crack gages consist of 20 individual strands with a spacing of 2 mm perpendicular to the crack propagation direction and facilitate the calculation of G_{IC} . The strands in the gage are ruptured as the crack tip passes through the strand during propagation and the corresponding change in voltage is recorded by the acquisition system. The corresponding load and deflection histories are also recorded by the data system connected to the test machine. The combined data set consisting of force, displacement, and crack length are utilized to characterize the G_{IC} values through the use of the Modified Beam Theory (MBT) formulation provided in [16]. The governing MBT equation is given as:

$$G_{IC} = \frac{3P\delta}{2wa} \quad (1)$$

where P is the load, δ is the extension, w is the specimen width, and a is the crack length.

Figure 3 – Mode-I Fracture Test Configuration and Typical Load vs. Extension Behavior

4.3 Dynamic Mode-I Fracture

The dynamic fracture characteristics of the Carbon and E-Glass laminates were obtained using an advanced experimental approach. Dynamic fracture toughness was determined

using the wedge-insert-fracture (WIF) method [17], which utilized a double cantilever beam specimen geometry combined with a unique Split Hopkinson Pressure Bar (SHPB) setup shown in Figure 4. The specimen had a width of 12.70 mm (0.5 in.) and a thickness of 5.08 mm (0.2 in.) with a Teflon film insert used to create a precrack at the mid plane of the panel. The incident bar was 9.5 mm (0.375 in.) in diameter, 162 cm (64 in.) in length and was made of brass. The portion of the wedge insert in contact with the incident bar had a 12.7 mm (0.5 in.) outer diameter and the flat end in contact with the specimens was semi-cylindrical with a diameter of 3 mm (0.118 in.). The initial crack length (distance from the center of the semi-cylindrical tip of the inserted wedge to the crack tip), was 4.7 cm (1.85 in.) and 4.2 cm (1.65 in.) for the Carbon/Epoxy composite and E-Glass/Epoxy composite, respectively. The full theoretical derivation of this approach is provided in [10]

The experiments conducted at 5°C and -2°C utilized a wooden cooling box with glass fiber insulation as shown in Figure 5. Compressed air circulated in a copper tube, immersed in dry ice, was used to cool the specimen in the cooling box. The desired specimen temperatures were achieved by controlling the flow rate of the compressed air with real-time measurements of the temperature in the box recorded using an OMEGA K-type thermocouple. Upon the specimen reaching thermal equilibrium with the air in the cooling chamber, the striker was released to create the compressive pulse in the incident bar. Clay was used as a pulse shaper on the striker end of the incident bar to eliminate the high frequency components of the compressive pulse, thus effectively suppressing vibrations on the specimens caused by impact loading.

Figure 4 - Dynamic Mode-I Fracture Setup using Split Hopkinson Pressure Bar

Figure 5 - SHPB Cooling Box Schematic

The Mode-I critical strain energy release rate, G_{Icd} for the WIF specimen is calculated from Equation 2, using the maximum value of strains attained. In these experiments and in lieu of using the strain rate, the loading rate was calculated as the time derivative of the energy release rate.

$$G_{Icd} = \frac{h\varepsilon_s^2 E_{xx}(a+e)^2}{3a^2} \quad (2)$$

where h is the plate half thickness, ε_s is the maximum strain from the strain gage, a is the initial crack length, E_{xx} is the Young's modulus in the longitudinal direction, e is a correction factor for local deformation around the crack tip and taken to be 3 mm (0.118 in.) in these experiments

4.4 Impact and Flexure after Impact

The effect of water saturation coupled with decreasing temperature on the impact response of the respective composite materials was quantified through a two part test series. The samples were first impacted through the use of a instrumented drop tower to induce damage and was followed by a 4-point flexure test of the damaged samples. This approach is identical to the method

previously utilized in [15] with the primary objective for the impact tests being to generate barely visible impact damage (BVID) levels in the test specimens. A summary of the procedure is provided as follows.

A Instron Dynatup® Model 9210 instrumented drop test machine was used in the conduct of the impact tests. The specimens were of dimensions 139.7 mm x 12.7 mm x 0.508 mm (5.5 in. x 0.5 in. x 0.2 in.) and had the material warp direction oriented along the length of the samples. The drop tower impact head was fitted with a 6.35 mm (0.25 in.) radius line-load impactor and an accelerometer recorded the force time history during the impact event. During the impact testing the samples were configured in a three-point flexure arrangement with a span length between supports of 5.08 cm (2 in.) as shown in Figure 6. The objective purpose of the drop tower test was to: (1) record the force-time history of the impact head during contact with the composite specimens and (2) impart an observable level of damage to the specimens for the subsequent four-point flexural after impact testing. All specimens were “Thermally Soaked” for a minimum of one hour in a low temperature chamber to bring the specimens to the desired temperature after which the drop tower test was immediately conducted. The impacted specimens were then returned to the thermal chamber to maintain the temperature prior to the subsequent flexural tests. Preliminary test runs were conducted at room temperature to determine the appropriate levels of impact energy that result in BVID but short of complete failure of the specimens. These trials identified the energy levels to establish an acceptable energy threshold (AET) for producing BVID. The impact energy was determined for the room temperature specimens and used for the low temperature tests. The respective impact energies used in the study were 5.3J for the Carbon laminates and 8.6J for the E-Glass laminates. The post-impact material damage

levels for the drop test conducted at 5°C for the Carbon and E-Glass laminates are shown in Figure 7.

Subsequent to the conduct of the impact testing a four-point flexural bending test was conducted on each sample to quantify the coupled water saturation and low temperature effects on the residual flexure strength after impact of the composite materials. The four-point flexure loading was conducted within a thermal chamber connected to the Instron test machine with the flexure test temperature corresponding to the drop tower test. The span length used in the flexure tests was 114.3 mm and the span between loading supports was 38.1 mm with the loading fixture shown in Figure 6.

Figure 6 – Drop Tower Impact and Flexure after Impact Test Setups

E-Glass

Carbon

Figure 7 – 5°C Impact Test Damage Levels

5. EXPERIMENTAL RESULTS

5.1. Tensile and Compressive Modulus and Strength

The stress-strain behavior of the saturated and dry Carbon and E-Glass laminates under tension and compression loading are shown in Figure 8 for the tests performed at 5°C. While the five specimens tested in each condition showed high repeatability, only a single representative specimen is shown for clarity of the respective plots. While specific details of the material performance under each type of loading are discussed in the subsequent section this plot highlights the key differences in relative material behavior. Firstly, Both the Carbon and E-Glass are significantly stiffer in tension than compression, as well as have larger tensile strengths than compression strength. However, It is also noted that the compressive behavior of both materials, while less stiff, exhibits nearly two times the overall toughness than the corresponding tensile behavior.

Figure 8 - Tension and Compression Behavior of Carbon and E-Glass Laminates at 5°C

The influence of low temperature coupled with full water saturation on the tensile modulus and strength of the Carbon and E-Glass laminates are shown in Figure 9. All tensile tests on the saturated laminates were performed at 20°C, 5°C, and -2°C for consistency with the results presented previously in [10]. In all cases, both dry and fully saturated, the testing exhibited a linear material response up to failure with a brittle and rapid failure exhibited at the point of maximum loading. Consistent trends are evident in material moduli and strength responses over the temperature range considered in the study for the saturated and dry conditions, although the magnitudes are affected by water ingress. The mechanical characterization results highlight that in terms of stiffness performance, the Carbon is on average five times stiffer than the corresponding E-Glass laminate in both dry and saturated states. In terms of strength comparisons, the Carbon is nearly 2 times as strong as the E-Glass in the dry condition and 3 times as strong in the saturated condition. The difference in relative ratios in the strength is due to the significant drop in E-Glass strength in the saturated state.

The tensile results showed that across the temperature range of consideration, 20°C to -2°C, the Carbon and E-Glass laminates both exhibit reductions in modulus and strength when saturated with water. Examining the trend in the Carbon results for moduli and strength as a function of

temperature, it is shown that the difference between saturated and dry samples is more pronounced at -2°C than at 20°C . At -2°C there is a difference of 11.8 GPa in stiffness between the dry and saturated laminates, and a reduction of 70 MPa in strength. Correspondingly, the reduction in stiffness and strength at 20°C is 6.9 GPa and 28 MPa, respectively. The difference in magnitude changes as a function of both temperature and saturation state indicates a coupling effect as the entrained water expands with decreasing temperature. Conversely, while the E-Glass laminates exhibit a reduction in ~ 1 GPa from the dry to saturated laminates, the overall reduction is consistent across the temperature range as evidenced by the parallel lines in the E-Glass Young's modulus plot. The E-Glass strength also exhibits consistent reductions in strength across the temperature range of consideration. However, the overall reduction in tensile strength for the E-Glass going from a dry to saturated state is significant, nearly 100 MPa in strength corresponding to 30-35% reductions.

Figure 9 - Laminate Tensile Properties

Table 1 - Tensile Property Summary

Tensile Summary						
	20°C		5°C		-2°C	
	Dry	Wet	Dry	Wet	Dry	Wet
Carbon						
Modulus (GPa)	54.8 +/- 3.3	47.9 +/- 1.9	58.7 +/- 3.6	50.2 +/- 2.1	61.2 +/- 1.8	49.4 +/- 2.2
Strength (MPa)	595 +/- 16.0	567 +/- 24.3	607 +/- 35.1	563 +/- 17.4	637 +/- 39.3	567 +/- 37.2
E-Glass						
Modulus (GPa)	11.3 +/- 0.4	10.8 +/- 0.5	11.3 +/- 0.26	10.3 +/- 0.4	11.5 +/- 0.2	11.4 +/- 0.6
Strength (MPa)	288 +/- 16.1	185 +/- 8.4	288 +/- 13.3	191 +/- 8.9	293 +/- 10.8	209 +/- 6.4

The specimens after tensile testing are shown in Figure 10 for the dry and saturated samples of the E-glass/Epoxy and Carbon/Epoxy composite laminates. For the E-Glass laminates it is seen that the failure mode in the dry samples was dominated by fiber kinking as evidenced by the slip in the samples after failure, and the large damage area. Very little fiber breakage was observed in this case. Conversely, fiber breakage was the dominant failure mode in the saturated samples with failure mostly happening perpendicularly to the loading direction, leading to very small damage zones. Negligible fiber kinking was observed in the saturated samples. Finally, the effects of temperature could not be seen in the images and the failure modes for all three temperatures were very similar. For the Carbon/Epoxy composites, the postmortem images of the tensile samples can be seen in the lower images of Figure 10. Unlike in the case of the E-glass composites, there was no visible fiber kinking in both the dry and saturated samples. Nevertheless, the saturated samples showed damage on the other end of the sample where the breakage did not occur. This is an indication that there is material degradation caused by water saturation. Lastly, there was no visible variation between samples tested at different temperatures for both the dry and saturated samples.

Figure 10 - Tensile Failure Modes and Damage Characteristics

The compressive behavior of the Carbon and E-Glass laminates in terms of stiffness and strength as a function of both water saturation and temperature are presented graphically in Figure 11 and summarized in Table 2. Consistent with the tensile tests, all compression experiments were performed at 20°C, 5°C, and -2°C. The effects of both water saturation and temperature on the compressive performance of the materials are clearly seen in each of the respective plots in Figure 11. In terms of stiffness in compression, Carbon exhibits a stiffness of nearly double that of the E-Glass and strengths of 3-4 times larger. This is consistent for both the dry and saturated laminate.

The effects of water saturation on the compressive behavior of the respective materials are highly evident in comparing the trend lines in Figure 11 for both Young's modulus and strength. In all cases, at any given temperature there is an overall reduction in properties when the laminates become saturated with water. Observing the magnitudes of the baseline Young's modulus of the respective materials, the compressive Young's modulus exhibits a 40-50% reduction at 20°C due

solely to the presence of the entrained water. At -2°C the overall percentage in magnitude reduction is smaller but still evident, with the reduction in magnitude percentage being attributable to the Young's moduli reduction in the dry laminates. This highlights the observation that the Young's modulus of the saturated panels is constant across all temperatures tested, 3 GPa and 1.5 GPa for the Carbon and E-Glass, respectively. The constant compressive Young's moduli for the saturated panels indicate that the reduction in compressive Young's modulus occurs in the room temperature condition and is significant enough that no further reductions occurs with decreasing temperatures. The compressive strength of the Carbon laminate displays a 30% reduction at room temperature and a corresponding 10% reduction at -2°C . The difference in percent reduction of the strength is the result of differing trends in the dry and saturated laminates as a function of temperature. The dry carbon laminate displays a reduction in strength with decreasing temperature whereas the saturated laminate exhibits an increase in compressive strength with decreasing temperature. The E-Glass compressive strength is reduced 30-40% overall across the temperature range, with the reduction being slightly higher at the -2°C temperature. This is attributable to the dry laminates showing an increase in strength with decreasing temperatures, whereas the saturated laminates compressive strength is constant across the temperature range of consideration.

Figure 11 - Laminate Compressive Properties

Table 2 - Compressive Property Summary

Compression Summary						
	20°C		5°C		-2°C	
	Dry	Wet	Dry	Wet	Dry	Wet
Carbon						
Modulus (GPa)	5.6 +/- 0.7	2.94 +/- 0.5	4.3 +/- 0.4	2.8 +/- 0.6	3.8 +/- 0.3	3.0 +/- 0.4
Strength (MPa)	448 +/- 29.0	306 +/- 29.7	401 +/- 28.0	340 +/- 28.1	420 +/- 23.9	378 +/- 12.6
E-Glass						
Modulus (GPa)	2.4 +/- 0.1	1.45 +/- 0.3	1.8 +/- 0.2	1.5 +/- 0.2	1.8 +/- 0.06	1.4 +/- 0.3
Strength (MPa)	154 +/- 9.0	107 +/- 4.7	160 +/- 7.7	103 +/- 2.2	165 +/- 9.3	107 +/- 4.7

5.2.Short Beam Shear Strength Effects

The characterization of the short beam shear strength trends for the dry and saturated Carbon and E-Glass laminates are graphically highlighted in Figure 12 and summarized in Table 3. Observation of these results show two unique trends in the data. The first is that when the panels are in a fully saturated state there is a net reduction in short beam shear strength across all temperatures, and it is a near uniform reduction of approximately 14 and 6 MPa for the Carbon and E-Glass laminates, respectively. This is apparent by the parallel trend lines in each plot differentiating the saturated and dry laminate short beam shear strengths. The second trend is that for both the dry and saturated laminates, the short beam shear strength increases with decreasing temperature. This increase in strength from 20°C to -2°C is on the order of 20% for each material.

Figure 12 - Short Beam Shear Mechanical Characterization

Table 3 – Short Beam Shear Strength Summary

Short Beam Shear Strength						
	20°C		5°C		-2°C	
	Dry	Wet	Dry	Wet	Dry	Wet
Carbon (MPa)	52.8 +/- 0.6	39.9 +/- 0.7	62.6 +/- 0.9	47.5 +/- 0.9	66.1 +/- 0.6	51.5 +/- 0.8
E-Glass (MPa)	27.7 +/- 1.3	20.2 +/- 0.3	32.6 +/- 0.8	22.8 +/- 0.2	33.7 +/- 0.7	25.0 +/- 0.5

5.4 Static Mode-I Fracture

The effects of water saturation and temperature on the Static Mode-I Strain Energy Release Rate (G_{IC}) of the Carbon and E-Glass laminates are highlighted in Figure 13. From the respective laminate plots, it is observed there is a quantifiable influence of both water absorption and temperature on the dependence of G_{IC} , although quite different in nature between the materials. Comparison of the relative dry and saturated G_{IC} trends for the Carbon laminate shows a clear overall reduction in G_{IC} when the laminates become saturated. The reduction is quantified to be on the order of 30-40% for the respective temperatures. Considering both the saturated and dry conditions for the Carbon laminates, there is less dependence on temperature than the E-Glass laminates where there is a very small decrease in G_{IC} from 20°C to 5°C, and a statistically

constant value from 5°C to -2°C as indicated by the overlapping groupings of the individual samples at these temperatures. The G_{IC} results for the E-Glass laminates exhibit quite the opposite trends as observed in the Carbon laminates in two unique aspects. The first quantifiable trend is observed in the comparison of the saturated and dry laminates in that the G_{IC} increases with the presence of entrapped water. At a temperature of -2°C the increase going from a dry condition to a saturated one is on the order of 100%. Further observation of the E-Glass trends shows that in the dry condition there is a very clear reduction in G_{IC} with decreasing temperature. However, in examining the saturated results, this trend is reversed in that the G_{IC} values exhibit an equally clear increasing G_{IC} with decreasing temperature. The 20°C values for both the saturated, and the dry E-Glass laminates are nearly the same and as the temperature is decreased, the G_{IC} values diverge for the saturated, and dry laminates. It is noted that when making these observations, the failure of the carbon laminates is much more interfacial between the plies and dominated by the resin, whereas the E-Glass laminates exhibit much more contribution from the fibers themselves in the form of fiber bridging across the crack front. This indicates that the presence of water saturation influences G_{IC} differently based upon the specific fracture surfaces and mechanics, namely resin vs fiber dominated.

Figure 13 – Static Strain Energy Release Rate G_{IC} vs. Crack Length

Table 4 – Static Mode-I Strain Energy Release Rate Summary

Static Mode-I Strain Energy Release Rate (J/m^2)						
	20°C		5°C		-2°C	
	Dry	Wet	Dry	Wet	Dry	Wet
Carbon (J/m^2)	1492 +/- 106	894 +/- 140	1182 +/- 54	619 +/- 50	1239 +/- 67	808 +/- 311
E-Glass (J/m^2)	2175 +/- 418	1777 +/- 101	1666 +/- 104	2274 +/- 310	1366 +/- 113	2724 +/- 451

5.5 Dynamic Mode-I Fracture

The dynamic Mode-I critical strain energy release rate, G_{ICd} for the Carbon and E-Glass laminates at each of the temperatures considered and including the effects of water saturation are shown in Figure 14. The loading rate for the E-Glass laminates, taken to be the rate of change of the strain energy release rate with time, was calculated for the dry and saturated E-Glass to be 174.8 kJ/m^2 -s and 148.6 kJ/m^2 -s (1000 in-lb/in²-s and 850 in-lb/in²-s) respectively. The Carbon laminate loading rates were 113.6 kJ/m^2 -s and 78.6 kJ/m^2 -s (650 in-lb/in²-s and 450 in-lb/in²-s) respectively for the dry and saturated states.

The trends in the dynamic fracture results are consistent with the trends observed in the corresponding static experiments, with the primary difference being the magnitudes of the G_{IC} values. As seen from the Carbon G_{ICd} results, the influence of entrapped water is negligible across the temperature range considered. The G_{ICd} value at the respective temperatures are consistent within the variability of the data. Both the dry and saturated laminates also exhibit near constant values at 20°C and 5°C with a drop in G_{ICd} going from 5°C to -2°C. Conversely, the E-Glass laminates show differences between the dry and saturated states. There is a quantitative increase in G_{ICd} value when the E-Glass laminate becomes saturated with water. The net increase in G_{ICd} differs as a function of temperature, from 19% at 20°C up to 52% at -2°C. This change in percent increase is similar to the trend seen in the static values where the dry laminates have a decreasing G_{IC} with decreasing temperature and the saturated laminates show an increase in G_{IC} with decreasing temperature. Of significance is the observation that in comparing the dynamic to static values, the G_{ICd} values are markedly lower than the corresponding G_{IC} values. This can be better understood by looking at the interaction of the plastic zone and the carbon or glass fibers at low and high loading rate. Generally, the plastic zone size is directly proportional to the fracture toughness and inversely proportional to the square of the yield stress of the material [19]. Epoxy being a strain rate dependent material has a higher yield stress at high strain rates, which leads to a drastic drop in the plastic zone size and consequently a decrease in the G_{IC} value. In addition, the fracture surfaces shown in Figure 15 show that clean interfacial fracture between the fiber surface and the matrix occurred during dynamic experiments in carbon fiber composites. The glass fiber composites showed a mixed fracture surface with still predominant interfacial separation. This interfacial fracture toughness is much lower than the intrinsic fracture toughness of the matrix material.

Figure 14 –Dynamic Mode-I Critical Strain Energy Release Rate, G_{ICd}

Table 5 – Dynamic Mode-I Strain Energy Release Rate Summary

Dynamic Mode-I Strain Energy Release Rate (J/m^2)						
	20 $^{\circ}C$		5 $^{\circ}C$		-2 $^{\circ}C$	
	Dry	Wet	Dry	Wet	Dry	Wet
Carbon (J/m^2)	87 +/- 16	99 +/- 31	92 +/- 19	83 +/- 19	62 +/- 19	66 +/- 11
E-Glass (J/m^2)	317 +/- 132	393 +/- 110	281 +/- 80	403 +/- 79	214 +/- 65	446 +/- 27

Figure 15 - Dynamic Fracture surfaces for (a) E-Glass/Epoxy and (b) Carbon/Epoxy Composites

5.6 Impact and Flexure after Impact

The impact force time histories for the Carbon and E-Glass laminates in the dry and saturated conditions as a function of temperature are provided in the upper plots of Figure 16 as measured by the cross head of the drop tower. While the impact tests were shown to be repeatable from specimen to specimen, only a representative specimen for each temperature is shown for clarity of the time history plot. A summary of the peak impact force is provided in Table 6. In considering these time histories and magnitude comparisons, several key observations can be drawn. The first is that for both the Carbon and E-Glass laminates, the presence of water saturation in the laminates reduces the peak force recorded during the impact event, indicating less stiff laminates under impact loading conditions. The reduction in peak force for the Carbon laminates is 12% at 20°C and 8% for both 5°C and -2°C. The E-Glass laminates similarly show a reduction in peak force during impact, with the magnitude reduction being more significant, 28% at 20°C and 5°C and 21% -2°C. For all the laminates peak force increases with decreasing temperature, although the increase is small enough that there is overlap in the data when accounting for the respective data set standard deviations. Furthermore, while the presence of the entrapped water does reduce the overall peak force experienced by the specimens, it does not appear to alter the overall time duration or phasing of the impact event. The peak forces for a given material occur at nearly the same point in time, there is just a reduction in that respective peak force magnitude. Additionally, the damage mechanisms largely remain constant between the dry and saturated laminates for a given material. Primarily, there is brittle type damage for the Carbon laminates after peak impact while there is a controlled ductile like mechanism exhibited by the E-Glass laminates. In the E-Glass materials this is observed as the smoother force-time histories and longer duration of the impactor energy impact event.

The post-impact four-point flexural behavior of the respective laminates is shown in the lower images of Figure 16 (Note: Due to the large differences in flexural strength the axis limits have not been adjusted for each material). The inherent brittleness of the carbon laminates and their relative thin margin between retaining structural integrity and full failure is highlighted by the amount of scatter in the flexure data for these laminates. Interestingly, the Carbon laminates which have been saturated with water show less variability in the flexure results than the corresponding dry laminates, particularly at the 5°C and -2°C temperatures. Furthermore, the saturated Carbon laminates appear to exhibit larger flexure after impact strength than was observed for the dry laminates, between 18% and 31% depending on temperature of the testing. This wide range of strength values, even after highly controlled impact experiments, highlights the inherent unknowns and risks when utilizing the Carbon laminates for dynamic / impact loading events. Conversely, the E-Glass exhibits significant reductions in flexure after impact strength when accounting for the presence of water saturation. For the E-Glass laminates tested in flexure after impact at 20°C and 5°C there is a 44% decrease in residual flexural strength. Furthermore, the specimens impacted at -2°C sustained complete failure during the impact event (i.e.; zero residual strength). The specimens did remain intact after the impact, but the flexural testing indicated only a minimal resistance to loading and for this reason the -2°C flexural results have not been included in the relevant plot. Overall, for the 20°C and 5°C results there is a readily observable increase in residual flexural strength with decreasing temperature for the E-Glass laminates. Furthermore, The E-Glass laminates exhibit flexural strain values between 1.25% and 1.75% whereas the Carbon laminates have flexural strain values between 0.75% and 1.1%.

Figure 16 - Impact and Flexure After Impact Performance

Table 6 – Peak Impact Force Summary

	Peak Impact Force (N)					
	20°C		5°C		-2°C	
	Dry	Wet	Dry	Wet	Dry	Wet
Carbon (J/m ²)	6269 +/- 172	5457 +/- 281	6358 +/- 153	5792 +/- 121	6490 +/- 160	5980 +/- 176
E-Glass (J/m ²)	3401 +/- 94	2442 +/- 96	3608 +/- 122	2597 +/- 121	3548 +/- 102	2768 +/- 81

Table 7 – Flexural Strength after Impact Summary

Flexural Strength (MPa)						
	20°C		5°C		-2°C	
	Dry	Wet	Dry	Wet	Dry	Wet
Carbon (J/m ²)	277 +/- 77	339 +/- 85	298 +/- 114	433 +/- 34	345 +/- 132	446 +/- 13
E-Glass (J/m ²)	116 +/- 27	64 +/- 6	157 +/- 8	87 +/- 9	173 +/- 6	Failure Under Impact

6. CONCLUSIONS

A comprehensive experimental investigation of the effects of water saturation coupled with low temperatures on the mechanical, fracture, and impact dependent material properties of E-Glass/Epoxy and Carbon/Epoxy laminates has been conducted. The primary objective of the study was to quantify the mechanical and damage behavior of these advanced material systems when water saturation is coupled with decreasing temperatures down to the lowest expected water temperatures of the arctic / deep ocean environments. The significant findings of the study highlight that the considerations of the coupled effects of low temperatures and water saturation on composite laminates are critical when designing with these advanced materials. Designing a structure for use in arctic and deep water applications in the absence of detailed material characterization data performed at the expected operating conditions can lead to unexpected and premature failures. It is evident from the investigation that the use of baseline material data taken in controlled laboratory conditions in a dry and room temperature environment are largely insufficient for the design of critical structures to be deployed in low temperature, wetted applications. While not explicitly verified in the current study, it is hypothesized that the presence of full water saturation into the respective laminates results in the presence of

hygrothermal residual stress states which subsequently superimpose on and couple with the mechanical loading during material testing. Furthermore, it is noted that while the current study utilized distilled freshwater in the water ingress aspects of the study, additional work is needed to quantify the effects of salt and/or seawater on the material properties.

The key findings of the study were as follows:

- **Mechanical Performance**

- Tension: For all temperatures evaluated in the study there are measurable drops in both Young's moduli and tensile strengths with water saturation, although the magnitudes of the drops is dependent upon material property and temperature. For the Carbon laminates, the decrease in Young's modulus and tensile strength is larger at -2°C than at 20°C. Conversely, the E-Glass material exhibits a near-uniform drop in both moduli and strength in the presence of entrained water over the temperature range. For both the dry and saturated laminates there is an inverse relationship between tensile performance and temperature.
- Compression: The E-Glass and Carbon/Epoxy materials utilized in this study both exhibit reductions in both compressive Young's modulus and strength when saturated with water as compared to the dry condition. For both laminates evaluated in the study, the water-saturated specimens exhibit a constant Young's modulus with decreasing temperature while the dry laminates exhibit decreasing stiffness with decreasing temperature. The compressive strength of the E-Glass laminate is reduced by 30-40% across the temperature range.

- Short Beam Shear: The short beam shear strength characteristics of the E-Glass and Carbon/Epoxy laminates display common trends with water saturation and decreasing temperature. In a saturated state, there is a near-uniform decrease in short beam shear strength across the 20°C to -2°C temperature range. The experimental results also are characterized by a strength increase with decreasing temperature for both dry and saturated laminates.

- **Fracture Performance**

- Static Mode-I: Both of the E-Glass and Carbon laminates considered exhibit a direct dependence of G_{IC} on water saturation and temperature, although opposite effects in nature. The Carbon laminate shows a clear overall reduction in G_{IC} when the laminates become saturated, on the order of 30-40% depending on specific temperature. Conversely, for the E-Glass laminate comparison of the saturated and dry condition shows that the G_{IC} increases with the presence of entrapped water. These differences in behavior with saturation appear to be due to the differences in observed failure mode with the Carbon being interfacial ply failure and the E-Glass displaying significant fiber damage.

- Dynamic Mode-I: The Carbon/Epoxy laminate exhibits no quantifiable dependence of G_{ICd} on water saturation condition at a given temperature. The dry and saturated Carbon laminates each experience drops in G_{ICd} going from 5°C to -2°C. Similar to the static Mode-I fracture results, the E-Glass laminates exhibited higher G_{ICd} values in the saturated state than the dry state, from 19% at 20°C up to 52% at -2°C. For both the Carbon and E-Glass laminates the dynamic Mode-I critical strain energy

release rates are significantly lower than the static Mode-I critical strain energy release rates.

- **Impact and Flexure after Impact Strength Performance**

- Impact: The E-Glass and Carbon laminates show lower peak impact forces in a saturated state than the corresponding dry state, an indicator of reduced dynamic flexural stiffness. However, the overall timing, duration, and damage characteristic (brittle versus ductile) of the impact event appears unaffected by the presence of the water in the material. Each laminate (saturated and dry) displays negligible dependence on impact behavior as a function of temperature as the peak impact forces are consistent across the temperature range considered in the study.

- Flexure After Impact: The four-point flexure after impact behavior of the Carbon laminates result in a trend by which the saturated material has higher post-impact flexural strength than the dry Carbon laminates, between 18% and 31%. However, the E-Glass laminate in a saturated state exhibits a significant reduction in flexure after impact strength as compared to the dry laminate, ~44% at the two higher temperatures of the study. The saturated E-Glass at -2°C had zero residual strength after impact owing to the complete failure during the impact experiments.

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Table 1 - Tensile Property Summary

Tensile Summary						
	20°C		5°C		-2°C	
	Dry	Wet	Dry	Wet	Dry	Wet
Carbon						
Modulus (GPa)	54.8 +/- 3.3	47.9 +/- 1.9	58.7 +/- 3.6	50.2 +/- 2.1	61.2 +/- 1.8	49.4 +/- 2.2
Strength (MPa)	595 +/- 16.0	567 +/- 24.3	607 +/- 35.1	563 +/- 17.4	637 +/- 39.3	567 +/- 37.2
E-Glass						
Modulus (GPa)	11.3 +/- 0.4	10.8 +/- 0.5	11.3 +/- 0.26	10.3 +/- 0.4	11.5 +/- 0.2	11.4 +/- 0.6
Strength (MPa)	288 +/- 16.1	185 +/- 8.4	288 +/- 13.3	191 +/- 8.9	293 +/- 10.8	209 +/- 6.4

Table 2 - Compressive Property Summary

Compression Summary						
	20°C		5°C		-2°C	
	Dry	Wet	Dry	Wet	Dry	Wet
Carbon						
Modulus (GPa)	5.6 +/- 0.7	2.94 +/- 0.5	4.3 +/- 0.4	2.8 +/- 0.6	3.8 +/- 0.3	3.0 +/- 0.4
Strength (MPa)	448 +/- 29.0	306 +/- 29.7	401 +/- 28.0	340 +/- 28.1	420 +/- 23.9	378 +/- 12.6
E-Glass						
Modulus (GPa)	2.4 +/- 0.1	1.45 +/- 0.3	1.8 +/- 0.2	1.5 +/- 0.2	1.8 +/- 0.06	1.4 +/- 0.3
Strength (MPa)	154 +/- 9.0	107 +/- 4.7	160 +/- 7.7	103 +/- 2.2	165 +/- 9.3	107 +/- 4.7

Table 3 – Short Beam Shear Strength Summary

Short Beam Shear Strength						
	20°C		5°C		-2°C	
	Dry	Wet	Dry	Wet	Dry	Wet
Carbon (MPa)	52.8 +/- 0.6	39.9 +/- 0.7	62.6 +/- 0.9	47.5 +/- 0.9	66.1 +/- 0.6	51.5 +/- 0.8
E-Glass (MPa)	27.7 +/- 1.3	20.2 +/- 0.3	32.6 +/- 0.8	22.8 +/- 0.2	33.7 +/- 0.7	25.0 +/- 0.5

Table 4 – Static Mode-I Strain Energy Release Rate Summary

Static Mode-I Strain Energy Release Rate (J/m^2)						
	20°C		5°C		-2°C	
	Dry	Wet	Dry	Wet	Dry	Wet
Carbon (J/m^2)	1492 +/- 106	894 +/- 140	1182 +/- 54	619 +/- 50	1239 +/- 67	808 +/- 311
E-Glass (J/m^2)	2175 +/- 418	1777 +/- 101	1666 +/- 104	2274 +/- 310	1366 +/- 113	2724 +/- 451

Table 5 – Dynamic Mode-I Strain Energy Release Rate Summary

Dynamic Mode-I Strain Energy Release Rate (J/m^2)						
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E-Glass (J/m^2)	317 +/- 132	393 +/- 110	281 +/- 80	403 +/- 79	214 +/- 65	446 +/- 27

Table 6 – Peak Impact Force Summary

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Dr. James LeBlanc

26 April 2021

Dear Editor,

The authors have no financial or other conflicts of interest associated with the manuscript for submission.

Thank You,
James LeBlanc