MECHANICAL PROPERTIES OF CHEMICALLY DEGRADED AEROSPACE FIBRE-REINFORCED POLYMER MATRIX COMPOSITES

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ABSTRACT

Fibre-reinforced polymer matrix composites (FRPMCs) are widely used for structural applications in aerospace due to their high specific mechanical properties. However, because of the susceptibility of polymer matrix materials to chemical attack, FRPMCs have a potential vulnerability to chemically assisted degradation resulting from exposure to contaminating fluids. Furthermore, microstructural defects such as voids, cracks, channels and delaminations formed during fabrication promote accelerated degradation though capillary-action-aided diffusion. As a result, chemical degradation can affect the structural integrity of FRPMCs in critical aerospace applications.

This research investigates the effects of exposing an aerospace-grade, carbon-fibre reinforced epoxy pre-impregnated laminate to a selection of fluids prevalent in aerospace applications. Fluids under investigation include deionized water, artificial ocean water, hydraulic fluid, turbine oil, ethylene glycol and air (control). Degradation is quantified by analyzing matrix-dominated mechanical properties following fluid exposure, and an association between exposure duration, fluid ingress and mechanical degradation is developed. Composite samples are subjected to single-surface fluid exposure, to elicit a realistic in-service scenario. To accelerate ageing, and to imitate the upper range of in-service operating temperatures, fluid exposure takes place at elevated temperatures (85°C). Over a three-month duration, samples are periodically removed from conditioning for gravimetric analysis to evaluate relative rates of fluid ingress. Sequentially, samples are subjected to flexural testing and optical microscopy. Through statistical correlation of each parameter in association with the respective sample exposure durations, this work contributes to the generation of an experimentally validated approach to design allowable generation, in-service exposure limits and inspection frequency for aerospace FRPMCs.

1 MOTIVATION

Continued intransient performance of structural aircraft components is paramount; Collapse of critical, load-bearing structures in flight or during landing can lead to catastrophic failure and loss of life. Consequently, regulatory bodies, such as the United States Department of Transportation Federal Aviation Administration (FAA), have established stringent regulation which aid in ensuring continued airworthiness of aircraft components. To certify continued airworthiness, original equipment manufacturers (OEMs) must provide experimental evidence which demonstrates “material design values or allowables are attained with a high degree of confidence in the appropriate critical environmental exposures to be expected in service” [1]. Hence, OEMs must devise and perform experiments which emulate realistic, in-service conditions to predict component service-life, define design allowables, and to recommend inspection frequencies.

Fibre-reinforced polymer matrix composites (FRPMCs) are widely used for structural aerospace applications due to their high specific mechanical properties. However, FRPMCs demonstrate a high degree of variability in mechanical properties, when compared to conventional engineering materials, due to diversity and complexity of layup processes; anisotropicity; heterogeneity; and matrix sensitivity to environmental exposure [1]. One such factor which, empirically, has demonstrated to be detrimental is exposure to water. A large body of work has investigated the effects of moisture ingress on the static and dynamic properties of FRPMCs. Studies of this nature have investigated the effects of humidity and complete immersion in sub-ambient, ambient and elevated temperatures for various exposure durations, and have looked at a range of mechanical properties [2]–[5]. From this, two research gaps have been identified; the relative contribution of environment-specific fluids, apart from water, on mechanical properties and the effects of a realistic, single-sided fluid exposure on the rates of moisture ingress. While previous work has looked at the degradation of FRPMCs resulting from aerospace chemicals [6], [7], focus has not been given to static mechanical testing of conditioned monolithic laminates.

This work is a component of collaborative research between Carleton University and General Dynamics Missions Systems – Canada to evaluate the degradation of mechanical properties in FRPMCs due to environmental exposures common to maritime aircraft interiors. This study aims to evaluate the degradation of FRPMCs resulting from exposure to chemicals common to aerospace given a pragmatic exposure scenario. This report details coupon manufacturing, conditioning and testing methodologies followed; an evaluation of the conditioning procedure; test results and associated statistical analysis; conclusions drawn; and logical avenues for future work.

2 MANUFACTURING, CONDITIONING AND TESTING

Test specimens were manufactured from an eight-harness satin weave, AX-5180 curing epoxy-carbon prepreg from Axiom Materials, Inc. AX-5180 was selected for its recognized industrial adoption as a structural laminate for aircraft interior applications. Prepreg consisted of 54% fibre content by volume (60% by weight). Monolithic laminate was manufactured with a $[0_{8F}]$ stacking sequence and was cured using a two-temperature stage, autoclave process. Flexural coupons, which were derived from an early design stage cure cycle, contained 2.9% voids, as determined through microtomographic analysis. Test specimens were cut using a water-cooled diamond blade on a UKAM Smart Cut 6001 GP Sectioning Saw to a 32:1, span-to-thickness ratio, in accordance with ASTM D7264 standard [8].

Primary testing took a novel approach at sample conditioning, which emulated a more realistic, in-service exposure condition than that of traditional immersion tests. Flexural samples were mounted in one of six Polytetrafluoroethylene (PTFE) arrays each possessing nine sample 'wells'. The PTFE arrays consisted of two components; a bottom plate and a top plate. The bottom plate had nine, 1/16-inch-deep recesses to house the flexural samples relative to the top plate. The top plate contained nine through-hole sample wells, machined to contain liquid on one surface of the flexural samples. To produce a sufficient seal, a 1/8-inch-thick silicone gasket was positioned between the top plate and the flexural samples. To maintain the seal between the top plate and samples, 30, one-quarter inch through holes were machined in a complementary orientation in the top plate, gasket and bottom plate allowing for the clearance of socket-head cap screws. Once the flexural samples had been seated and the array assembled, the socket-head cap screws were torqued to 10 in·lb through three repetitions of a raster torque pattern, establishing an even pressure distribution. Figure 1 (a) displays a schematic of the top plate of an individual array. Prior to conditioning, sample masses were measured, as per ASTM D5229 [9], using a Sartorius AG, ENTRIS64-1S (± 0.0001g) analytical balance; length, width and thickness dimensions were evaluated using digital calipers (± 0.001in).

Conditioning was performed at 85°C in a Quincy Lab Inc., 30AFE-LT, low temperature, forced air laboratory oven. Four sample arrays were centered on separate shelves in the oven. Two thermocouples were adhered to the top surface of the top plate of each PTFE array using a polyimide tape; this was done in same location on each array to monitor temperature variation between arrays. Prior to liquid addition, assembled arrays were placed in the oven, at temperature, for 24 hours to reach thermal equilibrium. Artificial ocean water (hereafter referred to as ocean water) was synthesized using an archetypal ratio of constituent salts dissolved in deionized water [10]. Following this, liquid was added to each sample well, polyimide tape was applied to limit evaporation and the arrays were placed in the oven. The five contaminating liquids used; deionized water, ocean water, synthetic hydraulic fluid, turbine oil and ethylene glycol (a primary component in many deicing fluids) were selected for their prevalence in aerospace. Oven temperatures were continuously monitored and liquid levels were periodically inspected and topped-up as required. Figure 1 (b) illustrates a schematic of the 1.83 cubic feet conditioning oven with four sample arrays, separated by approximately one inch headspace.

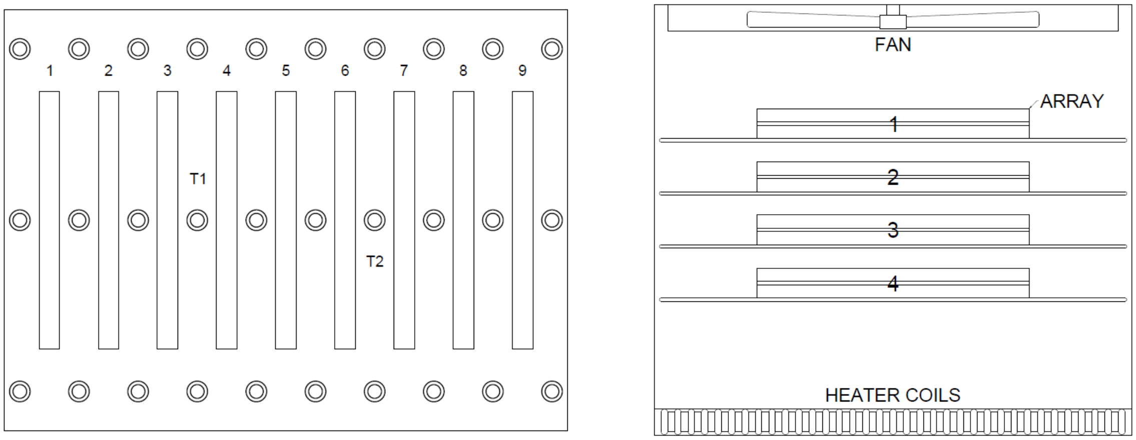

Figure 1. Schematics of a) array top plate with wells and thermocouples labelled and b) array positioning in oven, respectively

Following conditioning, samples were removed from the arrays, sealed in individual plastic bags and allowed to cool to room temperature (~ 23°C). Once cooled, surface moisture was removed using a standardized wiping procedure and sample masses were remeasured. Subsequently, sample dimensions were remeasured and flexural properties were determined in accordance with ASTM D7264 [8]. Samples were subjected to three-point bend testing using a custom designed fixture in a MTS 810 load frame. Finally, post-flexural, optical microscopy was performed to characterize visual degradation on liquid exposed surfaces.

3 CONDITIONING EVALUATION

As a novel conditioning technique, there were a number of nuances and obstacles which, inherent to the method or encountered through testing, required deviation from ASTM D5229 standard [9]. The following sections detail these complications, their effect on testing and how they were addressed.

3.1 Temperature Distribution

ASTM D5229 standard specifies conditioning temperature shall be maintained at the temperature of interest, ± 3°C [9]. Preliminary temperature measurements revealed that the conditioning oven was unable to achieve this distribution at 85°C for all sample when four arrays were positioned as shown in Figure 1, b). While temperatures were stable in all positions, the large surface area of the arrays hindered effective heat transfer through convection currents. To mitigate temperature variance, sample arrays were repositioned weekly to allow an equal exposure duration at each array position. Table 1 presents the average and standard deviation of temperatures for each array over their conditioning duration. Each duration is represented by two sets of temperatures as two arrays were required to accommodate the 18 samples conditioned at each duration.

Target Duration	Temperature (°C)
1 Day (24 hours)	86.85 ± 0.97
	80.86 ± 0.71
1 Week (168 hours)	84.30 ± 3.18
	82.17 ± 3.62
1 Month (720 hours)	78.29 ± 9.77
	77.69 ± 10.10
2 Month (1440 hours)	78.56 ± 7.45
	78.73 ± 6.93
3 Month (2160 hours)	77.78 ± 9.53
	78.20 ±9.44

Table 1. Array temperature distribution over conditioning durations

Shorter duration sample arrays exhibited higher average temperatures with lower deviation due to their position in the oven and the limited period for which the oven door was open during conditioning, but demonstrated a greater difference in average temperature between arrays. Conversely, longer condition durations yielded smaller temperature distributions between arrays but had lower average temperatures and higher deviation due to heat released during the opening of the oven door. Through linear interpolation of the obtained temperature data, the average temperature for each sample over its exposure duration was estimated. Plotting the resulting flexural properties and mass uptake values as a function of the interpolated temperature data did not reveal any strong correlations. Hence, it has been concluded that any variance due to larger than specified temperature distribution is negligible.

3.2 Removal from Conditioning

Periodically throughout conditioning, samples exposed to the higher volatility liquids would have their sample wells ‘run dry’, due to an imperfect seal, and would no longer be subjected to liquid exposure. To mitigate moisture losses, samples were conditioned for additional days, equivalent to the number days for which they were without liquid. Such procedural alterations were required for four samples from each of the deionized water and ocean water, one-month and three-month conditioning durations. While not verified, it is expected that moisture absorption and desorption rates are not equivalent. Despite this, samples which ran dry and were subsequently re-exposed did not show a significant variation in mass change or flexural properties from their consistently exposed counterparts and thus, were included in results.

3.3 Halting Short Duration Fluid Uptake Measurements

Over the first three weeks of conditioning, three-month conditioning duration samples had fluid uptake measurements performed every three days to plot moisture uptake with greater resolution through a period of predictably rapid moisture uptake and to allow for determination of saturation. With each measurement, the sample arrays became more difficult to reassemble due to deformation of the silicone gasket material brought about by hydraulic fluid and turbine oil uptake. Thus, periodic mass uptake measurements for the three-month samples were halted to preserve the integrity of the full duration uptake and flexural results.

4 RESULTS AND ANALYSIS

4.1 Moisture absorption

Moisture absorption curves in Figure 2 summarize change in sample mass as a function of conditioning duration, expressed in square root hours, for the five chemicals investigated. A control group was subjected to comparable temperatures and conditioning environment without chemical exposure. Each data point represents the average percent change in mass of three samples for a given duration, apart from one-month control which is based on the average of only two samples due to sample well cross-contamination. Samples under investigation exhibited a net positive increase in moisture uptake as a function of exposure duration while the control samples showed negligible mass variation. Deviation between samples within a single sample group (a single liquid over a single duration) may be attributed to variations in local temperature, surface moisture removal method, void distribution and void concentration. It is hypothesized that the greater absorption rate of ethylene glycol, deionized water and ocean water versus hydraulic fluid and turbine oil is attributed to a combination of the relatively small molecular size and polarity of the former. These attributes promote ingress through smaller, nanoscopic apertures through favourable, hydrophilic interactions between the absorbed solvent and epoxy hydroxyl groups. The decrease in mass of the deionized water samples after a single day of conditioning may be attributed to the elution of matrix material from the sample's surface which was then removed through the wiping procedure, applied to remove surface moisture. A similar decrease in mass was not observed for the ocean water samples of the same conditioning duration likely due to mass gained from sub-surface salt precipitation.

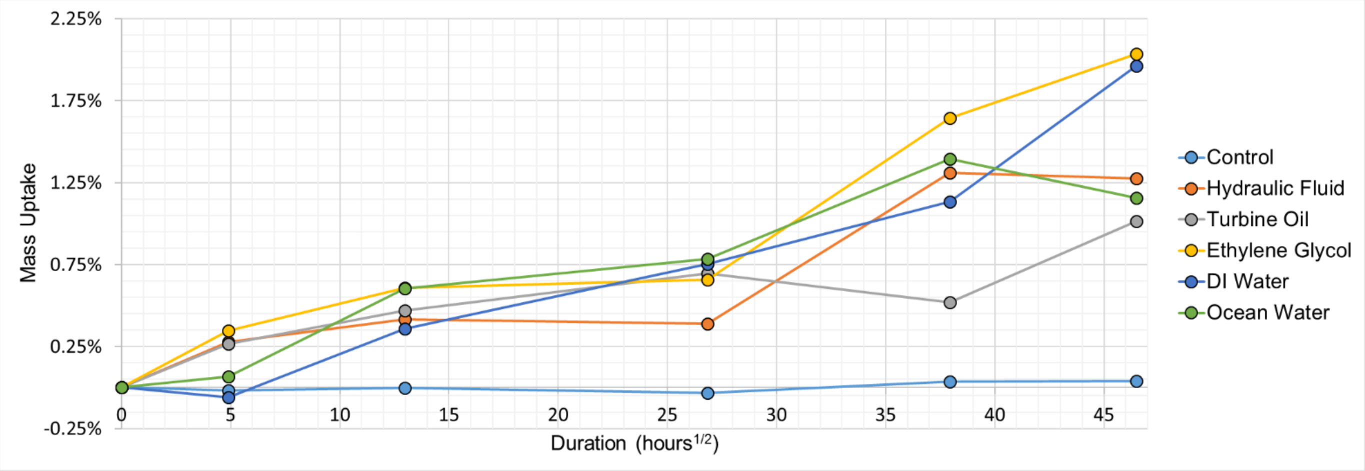

Figure 2. Mass uptake of flexural samples conditioned though elevated temperature, single-sided liquid exposure

Composite diffusion models, Fickian and other, predict a linear region of mass increase followed by a plateau of moisture absorption associated with laminate saturation [11]–[13] which has been demonstrated empirically for FRPMC exposed to both humid environment and full immersion. Due to a limited number of data points and lack of an appropriate measurement interval, it is difficult to confirm linearity. As no plateau was observed, it may be deduced that the samples did not reach moisture saturation over three months. This duration was selected as literature indicated that FRPMCs, of comparable stacking sequence and make up to that of the material under investigation, could achieved moisture saturation in less than three months at equivalent or lower conditioning temperatures [6], [7], [14], [15]. This result brought to attention the differences in moisture absorptions rates of samples conditioned through traditional immersion compared to that of single-sided exposure. Through a combination of an increased surface area to volume ratio and edge effects brought about by exposed laminate edges [16], complete sample immersion allows for an accelerated rate of absorption unachievable for conventional, in-service composite laminates. While immersion tests can be implemented to establish achievable service-life design allowables when predicting 'worst-case' mechanical properties based on moisture absorption, they are inadequate for predicting degradation rates. This result highlights the need for secondary testing to establish percent moisture at saturation for the FRPMC under investigation through longer term, higher frequency single-sided exposure. Single-sided exposure testing represents the pooling of a contaminant on a composite surface. From a pragmatic perspective, it would be uncommon to find circumstances for which a composite used in an internal application would reach saturation through continuous, single-sided exposure. Hence, inspection frequencies may be proscribed based on established, component-specific design allowable and the observed, short-term relationship between moisture ingress and decrement to mechanical properties. This, however, negates cumulative contributions to degradation brought about by cyclic exposure, necessitating further investigation.

4.2 Surface Microscopy

Figure 3 shows optical microscopy performed on sample surfaces following direct liquid exposure for three months compared with a control sample. Microscope images were captured under identical lighting conditions, subject to 3.5x magnification, using an Olympus SZH-10 stereo microscope. All samples were directly exposed to liquid on their 'bag-side'; what are observed as small square tessellations is the impression of the plain-weave nylon peal-ply used to extract the composite laminate following curing. All three-month conditioned samples exhibited a distinctive yellow tinge, most notably for ethylene glycol samples, for which colour change was noted after as early as one week. Interestingly, conditioning ethylene glycol displayed a transition from colourless to yellow which increased in intensity with increasing exposure duration. Subsequent testing revealed that this appearance of colour was correlated with ethylene glycol's exposure to the composite sample and not do to leaching of the sample array's constituent components, nor elevated temperature exposure alone. Further chemical analysis is required to establish the cause of this colour change.

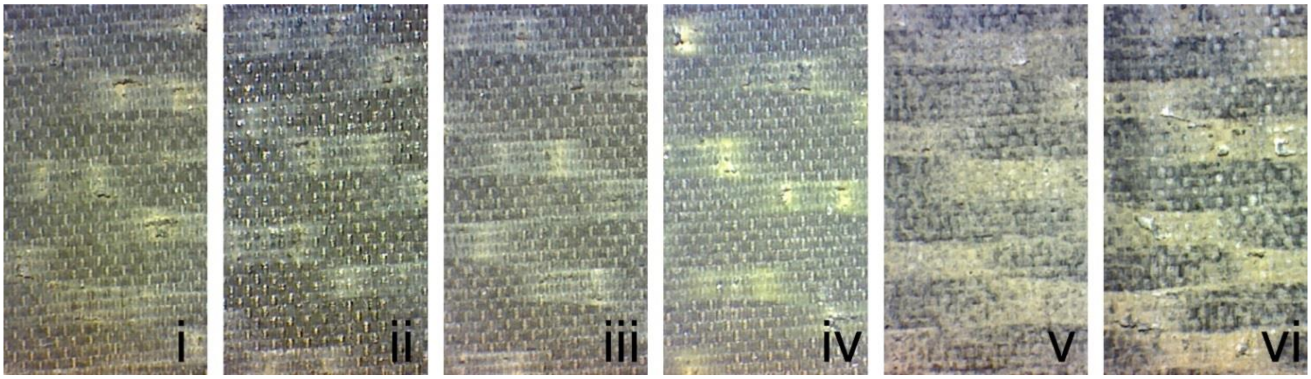

Figure 3. Surface structures of samples exposed to i) air (control), ii) hydraulic fluid, iii) turbine oil, iv) ethylene glycol, v) ocean water and vi) deionized water, respectively, after a three-month conditioning duration

The control, hydraulic fluid and turbine oil samples exhibited the same degree of surface voids with a visually indistinguishable variation in size and distribution. Conversely, deionized water samples demonstrated considerable surface degradation, with large voids evident in the surface epoxy. Sheen, a characteristic of light reflecting from the polished fibre surface, is visible in larger voids, illustrating degradation down to the top fibre layer. Figure 4 shows changes in surface degradation as a function of exposure duration for deionized water samples. Increasing exposure duration demonstrated a clear correlation to surface degradation which is more apparent in deionized water samples than for the other liquids investigated. The unconditioned sample surface exhibits a representative degree of void formation inherent to the curing process, as well as the composite's innate, black colour, as prescribed by the supplier. Notably, after one-day deionized water exposure, the sample's surface exhibited a stark contrast between bevelled regions in the top-ply where more epoxy could collect during impregnation and regions where top-ply fibres were closer to the vacuum bag and thus, are coated in a thinner layer of epoxy. Thicker epoxy regions transition from black to white, with increased void formation more prominent in resin-rich regions. Voids in ethylene glycol samples were larger and appeared in a greater concentration than those observed in the hydraulic fluid or turbine oil samples but were less prevalent than those observed in the deionized water samples. Ocean water samples exhibited a coating of salt precipitate on the exposed surface. While voids were visible, the precipitate largely masks visibility of the sample surface and thus, hides possible degradation of surface epoxy. Microscopy results call for further, quantitative work investigating the association between void formation and observed decrease of mechanical properties resulting from environmental degradation.

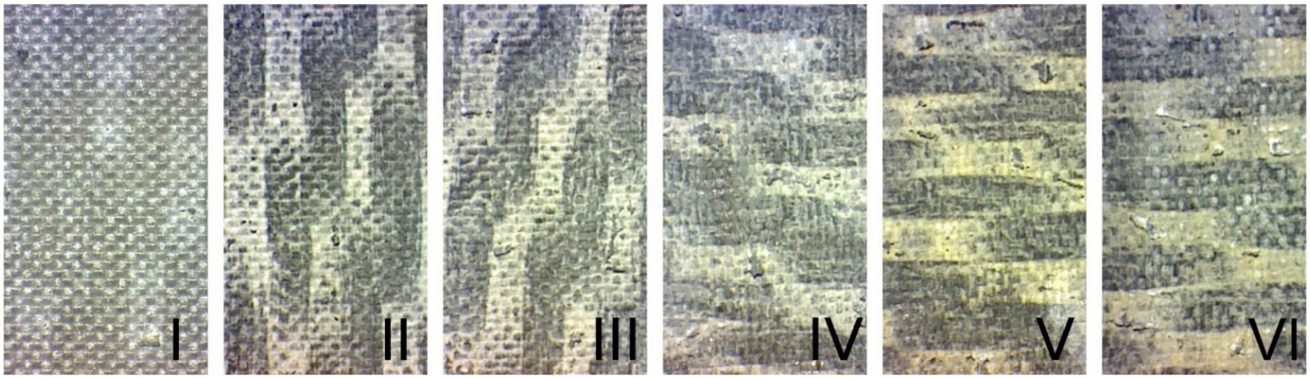

Figure 4. Surface structures of deionized water samples conditioned for I) 0, II) 24 III) 168, IV) 720, V) 1440 and VI) 2160 hours, respectively

4.3 Flexural Properties

Figure 6 and Figure 6 summarize the flexural data obtained for all conditioned samples with each column representing the average, normalized maximum flexural strength (flexural strength) or flexural modulus of elasticity (flexural modulus) of its respective sample group. Horizontal, dashed lines about the one value represent the standard deviation of equivalent, unconditioned flexural samples. Due to material availability constraints, sample groups fell in to one of two categories; visible bag surface fibres parallel to, or visible fibres transverse to sample length. This distinction was made to limit uncontrolled variability as preliminarily testing demonstrated a bimodal distribution of maximum flexural strength correlated with surface fibre orientation. One-day and one-week samples (represented in yellow and grey) possessed parallel fibres and longer durations (represented in blue and green) possessed transverse fibres. Flexural properties were normalized for each sample with respect to their associated bag-side fibre orientation.

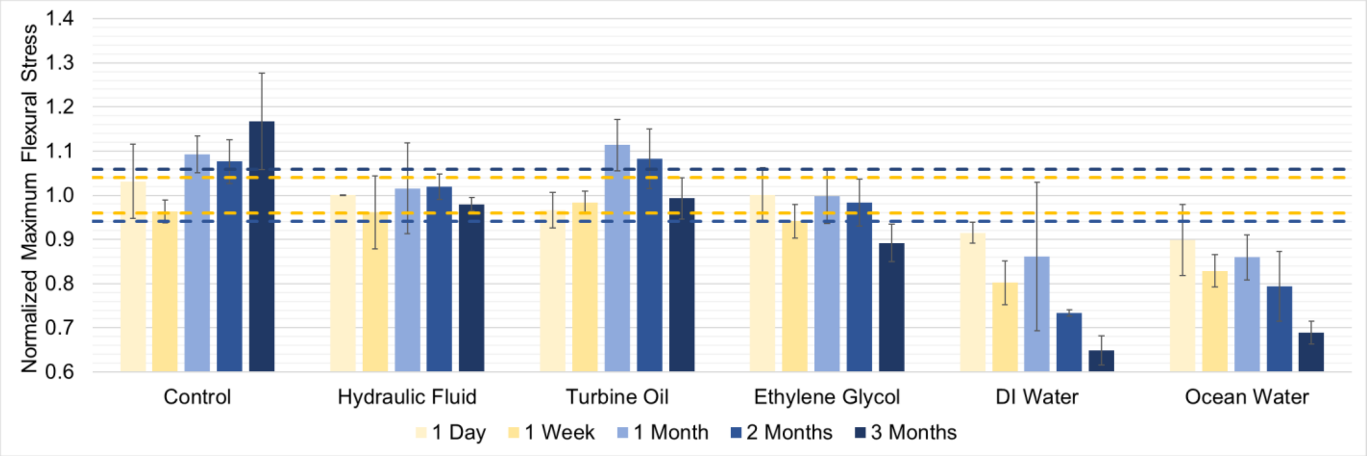

Figure 5. Normalized flexural strengths as a function of conditioning liquid and duration

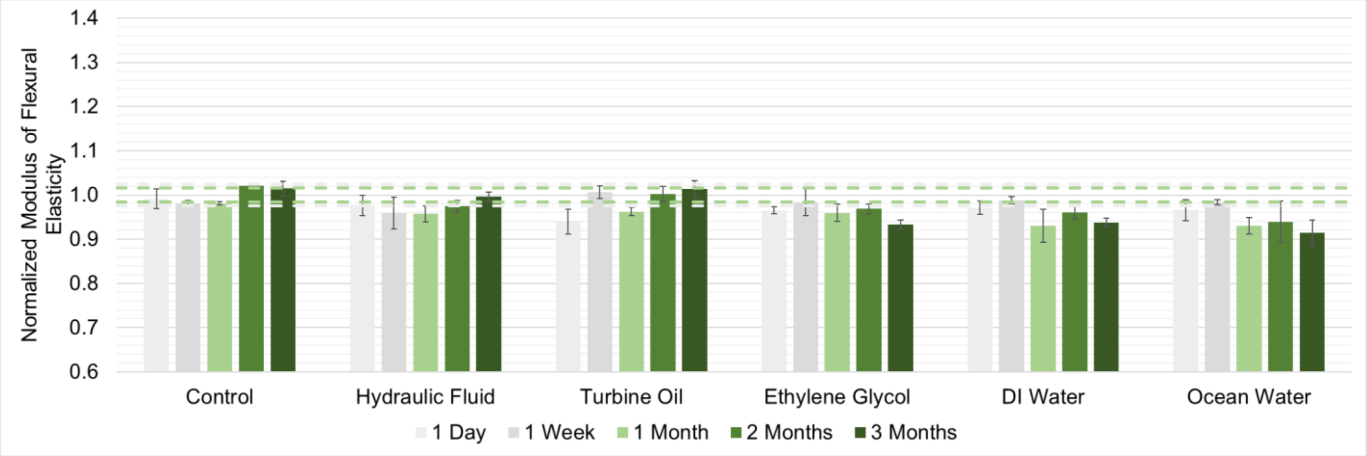

Figure 6. Normalized flexural moduli as a function of conditioning liquid and duration

Flexural strength results demonstrated a wider distribution of values for each sample group compared to the values of flexural modulus. As a matrix-dominated parameter, the flexural strength is highly dependent on void content. Hence, large standard deviation may be attributed to the laminate's observed high void content. This conclusion is supported by preliminary testing which measured the flexural properties of a 6061-aluminum standard using the same testing apparatus. Similarly, changes to the flexural moduli were less substantial than changes to flexural strengths as the observed degradation is attributed to chemical changes to the epoxy matrix. Micro-stresses brought about by irregularity in the bag-side compression surface may also contribute to mechanical variability.

Observed trends in flexural strength mirrored those of flexural moduli: with increasing duration control samples showed a net increase in flexural properties; ethylene glycol, deionized water and ocean water samples showed a net decrease in flexural properties; and hydraulic fluid and turbine oil samples showed a negligible change in flexural properties. Through statistical analysis, assuming all sample groups exhibited a normal distribution, using a two-tailed, t-statistic with a 99% confidence interval, all deionized water flexural strengths, with exception of the three-month duration, showed a statistically significant ($p < 0.01$) decrease. Likewise, ocean water flexural strengths, excluding one-day and one-month durations, showed a statistically significant decrease. However, deionized and ocean water samples of the same duration did not show a statistically significant difference from one another, demonstrating that the aqueous salts dissolved in the ocean water did not have an appreciable contribution to the degradation of epoxy FRPMCs as supported by literature investigating similar materials [2]. Notably, deionized and ocean water samples showed an appreciable (8.5% and 10.2%, respectively) degradation after a single day and substantial degradation (35.1% and 31.1%, respectively) after three months of exposure. Similar analysis of control, hydraulic fluid, turbine oil, and ethylene glycol samples did not show a significant change in flexural strength, forcing the acceptance of the null hypothesis: The exposing chemical does not cause a change in the tested composite's flexural strength. The observed increase in flexural strength of control samples may be the result of a long term post-cure which demonstrates significance over longer durations. Likewise, the decrease in flexural strength of ethylene glycol exposed samples may prove significant over longer durations.

Statistical analysis of flexural moduli showed no statistically significant results over one-day and one-week exposures. Over longer-term durations, deionized water, ocean water and ethylene glycol all exhibited statistically significant decreases in flexural moduli however, inconsistently. This result indicates that ethylene glycol has a greater propensity to disrupt bonds at the fibre-matrix interface, decreasing load transfer from matrix to fibre, than it does to break internal bond within the epoxy matrix. No control groups showed statistically significant changes from the moduli of unconditioned samples. Hydraulic fluid and turbine oil showed a statistically significant drop in flexural moduli only for one-month samples although neither demonstrated a common trend. Noted anomalous data from the one-month sample groups indicated greater than anticipate flexural strength and lower than anticipate flexural moduli. Hence, it is believed that non-material specific differences resulted from either one-month sample conditioning or one-month sample flexural testing, although explicit causality is unclear.

Figure 7 compares changes in the material's flexural strength as a function of moisture absorption. Linear trendlines show that hydraulic fluid and turbine oil do not exhibit a correlation between liquid uptake and decrement of flexural strength, however, there is an evident association between moisture uptake of deionized water, ocean water and ethylene glycol and a decrease in flexural strength. Such a result demonstrated that, while carbon-fibre reinforced epoxy has the propensity to physically absorb both polar and nonpolar liquids, there is a unique, degradative interaction between investigated polar solvents and the epoxy matrix. Comparing the obtained results and the molecular structures of the tested liquids, it seems logical that the degradation of the epoxy matrix is brought about by hydroxyl interactions between the liquids and hydrogen bonds of the epoxy matrix. Supported by literature [17], [18], water disturbed hydrogen bonding of the epoxy matrix lowering matrix-dominate mechanical properties. This process may then be reversible, contingent on the removal of the liquid (i.e. through evaporation) while preventing diffusion of the cleaved units from the bulk matrix. With longer duration exposure, deionized water sample wells exhibited a cloudiness resulting from the formation of white precipitate. It is believed that this precipitate is cleaved epoxy subunits which have been freed from the bulk matrix and allowed to diffuse into solution. Inducing such a process of matrix leaching, subunit migration, subunit re-deposition and/or removal may lead to accelerated degradation beyond that of static exposure to moisture, the evaluation of which is paramount based on the cyclic nature of aircraft environmental exposure. Literature investigating the effects of cyclic exposure of epoxy matrix composites to condensation and ultraviolet radiation has demonstrated a synergistic degradation brought about by cyclic exposure [15]. Hence, these observations call for further work to identify the molecular structure of the observed precipitate and to investigate the effects of long-term cyclic exposure of epoxy matrix composites to water-based solvents on flexural properties. Furthermore, validation of results should be performed through the exposure of an epoxy, FRPMC to a comparable, short chain, hydroxyl bearing solvent, such a propanol or butanol. Analysis of flexural properties has demonstrated OEM qualification testing may be confined to water based conditioning for regular internal, epoxy based FRPMC applications. This practice can effectively characterize the most common and most detrimental contaminant while offering cost-savings through the reduced material and labour requirements of a more focused test campaign.

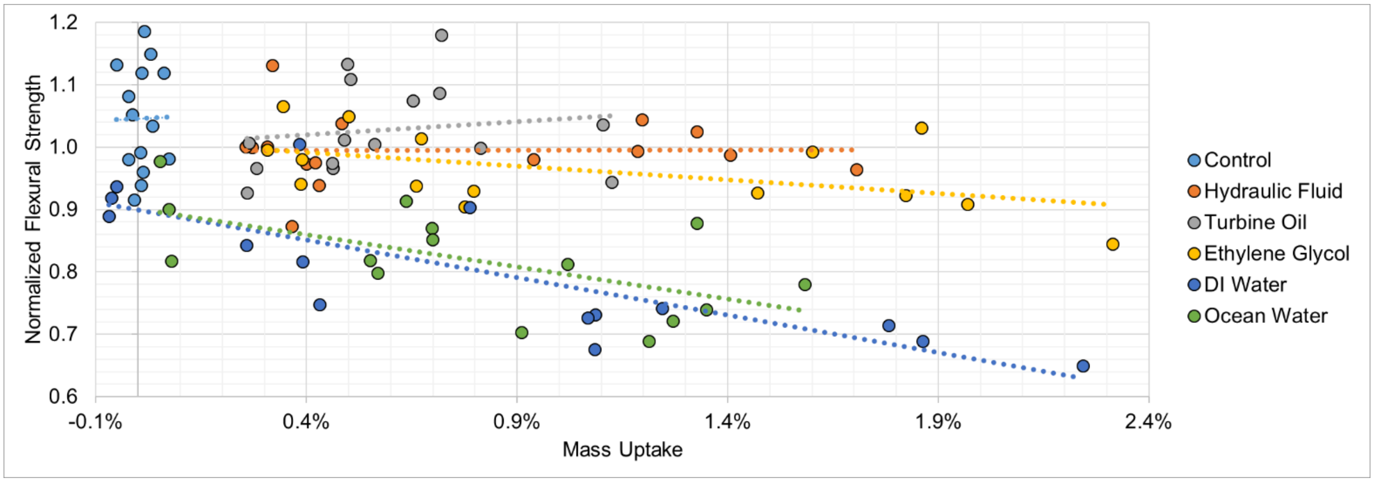

Figure 7. Normalized flexural strength as a function of percentage change in mass resulting from fluid uptake

5 SUMMARY AND CONCLUSIONS

This investigation characterized surface degradation and quantified liquid absorption rates and decrement to flexural properties of an epoxy FRMPC, brought about by single-surface exposure to various aerospace fluids at elevated temperatures over a three-month period. Results demonstrated a statistically significant, decrease in flexural properties of the FRPMC samples exposed to both deionized and ocean water while simultaneously demonstrating an insignificant change to the flexural properties of samples exposed to both hydraulic fluid and turbine oil. Exposure to ethylene glycol showed some statistically significant detriment to flexural properties, however damage induced was smaller in magnitude than that observed for samples exposed to water. Microscopy presented visual surface degradation mirroring results of flexural property analysis. Interpretation of moisture absorption data demonstrated the propensity of the FRPMC to absorb liquids is independent of their molecular structure for tested liquids, although molecular structure has a clear affect on moisture absorption rate and induced degradation. Additionally, single-side exposure testing revealed the unsuitability of common, immersion testing to predict absorption rates of FRPMC in a realistic exposure scenario.

The performed study leads to future work investigating longer-term rates of moisture absorption and saturation levels for aerospace fluids in FRPMCs; chemical interactions between hydroxyl possessing solvents and epoxy matrix composites; quantitative analysis of void formation and its correlation to mechanical properties; and the effects of cyclical liquid exposure on degradation rates.

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