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Accelerated Hygrothermal Stabilization of Composite Materials

J. A. Gale
(M.S. Thesis)

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Accelerated Hygrothermal Stabilization of Composite Materials

by

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THESIS

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Abstract

An accelerated moisture conditioning scheme to prepare laminated graphite/epoxy (Gr/Ep) composites for use in precision applications was investigated. The experimentation validated a simple moisture conditioning scheme to prepare Gr/Ep composite parts for use in precision applications by measuring dimensional changes over 90 days. It was shown that an elevated temperature moisture conditioning scheme produced a dimensionally stable part from which precision structures could be built/machined without significant moisture induced dimensional changes after fabrication. Conversely, it was demonstrated that unconditioned Gr/Ep composite panels exhibited unacceptably large dimensional changes (i.e., greater than 125 ppm) for use in precision applications. It was also shown that the time required to produce stable parts was shorter, by more than an order of magnitude, employing the conditioning scheme than using no conditioning scheme (46 days versus 1000+ days). Two final use environments were chosen for the experiments: 50% RH/21°C (70°F) and 0% RH/21°C (70°F). Fiberite 3034K was chosen for its widespread use in aerospace applications. Two typical lay-ups were chosen, one with low sensitivity to hygrothermal distortions and the other high sensitivity: $[0, \pm 45, 90]_S$, $[0, \pm 15, 0]_S$.

By employing an elevated temperature, constant humidity conditioning scheme, test panels achieved an equilibrium moisture content in less time, by more than an order of magnitude, than panels exposed to the same humidity environment and ambient temperature. Dimensional changes, over the 90 days of the experimentation, were up to 4 times lower in the conditioned panels as compared to the unconditioned panels.

Analysis of weight change versus time of test coupons concluded that the out-of-autoclave moisture content of Fiberite 3034K varied between 0.06 and 0.1%. These values agreed with other findings.

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List of Symbols

a	fitting coefficient
A	axial stiffness matrix from laminated plate theory
A'	manipulated form of axial stiffness matrix
b	fitting coefficient
c	moisture concentration
C	specific heat
B	extension/bending coupling matrix from laminated plate theory
B'	manipulated form of extension/bending coupling matrix
CME	coefficient of moisture expansion
CTE	coefficient of thermal expansion
D	moisture diffusion coefficient
D	bending stiffness matrix from laminated plate theory
D'	manipulated form of bending stiffness matrix
FUE	final use environment (level of environmental saturation, % RH)
h	laminate thickness
K	thermal conductivity
M	panel moisture content
M_i	initial panel moisture content
M_m	maximum panel moisture content
<u>M</u>^{NM}	non-mechanical moment vector from laminated plate theory - analogous to <u>M</u>
<u>N</u>^{NM}	non-mechanical load vector from laminated plate theory - analogous to <u>N</u>
N_O	Avagadro's number

p_i, P_i	partial pressure of component i
Q_{ij}	elastic constants, laminated plate theory
ppm	parts per million
R	gas constant
R	universal gas constant
s	panel thickness
S_0	proportionality coefficient
t	time, thickness
T	absolute temperature
x, y	in plane, orthogonal directions of plate
z	through-the-thickness direction of plate, orthogonal to x, y
α	coefficient of thermal expansion
β	coefficient of moisture expansion
ΔH	heat of solution
$\underline{\epsilon}^0$	vector of plate mid-plane strains
$\underline{\kappa}$	vector of plate curvatures
μ_i	chemical potential of component i
μ_i^0	Gibbs free energy per mole of component i at unit pressure and same temperature as solution of interest
ϕ	relative humidity
χ_i	mole fraction of component i

Introduction

Owing to their high specific strength and stiffness, graphite/epoxy (Gr/Ep) composites have established themselves as highly versatile materials for aerospace applications. These materials have been implemented in aerospace applications requiring a high degree of dimensional stability such as telescope metering tubes, satellite dish supporting structures and optical benches (hereafter referred to as precision¹ applications). Unfortunately, these materials also exhibit undesirable hygroscopic behavior (i.e., they absorb moisture from the environment) that results in alteration of the residual stress state, degradation of the matrix dominated material properties and unacceptably large dimensional changes due to swelling of the matrix (referred to as the moisture problem). In several precision space applications the moisture problem required significant design and engineering effort to resolve: for example, the Mars Observer Camera [2], Thematic Mapper [3], and TEAL-RUBY [4] programs. An effective processing scheme to minimize the effects of this hygroscopic behavior is essential to the continued use of Gr/Ep composites in precision applications.

The research addressed the problems of implementing hygroscopic Gr/Ep composites in precision applications: (1) Dimensional changes due to moisture sorption can be orders of magnitude larger than dimensional tolerances; (2) At ambient temperature, the equilibration time (i.e., the time beyond which no dimensional changes take place) can be months to years long. The goals of the research were to validate an elevated temperature moisture conditioning process to shorten the equilibration time and verify that the parts subjected to the process were dimensionally stable. A computer code,

¹In the context of this work, the term precision is defined as dimensional changes less than or equal to 125 ppm (i.e., 0.025 mm (0.001 in) over a 203 mm (8 in) gage length). Krumweide, et al [1] have categorized response to moisture as a micro distortion (a source of dimensional instability on the order of 0.025 μm (1 μin). With the sensitivity to moisture of the materials under consideration, the moisture induced distortions fall under both this author's definition of a macro distortion (order of 25.4 μm (1 mil)) and the above definition of precision.

based on laminated plate theory, was used to choose two lay-ups for the experimentation based on sensitivity to moisture induced dimensional changes. The same code generated theoretical predictions. Gravimetric and dimensional measurements provided data.

In the area of precision composites, much work has focused on thermally induced dimensional instability (expansion, creep/stress relaxation). The magnitude of the problem has been extensively quantified. The problem investigated in the following work was moisture induced dimensional instability. As an example and direct comparison of the magnitudes of the respective effects, consider a thin, 1.4 mm (0.056 in) quasi-isotropic flat panel 254 mm (10 in) long that is to be used in a low earth orbit (LEO) satellite. The panel is fabricated from Fiberite 3034K (T300 fibers and 934 resin system). After the panel is removed from the autoclave, it is handled and/or stored for 2 months in a room temperature, 50% RH environment before being incorporated into the satellite and launched. In orbit, this panel experiences uniform temperature excursions on the order of 121°C (250°F). These uniform temperature excursions correspond to in-plane dimensional changes of approximately 0.13 mm (0.005 in). The same panel, when exposed to the dry environment of space, desorbs the moisture gained during the 2 month handling/storage (0.6% by dry weight). This desorption causes an in-plane dimensional change of approximately 0.6 mm (0.0023 in). In light of the relative magnitudes, the moisture problem plays a significant role in the use of graphite/epoxy (Gr/Ep) composites in precision applications.

One partial solution to the moisture problem in terms of dimensional stability is to obtain a chemical potential² of water in the composite part equal to that in the intended final use environment (FUE) through processing control. Other schemes involve the use of moisture barriers (coatings) [5] or the selection of materials less susceptible to the

²The chemical potential of water is a quantification of the driving force for moisture absorption and subsequent diffusion.

moisture problem (thermoplastic resins, hydrophobic resins, etc.). Assuming that a Gr/Ep material system has been chosen for a particular precision application, then coatings and processing control are the two remaining alternatives. Coatings are a viable option for applications where the FUE moisture level is unknown and/or changing. With the use of coatings comes penalties: increased weight and, where zero CTE (coefficient of thermal expansion) laminates have been designed, the loss of this desirable trait [5].

Processing control, conversely, works well only when the FUE moisture level is both known and constant. Another disadvantage of processing control is the length of time required to condition the parts to the FUE moisture level. By way of example, the panel in the previous case would take approximately 1300 days to reach equilibrium under room temperature (21°C, (70°F)) and 50% RH. Fortunately there has been much work in the area of accelerated moisture conditioning of composites, notably the contributions of Ciriscioli, et al [6] that have established schemes to reduce conditioning times by more than an order of magnitude as compared to above.

While the literature revealed extensive work in rapid moisture conditioning of composites, it also indicated much of this work was in support of moisture degraded material property tests. Several studies were conducted to understand the role moisture plays in altering the visco-elastic response and how it affected dimensional stability. The experimentation intended to validate a simple moisture conditioning scheme to prepare Gr/Ep composite parts for use in precision applications by measuring dimensional and weight changes over 90 days.

Summary of Relevant Work

Recent work in the areas of rapid moisture conditioning and dimensional stability of organic matrix composites has largely occurred independently. The primary motivation for rapid conditioning of these materials has been to quickly prepare test samples for use in determining moisture modified material properties: tensile, compressive, short beam shear strengths, elastic moduli, glass transition temperature, to name a few [7]. Studies concluded that to predict dimensional stability, an understanding of both the extent to which moisture alters the visco-elastic properties of the matrix and the mechanisms of diffusion were required.

In the area of rapid conditioning, Ciriscioli's work is well recognized. In this referenced work, Ciriscioli et al developed and validated a conditioning method that shortened conditioning times by up to a factor of three for thick laminates as compared to the scheme used herein.

Several matrix materials have been studied for dimensional stability: Fiberite 930, 934 [8], Ferro CE-339 [9], and Narmco 5209 [10, 11]. Precision ball point micrometers [10] with 0.03 mm (0.1 mil) resolution to intricate laser optical comparators [11], laser interferometry and Fabry-Perot schemes have been used to measure the phenomena. A common technique to amplify the moisture induced distortions and to assess the alteration of residual stresses was to use unsymmetric laminates and measure changes in cord height of the curved panel as a function of absorbed moisture [10, 11, 12].

The dual role moisture plays in causing dimensional instability has been investigated: causing volumetric changes and plasticizing the matrix. The dual role of moisture absorption has been described in the following manner: Moisture absorption causes strains due to volumetric expansion of the matrix and also alters the visco-elastic properties of the matrix. These strains give rise to internal stresses which then relax due to the visco-elastic nature of the matrix. The relaxation of the residual stresses recovers a portion of the volumetric strains, causing dimensional instability. These studies concluded

that in order to predict long term dimensional stability, an understanding of how moisture alters the visco-elastic properties of the matrix materials was required. Crossman et al [11] have found that the effect of moisture on the visco-elastic response of 934 resin was describable using a time/temperature superposition technique. This shifting technique allows construction of a master curve of relaxation modulus ($E_R(t) = \sigma(t)/\epsilon(t)$) versus log time over various equilibrium moisture levels and temperatures. By knowing the applied and/or residual stress level and a time of interest, one could calculate the magnitude of creep as a function of moisture content at any length of exposure. The authors also found that a given level of creep was reached at a lower temperature in a wet specimen as compared to an identical dry specimen due to the plasticizing phenomena (i.e., the addition of moisture is analogous to heating in terms of increased creep). These and other authors [13] have concluded that moisture and temperature have a coupled effect on the shifted data.

Several groups of researchers [10, 11, 12] have investigated the coupled role of moisture as a swelling agent and plasticizer by measuring the residual stresses in an unbalanced laminate (by means of change in curd height of a curved test piece) as a function of moisture content and exposure temperature. An important finding from that work was that the alteration of the residual stress state due to moisture absorption was influenced by the temperature at which the exposure took place, and that as the exposure temperature increased, the moisture-induced swelling reduced the residual stresses less and less [11].

Considerable effort has been expended in the study of moisture diffusion phenomena: rate and equilibrium moisture content. While many studies have concluded that the diffusion phenomena was describable by Fick's Law, Blikstad et al [14] have noticed non-Fickian behavior after an extended duration (400⁺ days) in several matrix materials: Fiberite 934, 914, 920, and Narmco 5208. In the case of Fiberite 934, the authors found that an apparent moisture equilibrium was reached as predicted by Fick's

law, however, over long exposures this initial plateau changed and a second phase of moisture absorption was exhibited. One author found weight changes between plateaus on the order of 0.3% by weight. Had the sample example panel (page 1) been mistakenly equilibrated to the first plateau, the subsequent moisture absorption would have caused additional dimensional changes on the order of 130 ppm.

Analytical Background

For this study, theoretical calculations provided necessary experimental design information. The calculations determined (1) expected mid-plane strains and curvatures as a function of time and moisture distribution; (2) time to reach equilibrium moisture content as a function of temperature; (3) equilibrium moisture content as a function of environmental saturation. The derivations of the equations used to determine the above quantities are detailed in Appendix A.

Mid-plane strains and curvatures were determined using laminated plate theory modified for hygrothermal distortions (moisture and temperature). In matrix form, the equations solved took the following form, where the matrices A' , B' , and D' are derived from the axial stiffness matrix A , extension/flexure coupling matrix B and bending stiffness matrix D :

$$\begin{Bmatrix} \epsilon^0 \\ \kappa \end{Bmatrix} = \begin{bmatrix} A' & B' \\ B' & D' \end{bmatrix} \begin{Bmatrix} N^{NM} \\ M^{NM} \end{Bmatrix} \quad (1)$$

where $\underline{\epsilon}^0$ = vector of mid-plane strains (or $\{\epsilon^0\}$)

$\underline{\kappa}$ = vector of plate curvatures (or $\{\kappa\}$)

A' = manipulated form of axial stiffness matrix A

B' = manipulated form of extension/bending coupling matrix B

D' = manipulated form of bending stiffness matrix D

$\underline{N}^{NM}$ = non-mechanical load vector (or $\{N\}^{NM}$)

$\underline{M}^{NM}$ = non-mechanical moment vector ($\{M\}^{NM}$)

The necessary input to solve for the mid-plane strains $\{\epsilon^0\}$ and curvatures $\{\kappa\}$ were the temperature and moisture profiles through the thickness of the panel as functions of environmental boundary conditions and time. At discrete points in time these two profiles were used to generate the non-mechanical load and moment vectors, $\{N\}^{NM}$ and $\{M\}^{NM}$, from which discretized mid-plane strain and curvature values were determined.

To obtain these two profiles required solving the two governing differential equations describing heat and moisture transport in the panels: one-dimensional Fick's and Fourier's laws, respectively (see Appendix A, Figure 13, for coordinate system definition).

$$\frac{\partial c}{\partial t} = \frac{\partial}{\partial z} D_z \frac{\partial c}{\partial z} \quad (2)$$

$$\rho C \frac{\partial T}{\partial t} = \frac{\partial}{\partial z} K_z \frac{\partial T}{\partial z} \quad (3)$$

where c = moisture concentration

C = specific heat

D_z = coefficient of moisture diffusion in plate thickness direction

t = time

ρ = density

K_z = thermal conductivity

T = absolute temperature

Springer [15] developed a one dimensional finite difference code, W8GAIN, to generate moisture profiles for varying environmental boundary conditions. Several assumptions were used to obtain a simplified set of governing equations:

- (1) Temperatures inside the panels equilibrate much faster than the moisture, so at each point in time, the internal panel temperature is equal to the ambient temperature (essentially uncoupling heat and moisture transport);
- (2) ρ , C , K_z , D_z depend only on temperature.

The second piece of experimental design information was the time to reach moisture equilibration as a function of temperature and under a constant moisture boundary condition. Assuming D was independent with respect to moisture

concentration³, this amounted to solving equation (2). The solution to the equation governing this type of problem (Fick's 2nd Law), after simplification, was given by Jost [17]:

$$t = \frac{s^2}{D_z} \left[-\frac{1}{7.3} \ln \left(1 - \frac{M - M_i}{M_m - M_i} \right) \right]^{\frac{1}{0.75}} \quad (4)$$

where s = panel thickness (for two sided exposure)

s = twice panel thickness (for one sided exposure)

D_z = through thickness diffusion coefficient (f(T), only)

M = target moisture content of panel (% of panel dry weight)

M_i = initial moisture content of panel (% of panel dry weight)

M_m = maximum moisture content of panel at a given environmental concentration (% of dry panel weight)

The last piece of information was the result of work by Shen and Springer [18] who derived an expression for M_m (independent of temperature) based on relative humidity:

$$M_m = a\phi^b \quad (5)$$

where ϕ is the relative humidity and a and b are fitting coefficients ($a=0.017$ and $b=1$ for Fiberite 3034K, [16]).

³Loos and Springer [16] have experimental data to suggest D is independent of moisture concentration for Fiberite 3034K material system.

Experimental Program and Methods

The overall goal of the experimental program was to validate a rapid moisture conditioning scheme for use in the manufacture and processing of Gr/Ep composites for precision applications. The intent was to show that the use of the rapid conditioning scheme would produce a dimensionally stable panel in a given constant moisture environment in a short time and that unconditioned panels would exhibit dimensional changes in excess of 125 ppm.

A second goal and potential area of further research was to establish the moisture content of parts fabricated from Fiberite 3034K directly out of the autoclave. There was only a brief reference to a range of values, 0.2% - 0.3%, found in the literature [19]⁴. While this value was determined experimentally for a single resin system during the course of the experimental program, this area deserves further analytical and experimental work.

The main portion of the experimental program involved conditioning one group of Gr/Ep panels to two different FUE moisture levels using an elevated temperature (82°C (180°F)) conditioning scheme. A second, identical group of panels was allowed to equilibrate to the two different FUE moisture levels at ambient temperatures (21°C (70°F)). Subsequent measurement of several reference dimensions as a function of exposure time at the FUE moisture level quantified the dimensional stability of the two groups of panels. Figure 1 shows a flow chart of the experimental program.

The material system, chosen for its wide use in aerospace applications, was Fiberite 3034K, consisting of Amoco T-300 fibers (12K filaments per tow, 220-241 GPa (32-35 Msi) modulus and 934 resin system (177°C (350°F) cure). A total of 8 panels roughly 305 mm x 406 mm x 1.4 mm (12"x16" x 0.056") were laid up in two equal groups of $[0, \pm 45, 90]_S$ and $[0, \pm 15, 0]_S$ configurations. Initial analytical work established the choice of lay-ups based on the desire to have "low" and "high" sensitivities

⁴It is reasonable to conclude that the amount of initial moisture in an autoclave cured part is a function of resin system, cure cycle (temperatures, pressure, vacuum), lay-up, part geometry, etc.

Figure 1. Program Flowchart

to moisture induced dimensional changes. The lay-ups were also chosen to be commonly used configurations. The 8 laminates were separated into two groups based on conditioning scheme: rapid versus ambient (unconditioned). The panels within each group were segregated by FUE moisture level and sensitivity. The two FUE moisture levels were space and nominal where the respective level of saturation in each environment was 0% RH⁵ and 50%, respectively. Figure 2 schematically shows the distribution of parts in terms of conditioning scheme, FUE, and sensitivity.

⁵ See Appendix B

Figure 2. Distribution of Panels

The parts were fabricated following the manufacturer's instructions (lay-up procedure L-6 and cure cycle C-9, [20] for autoclave curing. Figure 3 shows a cross-section of the components in the lay-up. Upon cure, samples from each laminate were removed and tested for fiber and void volume fractions using an acid digestion method [21], and density using a water immersion technique. In addition, small sections parallel and perpendicular to the zero degree direction of the panel were cut, polished and photographed. The photomicrographs showed no visible voids or de-laminations at 80x magnification. Figure 4 shows a photomicrograph at 500X of the bottom 2 plies of a rapidly conditioned, low sensitivity panel $[0, \pm 45, 90]_s$, exposed to the nominal moisture environment (50% RH). The same samples were viewed on a Nikon Profile Projector where the ply thicknesses were measured. These measurements, along with the fiber and void volume fractions, the densities and photomicrographs were all used to evaluate both the quality of the cure and of the parts derived from that cure. At the same time, small coupons, roughly 102 mm x 102 mm (4"x4"), were removed, identified with the parent plate identifier code, weighed and placed in a vacuum oven operating at 82°C (180°F). Weights of each coupon were taken at 2 day intervals for the first 16 days then at 4 day

intervals, using a Mettler analytical balance (0.00001 g resolution) until effective equilibrium was reached⁶. This final weight was used in computing the moisture content of each panel after cure.

Lay-up Components:

A: Peel Ply	G: Solid Release Film
B: Porous Teflon	H: Vacuum Bag
C: Solid Release Film	I: Surface Breather
D: Upper Caul Plate	J: Resin Dam
E: Pre-preg	K: Lower Caul Plate
F: Peel Ply	

Figure 3. Vacuum Bag Components and Layering

⁶ The definition of "effective equilibrium" is given by ASTM procedure D 5229 [22]. A part is considered to be in equilibrium with a surrounding moist environment when the change in weight of the part between two successive readings, at least one day apart, has changed by less than 0.01% of the initial baseline weight of the part.

Figure 4. Photomicrograph at 500x of Bottom 2 Layers of a $[0, \pm 45, 90]_s$ Panel

Immediately following coupon removal, the panels were rough trimmed to 280 mm x 280 mm (11" x 11") and weighed on a Mettler balance of 0.0001 g resolution. As soon as possible after the initial weighing, reference surfaces were attached to each panel in the general configuration shown in Figure 5. The reference surfaces consisted of Gr/Ep blocks (13 mm x 13 mm x 13 mm (1/2"x1/2"x1/2")) and 6.4 mm (1/4") diameter stainless steel ball bearings (grade 50) bonded to the center of each block. Lateral edges were sealed at the same time using 13 mm (1/2") wide aluminum foil and 2135 epoxy adhesive (Tra-Con

Polymers) to encourage moisture absorption and diffusion through the faces of each panel. An overcoat of Seran F310 sealer was applied to the bond lines between the blocks and the panels, the balls and the blocks and the exposed surfaces of the blocks themselves. The panels were weighed again after this step to establish the weight of the reference surfaces, aluminum foil, adhesive and sealing material.

Figure 5. Reference Surface Attachment Locations

Once all the panels were prepared, the panels in the ambient equilibration groups were inspected using a DEA Coordinate Measuring Machine (CMM, model 0107) to establish the baseline reference dimensions. These panels were then placed in two humidity

chambers representing the two FUE's, i.e., space: 21°C (70°F)⁷ and 0% RH; and nominal: 21°C (70°F) and 50% RH. A vacuum chamber running at ambient temperature served as the space chamber, while the nominal chamber was an insulated Plexiglas container employing distilled water and three fans to maintain the desired environment. A PC controlled both the chamber temperature and humidity and logged the data at 30 minute intervals.

At the same time as the panels in the ambient equilibration group were undergoing initial inspection, the conditioning process was begun for the panels in the rapid equilibration group. The two panels for the space FUE were placed in a vacuum oven set at 82°C (180°F) and monitored for weight changes at 2 day intervals until effective equilibrium was reached. This procedure was also used on the nominal panels but a Tenney humidity chamber set to 82°C (180°F) and 50% relative humidity was used. Upon completion of the rapid conditioning, the panels were inspected using the CMM to establish baseline dimensions and then placed in their respective FUE humidity chambers alongside the ambient temperature equilibrated panels. At 7 day intervals, the ambient panels were removed from their humidity chambers, weighed, inspected and returned. This process was duplicated for the rapid panels once they had achieved equilibrium. The panels were transported to and from the chambers and the weighing and inspection facilities inside sealed plastic bags, each containing a bag of desiccant. The panels were also stored in these bags while awaiting weighing and inspection. As the data will show, this bagging procedure was only partially successful in limiting the variation in the environment seen by the panels during transportation, weighing and inspection.

All mass measurements were conducted following NIST standard operating procedures [23] for single substitution mass determination.

⁷ While it is obvious that the temperature in space is not 21°C (70°F), the intent of the work was to quantify the effect of moisture on the dimensional stability, not temperature. Therefore, the 21°C (70°F) was chosen to match the temperature of the room in which the dimensional measurements were taken.

Experimental Results

Data acquisition was terminated for this paper after 84 days of exposure due to time constraints and the lengthy process of ambient temperature equilibration. Data will be taken for several more months at 1 month intervals to investigate longer term behavior and to corroborate or discount two-phase diffusion behavior as reported by Blikstad [14].

During processing, several steps were taken to assess the quality of the post-cured panels. Photomicrographs of small sections of each panel were examined. Individual ply thicknesses were measured, the results of which can be found in Table 2, in Appendix C. The average thicknesses were in good agreement with each other and with the theoretical value. Fiber and void volumes were determined using an acid digestion technique. The calculated fiber volumes are listed in Table 3 in Appendix C and were within the target range of 57-63%.

Panel Data

Dimensional data for the rapidly equilibrated panels were taken at 7 day intervals for a total of 42 days. Panels exposed to ambient conditions were inspected at 7 day intervals for 84 days. Dimensional changes after equilibration for the rapidly equilibrated panels were all less than 125 ppm (Figures 6-9) with the exception of the Y direction of Figure 6 ($[0, \pm 15, 0]_S$ lay-up, 50% RH environment). Comparisons across equilibration schemes indicated the dimensional changes for the rapidly equilibrated panels were up to 4 times less than those of panels exposed to ambient conditions, as expected. For example, the panel RNB⁸ (rapid equilibration, 50% RH, $[0, \pm 15, 0]_S$ lay-up) experienced a maximum dimensional change in the X direction of 25 $\mu\epsilon$ ($\Delta\text{length}/\text{length} \times 10^{-6}$) and 326 $\mu\epsilon$ in the Y direction over the course of the inspections. Panel ANB (ambient equilibration, 50% RH, $[0, \pm 15, 0]_S$ lay-up) experienced dimensional changes of 72 $\mu\epsilon$ and 963 $\mu\epsilon$ in the same directions (Figure 6). Similar behavior was shown across all lay-ups and FUE's (see Figures 7-9). The shrinking of the Y dimension of Figure 9 was consistent

⁸ See Appendix C, Table 2 for explanation of panel code.

with predictions. The maximum change in strain from the initial state in the Y axis (unconditioned) of Figure 6 was the result of absorbing less than 50% of the equilibrium amount of moisture.

Experimental results correlated reasonably well with theoretical predictions as depicted in Figures 10, 11 and 23-29 (Appendix C). The fiber dominated direction of each laminate exhibited better correlation, while the matrix direction exhibited poorer correlation. This was due to the type of analysis performed to obtain the theoretical predictions: modified laminated plate theory was used and with it an assumption of linear-elastic behavior, where a visco-elastic model was probably more valid (see Discussion).

As expected, the transverse direction (Y axis) of the $[0, \pm 15, 0]_S$ lay-up was most sensitive to moisture induced dimensional changes (Figure 11). The response in the transverse direction was dictated by the hygroscopic behavior of the matrix, while the longitudinal direction (X axis) was controlled by the insensitive hygroscopic response of the fibers. This same effect was evident in the $[0, \pm 45, 90]_S$ lay-up but to a lesser degree owing to the lack of directionality of the expansion properties in the pseudo-isotropic laminate (Figure 10).

Every effort was made to limit the time each panel spent outside the environmental chamber. As the weighing and inspection facilities were not in the same location, the panels spent an average of 90 minutes outside the environmental chamber from the time they were removed, weighed, inspected and then returned. Of that time, the panels spent an average of 10 minutes outside of the plastic storage/transportation bags while being weighed and inspected. The 90 minute weighing/inspection duration was unavoidable and had an effect on the data. While the time out of the environmental chamber averaged 90 minutes in length, it was not constant. This inconsistency may partially explain the varying dimensional change response depicted in Figures 6-9. As expected, the matrix dominated direction of each laminate was most sensitive to the variation in time spent out of the environmental chamber.

Inspection of a reference dimension occurred at the beginning of each inspection session. The variability in this dimension, unaffected by moisture, served as a measure of the repeatability of the CMM. Corrected for temperature, the CMM repeatability was calculated to be 12.5 ppm (i.e., a 0.0025 mm (0.0001 in) change over a 203 mm (8") gage length).

Coupon Data

The coupons cut from each panel were stored in a vacuum oven operating at 82°C (180°F) and periodically weighed to generate weight vs. time data. Figure 12 depicts the average calculated moisture content vs. time response for the coupons (graphs of each coupon can be found in Appendix C). Subsequent processing of this data indicated initial moisture contents ranging from 0.06 to 0.10%. These are in good agreement with reported values [19].

Due to the high resolution of the balance used in weighing the coupons (0.00001 g), moisture sorption during the weighing process was discernible. Nominally the mass of individual coupons changed by 0.1 - 0.2 mg over the course of the 90 second weighing (corresponding to 0.0015% moisture content). This systematic error was minimized by employing consistent weighing procedures, i.e., taking a reading at exactly 90 seconds after placing the sample on the balance and weighing the coupons in the same sequence each time. Every effort was made to limit the time each coupon spent out of the vacuum oven. This out time averaged 20 minutes.

Correcting for temperature, humidity, and barometric pressure, following NIST standard operating procedures, the mass measurements for the coupons and panels had a standard deviation of no more than 0.1 mg.

Figure 6. Dimensional Change Comparison for $[0, \pm 15, 0]$, Lay-up in 50% RH (Exposure Time for Rapid Panels Began After Equilibration, t_0+46 days)

Figure 7. Dimensional Change Comparison for $[0, \pm 45, 90]$, Lay-up in 50% RH (Exposure Time for Rapid Panels Began After Equilibration, t_0+46 days)

Figure 8. Dimensional Change Comparison for $[0, \pm 45, 90]_s$ Lay-up in 0% RH (Exposure Time for Rapid Panels Began After Equilibration, t_0+46 days)

Figure 9. Dimensional Change Comparison for $[0, \pm 15, 0]_s$ Lay-up in 0% RH (Exposure Time for Rapid Panels Began After Equilibration, t_0+46 days)

Figure 10. Experimental and Theoretical Dimensional Change for $[0, \pm 45, 90]_s$ Lay-up Exposed to 50% RH at 21°C (70°F) (ANA)

Figure 11. Experimental and Theoretical Dimensional Change for $[0, \pm 15, 0]_s$ Lay-up Exposed to 50% RH at 21°C (70°F) (ANB)

Figure 12. Average Coupon Moisture Content

Discussion

The experimental results demonstrated that the rapid equilibration scheme was capable of quickly preparing Gr/Ep composites for use in precision applications under constant temperature and humidity (46 versus 1000+ days (theoretical)). The results also showed that moisture induced dimensional changes for common structural elements were greater than the accepted definition of precision (>125 ppm).

A common structural element in which the application of this conditioning scheme would be useful is a honeycomb panel consisting of two thin face sheets. By neglecting to obtain an equilibrium moisture content in the face sheets prior to bonding to the honeycomb, the subsequent non-uniform moisture distribution through the face sheets, due to non-uniform moisture boundary conditions, could cause both in-plane dimensional changes and curvatures. These dimensional changes should result in face sheet/honeycomb de-bonding, and both unacceptable surface flatness and in-plane dimensional tolerances. The use of the rapid conditioning scheme would properly distribute the moisture through the face sheets prior to bonding to the core and would do so in a much shorter time as compared to the ambient temperature process. The use of room-temperature cure adhesives in these structures precludes the use of a high temperature cure cycle which would tend to drive off the moisture purposely put into the face sheets and would require re-equilibration of the face sheets.

The use of an elevated temperature conditioning process raises the issue of the level of residual stress in the part after conditioning. As mentioned earlier, the addition of moisture reduces the residual stresses in the laminate which are the result of fiber/matrix CTE differences and cooling from the cure temperature. Crossman et al [11] have stated that the reduction of these stresses by the absorption of a given amount of moisture is reduced less and less as the exposure temperature is increased. These findings suggest that in the 50% RH (nominal) FUE, the residual stresses in the rapidly equilibrated panels were reduced less by the addition of an equilibrium amount of moisture than the stresses in the

ambient (unconditioned) panels due to the elevated exposure temperature (82°C vs. 21°C). This further implies that the rapidly equilibrated panels are more susceptible to subsequent creep (dimensional change) due to the higher residual stress state than the unconditioned panels. An analogous situation occurs for the 0% RH (space) FUE. The increase in residual stress due to moisture desorption is lessened by the elevated exposure temperature. This implies that the panels exposed to ambient temperature are more susceptible to creep than the panels exposed to elevated temperature.

Many different authors have recognized that the outer plies control the rate of change of the laminate strains (dimensions) [24, 25]. More important to dimensional stability is designing the lay-up to reduce residual stresses by limiting the differences between transformed hygrothermal properties in adjacent plies. These authors state that it is these stresses that cause delamination and matrix micro-cracking which in turn cause permanent and unpredictable hygrothermal deformations. Wolff [24] showed evidence that lay-ups which minimize the difference in adjacent ply angle required the largest change in moisture content to be on the verge of matrix micro cracking.

In the 50% RH environment, both rapid equilibration panels exhibit larger dimensional changes than predicted by analysis (see Figures 25 and 26), possibly due to a higher moisture content. Experimental results indicate that both panels RNA and RNB (see Table 2, Appendix C for panel code) have exceeded the predicted maximum moisture content by up to 18% (see Figures 30 and 31). Wolff [24] suggests that a post cure increases the maximum moisture content, M_m implying that the elevated temperature (82°C, (180°F)) equilibration scheme acts as a post cure.

In all rapidly equilibrated panels, the departure of experimental results from analytical predictions is more significant in the transverse direction (Y axis), implying that the assumption of linear elastic materials is partially to blame. In the $[0, \pm 15, 0]_3$ laminates it is clear that the transverse direction is less likely to behave in a linear elastic manner due to the matrix dominated material properties in that direction. Even in the quasi-isotropic

$[0, \pm 45, 90]_s$ laminates, the outer 0° ply, which controls the rate of change in laminate strains, is matrix dominated in the Y laminate direction. It is unclear if the departure is solely due to visco-elastic response, however, due to an inability to quantify changes in strain from reported relaxation modulus data. Using relaxation data from [25], over a time duration of 40 days and at 20°C (70°F), the relaxation modulus ($E_r = \sigma(t)/\epsilon(t)$) drops by approximately 14% for a 1% moisture content. In an unconstrained panel, this decrease is taken up in both internal stress relaxation and increased strains (creep). Therefore nothing can be said about the change in strains, only the ratio. Over 1 year's time, this ratio drops by 19%.

The large discrepancy between experimental and analytical dimensional changes in the Y axis for the unconditioned panels also may be due to the inappropriateness of a linear elastic material model in predicting visco-elastic response (Figures 10 and 11). Flagg and Crossman [25] investigated the appropriateness of linear-elastic or visco-elastic material behavior for hygrothermal exposure of composites and found significantly better experimental/theoretical correlation of dimensional and residual stress behavior employing a visco-elastic model. In the case of panel ANB (Figure 11) the large over prediction of laminate response would be lessened by a visco-elastic treatment, which allows for residual stress relaxation and subsequent recovery of a portion of the moisture induced elastic strain from the previous time step [25]. Whether a visco-elastic treatment would reduce the theoretical/experimental difference to within experimental error is uncertain. In defense of the linear elastic treatment, the original purpose of the analysis was to rank candidate lay-ups in terms of the magnitude of hygrothermal distortions. A linear elastic treatment was easy to code and therefore used. Either treatment would have yielded the same relative laminate ranking.

The dependency of stiffness properties, Q_{ij} , on moisture content was not taken into account in the theoretical analysis. It is also unknown if the large

theoretical/experimental difference would be significantly reduced by incorporating this dependency in the theoretical predictions.

In the data to date, no two-phase diffusion behavior has been exhibited as reported by Blikstad. Weighing and inspections over the next several months will provide data to corroborate or contradict that finding.

The anomalous data point in Figure 23 is partially due to foreign matter on the reference surfaces. The CMM calculates the diameter of each reference surface (sphere) and in this case, the diameter differed by approximately 0.051 mm (0.002 in) from those taken during inspections before and after the one in question. The anomalous results in Figures 25 and 27 cannot be explained in the same way and are not due to CMM resolution errors. The errors are most likely due to the variability in the time each panel spent outside the environmental chamber, the amount of moisture sorbed during this time and the concurrent dimensional change. In both cases, the time spent out of the environmental chamber while taking the measurements was significantly different, by up to 80%, as compared to the time spent taking the data on either side of the point in question.

Conclusions

The data indicates that the two primary goals of the research were met: (1) An elevated temperature moisture equilibration process was shown to shorten the equilibration time of a set of Gr/Ep composite panels to roughly 46 days, as compared to 1000+ days at ambient temperature (calculated); (2) The resultant parts from this process were shown to be dimensionally stable, i.e., have dimensional changes less than 125 ppm. A honeycomb sandwich panel was identified as a common structural element to which this type of scheme would be useful.

Dimensional change data for the ambient conditioned panels demonstrated the need for an equilibration scheme like the one used herein for precision applications employing Gr/Ep composites. The data showed that significant dimensional changes occurred in a short time after removal from the autoclave, due to absorption of less than 50% of the equilibrium amount of moisture. Dimensional changes up to 963 ppm were measured for the highly orthotropic $[0, \pm 15, 0]_S$ laminate under a 21°C, 50% RH environment. Under the same conditions, the pseudo-isotropic laminate $[0, \pm 45, 90]_S$ grew by 514 ppm.

Correlation between experimental and theoretical dimensional change data was exceptional in the X axis and poorer in the Y axis. It was hypothesized that the degree of correlation was related to the appropriateness of the assumption of linear elastic material behavior in the fibers and matrix as well as an under prediction of the equilibrium moisture content. While not able to quantify, it was concluded that over a years time or longer at 20°C (70°F), visco-elastic response of the rapidly equilibrated panels will contribute to dimensional changes.

The Y axis of both lay-ups was most sensitive to moisture induced dimensional changes, owing to the hygroscopic and visco-elastic nature of the matrix.

Out-of-autoclave moisture content data agreed with published values. Initial moisture content ranged between 0.06% and 0.1%, by dry panel weight.

No two-phase diffusion had been noted. Subsequent monitoring will provide data to corroborate or contradict this reported finding.

The few anomalous inspection readings were the result of reference surface contamination in one case and were most likely due to the variability in the time each panel spent outside of its environmental chamber in the other cases. The anomalous data could not be explained by errors in the CMM alone, whose repeatability error was calculated to be 12.5 ppm.

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Appendix A - Relevant Equation Derivations

This section details the derivations of the equations used to choose the lay-ups for the experimentation. These equations were used to predict mid-plane strain and curvature behavior of the test panels as a function of time and moisture distribution.

Laminated plate theory is the basis for determining the stress/strain behavior of a laminated composite panel. Incorporating hygrothermal strains is analogous to the treatment of mechanical strains. The definitions of thermal and moisture strains are as follows:

$$\epsilon^T = \alpha \Delta T \quad (6)$$

$$\epsilon^M = \beta \Delta M \quad (7)$$

where α = coefficient of thermal expansion $\left(\frac{\Delta l}{l \cdot \text{degree}} \right)$

β = coefficient of moisture expansion $\left(\frac{\Delta l}{l \cdot \% \text{moisture}} \right)$

ΔT = difference in temperature (degrees)

ΔM = difference in moisture content (% moisture)

Considering a single lamina of the panel, the stress/strain relations can be described using Hooke's law, which relates stress to strain through elastic constants. In principal material directions (1,2) Hooke's law is written as follows (Jones, [28]):

$$\begin{Bmatrix} \sigma_1 \\ \sigma_2 \\ \tau_{12} \end{Bmatrix} = \begin{bmatrix} Q_{11} & Q_{12} & 0 \\ Q_{12} & Q_{22} & 0 \\ 0 & 0 & Q_{66} \end{bmatrix} \begin{Bmatrix} \epsilon_1 \\ \epsilon_2 \\ \gamma_{12} \end{Bmatrix} \quad (8)$$

A standard transformation matrix (equation (9)) allows the stresses in arbitrary axes (X, Y, defined by the angle θ , see Figure 13) to be expressed in terms of strains in the same axes and transformed elastic constants $\bar{Q}_{ij}$.

Figure 13. Laminate (X, Y) and Material (1, 2) Axes Definitions and Positive Angle Rotation

$$[T] = \begin{bmatrix} \cos^2 \theta & \sin^2 \theta & 2 \sin \theta \cos \theta \\ \sin^2 \theta & \cos^2 \theta & -2 \sin \theta \cos \theta \\ -\sin \theta \cos \theta & \sin \theta \cos \theta & \cos^2 \theta - \sin^2 \theta \end{bmatrix} \quad (9)$$

$$\begin{Bmatrix} \sigma_x \\ \sigma_y \\ \tau_{xy} \end{Bmatrix} = \begin{bmatrix} \bar{Q}_{11} & \bar{Q}_{12} & \bar{Q}_{16} \\ \bar{Q}_{12} & \bar{Q}_{22} & \bar{Q}_{26} \\ \bar{Q}_{16} & \bar{Q}_{26} & \bar{Q}_{66} \end{bmatrix} \begin{Bmatrix} \epsilon_x \\ \epsilon_y \\ \gamma_{xy} \end{Bmatrix} \quad (10)$$

Returning to the hygrothermal strains, these too can be expressed in arbitrary axes by transforming the material properties⁹, $\underline{\alpha}$ and $\underline{\beta}$, to the arbitrary axes by means of the inverse transformation matrix T^{-1} .

⁹Bold typeface or square brackets, [], indicates matrix quantities, while underlining or curved brackets, { } denotes vector quantities

$$\begin{Bmatrix} \alpha_x \\ \alpha_y \\ \frac{1}{2}\alpha_{xy} \end{Bmatrix} = [T]^{-1} \begin{Bmatrix} \alpha_1 \\ \alpha_2 \\ 0 \end{Bmatrix} \quad (11)$$

$$\begin{Bmatrix} \beta_x \\ \beta_y \\ \frac{1}{2}\beta_{xy} \end{Bmatrix} = [T]^{-1} \begin{Bmatrix} \beta_1 \\ \beta_2 \\ 0 \end{Bmatrix} \quad (12)$$

The (1/2) coefficient in front of the α_{xy} and β_{xy} terms is the result of using the same transformation equation T^{-1} to transform stresses and coefficients of thermal and moisture expansion from material to laminate axes. Since CTE and CME transform like strains, to use the above transformation matrix requires tensor values, i.e., one half the engineering values, hence the (1/2) coefficient. The transformed hygrothermal strains can be written directly in terms of transformed properties.

$$\begin{Bmatrix} \varepsilon_x^T \\ \varepsilon_y^T \\ \gamma_{xy}^T \end{Bmatrix} = \begin{Bmatrix} \alpha_x \Delta T \\ \alpha_y \Delta T \\ \alpha_{xy} \Delta T \end{Bmatrix} \quad (13)$$

$$\begin{Bmatrix} \varepsilon_x^M \\ \varepsilon_y^M \\ \gamma_{xy}^M \end{Bmatrix} = \begin{Bmatrix} \beta_x \Delta M \\ \beta_y \Delta M \\ \beta_{xy} \Delta M \end{Bmatrix} \quad (14)$$

Using assumptions of thin plate theory [27] and analyzing the displacements of the geometric mid-plane of the whole laminate [28], the strain field can be described in terms of mid-plane strains $\underline{\varepsilon}^0$ and curvatures $\underline{\kappa}$.

$$\begin{Bmatrix} \epsilon_x \\ \epsilon_y \\ \gamma_{xy} \end{Bmatrix} = \begin{Bmatrix} \epsilon_x^o \\ \epsilon_y^o \\ \gamma_{xy}^o \end{Bmatrix} + z \begin{Bmatrix} \kappa_x \\ \kappa_y \\ \kappa_{xy} \end{Bmatrix} \quad (15)$$

The total strain state in a lamina is the summation of mechanical and non-mechanical (hygrothermal) components and can be described in terms of mid plane strains and curvatures as in equation (15).

$$\begin{Bmatrix} \epsilon_x \\ \epsilon_y \\ \gamma_{xy} \end{Bmatrix}^{Tot} = \begin{Bmatrix} \epsilon_x \\ \epsilon_y \\ \gamma_{xy} \end{Bmatrix}^{Mech} + \begin{Bmatrix} \epsilon_x \\ \epsilon_y \\ \gamma_{xy} \end{Bmatrix}^{Therm} + \begin{Bmatrix} \epsilon_x \\ \epsilon_y \\ \gamma_{xy} \end{Bmatrix}^{Moist} = \begin{Bmatrix} \epsilon_x^o \\ \epsilon_y^o \\ \gamma_{xy}^o \end{Bmatrix} + z \begin{Bmatrix} \kappa_x \\ \kappa_y \\ \kappa_{xy} \end{Bmatrix} \quad (16)$$

The total stress state can be evaluated by substituting equation (16) into equation (10).

$$\begin{Bmatrix} \sigma_x \\ \sigma_y \\ \tau_{xy} \end{Bmatrix}^{Total} = [\bar{Q}] \left[\{\epsilon\}^{Mech} + \{\epsilon\}^{Therm} + \{\epsilon\}^{Moist} \right] = [\bar{Q}] \left[\begin{Bmatrix} \epsilon_x^o \\ \epsilon_y^o \\ \gamma_{xy}^o \end{Bmatrix} + z \begin{Bmatrix} \kappa_x \\ \kappa_y \\ \kappa_{xy} \end{Bmatrix} \right] \quad (17)$$

As the stresses vary across the laminate, it is more convenient to express loading (mechanical and non-mechanical) on the laminate in terms of resultant forces and moments acting on the cross section. The resultant forces and moments are defined below and are represented in Figure 14 (where h = thickness of laminate).

$$N_x = \int_{-h/2}^{h/2} \sigma_x dz \quad (18a)$$

$$M_x = \int_{-h/2}^{h/2} \sigma_x z dz \quad (19a)$$

$$N_y = \int_{-h/2}^{h/2} \sigma_y dz \quad (18b)$$

$$M_y = \int_{-h/2}^{h/2} \sigma_y z dz \quad (19b)$$

$$N_x = \int_{-h/2}^{h/2} \tau_{xy} dz \quad (18c)$$

$$M_x = \int_{-h/2}^{h/2} \tau_{xy} z dz \quad (19c)$$

Figure 14. Definition of Resultant Forces and Moments

The above equations can be adapted to a multi-layered composite by integrating across the thickness of each lamina and summing over the total number of lamina.

$$\begin{Bmatrix} N_x \\ N_y \\ N_{xy} \end{Bmatrix} = \sum_{i=1}^{\# \text{lamina}} \int_{h_{i-1}}^{h_i} \begin{Bmatrix} \sigma_x \\ \sigma_y \\ \tau_{xy} \end{Bmatrix}_i dz \quad (20)$$

$$\begin{Bmatrix} M_x \\ M_y \\ M_{xy} \end{Bmatrix} = \sum_{i=1}^{\#lamina} \int_{h_{i-1}}^{h_i} \begin{Bmatrix} \sigma_x \\ \sigma_y \\ \tau_{xy} \end{Bmatrix} z dz \quad (21)$$

Substituting the expression for lamina stress (equation (17)) we have the following:

$$\begin{Bmatrix} N_x \\ N_y \\ N_{xy} \end{Bmatrix} = \sum_{i=1}^{\#lamina} \int_{h_{i-1}}^{h_i} \left([\bar{Q}] \left[\{\epsilon\}^{Mech} + \{\epsilon\}^{Therm} + \{\epsilon\}^{Moist} \right] \right) dz \quad (22)$$

$$\begin{Bmatrix} M_x \\ M_y \\ M_{xy} \end{Bmatrix} = \sum_{i=1}^{\#lamina} \int_{h_{i-1}}^{h_i} \left([\bar{Q}] \left[\{\epsilon\}^{Mech} + \{\epsilon\}^{Therm} + \{\epsilon\}^{Moist} \right] \right) z dz \quad (23)$$

The laminate stress state can also be described in terms of mid-plane strains and curvatures as shown in equation (17).

$$\{N\} = \sum_{i=1}^{\#lamina} \int_{h_{i-1}}^{h_i} [\bar{Q}] \left[\{\epsilon^o\} + z\{\kappa\} \right] dz \quad (24)$$

$$\{M\} = \sum_{i=1}^{\#lamina} \int_{h_{i-1}}^{h_i} [\bar{Q}] \left[\{\epsilon^o\} + z\{\kappa\} \right] z dz \quad (25)$$

By defining the following matrices, subsequent notation simplifies.

$$[A] = \sum_{i=1}^{\#lamina} [\bar{Q}_{ij}] (h_i - h_{i-1}) \quad (26)$$

$$[B] = 1/2 \sum_{i=1}^{\#lamina} [\bar{Q}_{ij}] (h_i^2 - h_{i-1}^2) \quad (27)$$

$$[D] = 1/3 \sum_{i=1}^{\#lamina} [\bar{Q}_{ij}] (h_i^3 - h_{i-1}^3) \quad (28)$$

Integrating equations (24) and (25), employing the definitions in equations (26)-(28) and equating to equations (22) and (23) we rewrite as follows:

$$[A]\{\epsilon^o\} + [B]\{\kappa\} = \sum_{i=1}^{n_{lamina}} \int_{h_{i-1}}^{h_i} \left([\bar{Q}] [\{\epsilon\}^{Mech} + \{\epsilon\}^{Therm} + \{\epsilon\}^{Moist}] \right) dz \quad (29)$$

$$[B]\{\epsilon^o\} + [D]\{\kappa\} = \sum_{i=1}^{n_{lamina}} \int_{h_{i-1}}^{h_i} \left([\bar{Q}] [\{\epsilon\}^{Mech} + \{\epsilon\}^{Therm} + \{\epsilon\}^{Moist}] \right) z dz \quad (30)$$

For the laminates and environmental conditions under consideration, the mechanical strains are equal to zero and equations (29) and (30) reduce to the following:

$$[A]\{\epsilon^o\} + [B]\{\kappa\} = \sum_{i=1}^{n_{lamina}} \int_{h_{i-1}}^{h_i} \left([\bar{Q}] [\{\epsilon\}^{Therm} + \{\epsilon\}^{Moist}] \right) dz \quad (31)$$

$$[B]\{\epsilon^o\} + [D]\{\kappa\} = \sum_{i=1}^{n_{lamina}} \int_{h_{i-1}}^{h_i} \left([\bar{Q}] [\{\epsilon\}^{Therm} + \{\epsilon\}^{Moist}] \right) z dz \quad (32)$$

Recognizing that $[\bar{Q}]$, ΔT and ΔM (equations (13) and (14)) are all independent of z and can be brought outside the integral sign, then integrating, we have the following:

$$[A]\{\epsilon^o\} + [B]\{\kappa\} = \sum_{i=1}^{n_{lamina}} [\bar{Q}_i] (h_i - h_{i-1}) [\{\alpha\} \Delta T + \{\beta\} \Delta M] \quad (33)$$

$$[B]\{\epsilon^o\} + [D]\{\kappa\} = 1/2 \sum_{i=1}^{n_{lamina}} [\bar{Q}_i] (h_i^2 - h_{i-1}^2) [\{\alpha\} \Delta T + \{\beta\} \Delta M] \quad (34)$$

The terms on the right hand side of each equation are known for each lamina and are functions of environmental conditions.

Rewriting equations (33) and (34) in matrix form, the following results are obtained:

$$\begin{bmatrix} A & B \\ B & D \end{bmatrix} \begin{Bmatrix} \epsilon^o \\ \kappa \end{Bmatrix} = \begin{Bmatrix} N^{NM} \\ M^{NM} \end{Bmatrix} \quad (35)$$

Solving for the hygrothermal (thermal and moisture) mid-plane strains and curvatures, we have the results below:

$$\begin{Bmatrix} \epsilon^o \\ \kappa \end{Bmatrix} = \begin{bmatrix} A' & B' \\ B' & D' \end{bmatrix} \begin{Bmatrix} N^{NM} \\ M^{NM} \end{Bmatrix} \quad (36)$$

where the prime matrices A' , B' and D' are defined as the following [22]

$$[A'] = [A^*] - [B^*][D^*]^{-1}[C^*] \quad (37)$$

$$[B'] = [B^*][D^*]^{-1} \quad (38)$$

$$[D'] = [D^*]^{-1} \quad (39)$$

and

$$[A^*] = [A]^{-1} \quad (40)$$

$$[B^*] = -[A]^{-1}[B] \quad (41)$$

$$[C^*] = -[B^*]^T \quad (42)$$

$$[D^*] = [D] - [B][A]^{-1}[B] \quad (43)$$

Equation (36) and all supporting equations were used in a spreadsheet to predict the hygrothermal response of the test specimens.

Appendix B - Determination of Space Moisture Environment

One issue of moderate uncertainty was the level of relative saturation to be used in laboratory conditions to simulate the conditions of low earth orbit (LEO) which, for the purpose of argument, is nominally 125 Km (78 mi) geometric altitude. At issue was the method by which equilibrium between a condensed (absorbed) phase and a surrounding vapor phase is established. A "gut" feeling was that the LEO environment is dry and that the appropriate 101 KPa (1 atmosphere) laboratory simulation of this would be 0 % RH. The following analysis determines the appropriate saturation level from thermodynamic considerations

Denbigh states that by definition, a condensed (absorbed) phase of component i of a solution is in equilibrium with its surrounding vapor phase when the chemical potential (μ) of component i in solution is equal to the chemical potential of component i in the vapor phase for any component that can pass freely between the phases. By the above definition, it can be written

$$\mu_i^{soln} = \mu_i^{vapor} \quad (44)$$

If the temperatures are such that the vapor pressures are sufficiently small, then the vapor can be assumed to behave as a perfect gas mixture¹⁰. If a gas mixture is perfect, the chemical potential of each component can be written as follows:

$$\mu_i = \mu_i^0 + RT \ln p_i \quad (45)$$

where: μ_i^0 = Gibbs free energy per mole of pure component i at unit pressure and same temperature as mixture under consideration = $f(T)$

R = Universal gas constant

T = Absolute temperature

¹⁰ A perfect gas mixture has the following traits Denbigh [29]: (1) the gas mixture as a whole obeys the equation of state, i.e., $pV = nRT$ or $p_i = n_iRT/V$; (2) there is no heat of mixing; (3) two mixtures are at equilibrium through a semi-permeable membrane when the chemical potential of each component on both sides of the membrane are equal.

p_i = partial pressure of component i

The chemical potential of the i^{th} component in the vapor can be written as:

$$\mu_i^{\text{vapor}} = \mu_i^{\circ} + RT \ln p_i \quad (46)$$

then by equating,

$$\mu_i^{\text{soln}} = \mu_i^{\circ} + RT \ln p_i \quad (47)$$

The last equation gives the chemical potential of the i^{th} component in the solution in terms of its partial pressure. By inspection, the partial pressure of the i^{th} component in solution must equal the partial vapor pressure of the i^{th} component in the vapor for equilibrium to exist

The next issue is to relate the composition of the solution to the partial pressure of each component in the vapor at equilibrium conditions. Henry's law relates the partial pressures of the components in the vapor phase to the composition of those components in the condensed phase, at equilibrium (i.e., $p_i(\text{soln}) = p_i(\text{vapor})$).

$$p_i = K_i \chi_i \quad (48)$$

where: K_i = proportionality constant of component i , $f(T, p)$

χ_i = mole fraction of component i in solution

p_i = partial pressure of component i

We now have a measure of the composition of the condensed phase (through mole fractions) in terms of the partial pressures of each component in the vapor phase at equilibrium conditions. Denoting the equilibrium measure of condensed water vapor in the polymer matrix composite as M_m , dividing by K_i and expressing the temperature dependence of the relationship between concentration and partial pressure through an Arrhenius relationship, we can write:

$$M_m = S_0 p \exp\left(\frac{-\Delta H}{RT}\right) \quad (49)$$

where: S_0 = experimentally determined proportionality
coefficient, $F(T)$

p = water vapor partial pressure

ΔH = heat of solution

The value S_0 can be determined from tests of equilibrium moisture content vs. water vapor partial pressure at varying temperatures. For the matrix material system under study (Fiberite 934), M_m was found to be experimentally insensitive to temperature across a range of 25°C - 89°C (77°F - 192°F) [16]. This leads to the conclusion that the equilibrium moisture concentration is directly related to the water vapor partial pressure of the surrounding environment.

In a more succinct way, others have stated that moisture contained in a wet solid exerts a vapor pressure and that the solid, exposed to a continuous supply of gas containing a partial pressure of water vapor equal to p_i , will lose/gain moisture until the vapor pressure of the moisture in the solid equals p_i . This moisture level is called the equilibrium moisture content at prevailing conditions. Therefore, knowing the partial pressure of water vapor at altitude would give the necessary information in determining the correct saturation level to use during the experimentation.

This piece of information is derived from MIL STD 1809 [30] which gives the number density of the principal species (Ar, O, O₂, H, He, N₂) as a function of geometric altitude. The data shows a significant drop at the 100 -125 Km (63-78 mi) range from number densities on the order of 1×10^{21} to 1×10^{16} particles per cubic meter. By calculating the total pressure and partial pressures of each of the principal species, a measure of maximum water vapor partial pressure is established. Table 1 gives the number density, calculated partial pressure and mole fraction for each of the principal species. The number densities used assumed the worst case calculation values (i.e.,

minimum number densities). Atmospheric conditions that can cause significant variation in the number densities, such as diurnal cycle, altitude and latitude, magnetic storms and season, were assumed to be non-influential.

Table 1. Principal Species Density, Partial Pressure and Mole Fraction at 125 Km (78 mi) Geometric Altitude

Species	Number Density (#/m ³)	Partial Pressure (N/m ²)	Mole Fraction $\chi_i = P_i/P$
Ar	8×10^{14}	3.3×10^{-6}	1.9×10^{-3}
H	1×10^{12}	4.0×10^{-9}	2.4×10^{-6}
O ₂	3×10^{16}	1.2×10^{-4}	7.1×10^{-2}
He	5×10^{13}	2.1×10^{-7}	1.2×10^{-4}
N ₂	1×10^{17}	4.1×10^{-4}	2.4×10^{-1}
O	1×10^{17}	4.1×10^{-4}	2.4×10^{-1}

$$\Sigma P_i = 9.4 \times 10^{-4}$$

$$\Sigma \chi_i = 0.56$$

The partial pressure of component i in terms of the number density and assuming ideal gas mixture behavior is given by:

$$P_i = \frac{n_i RT}{V} = \frac{N_i RT}{N_0 V} = \frac{N_i}{V} \frac{RT}{N_0} \quad (50)$$

where N_i/V = number density (particles/volume) of
component i

R = universal gas constant

T = absolute temperature

N₀ = Avagadro's number (6.02×10^{26}
molecules/kg-mole)

P_i = partial pressure of component i

The total pressure is calculated assuming perfect gas mixture behavior, that the composition of the mixture at 125 Km (78 mi) can be approximated as air and given the density of the mixture at altitude ($2 \times 10^{-8} \text{ kg/m}^3$).

$$P = \rho RT = \left(2 \times 10^{-8} \frac{\text{kg}}{\text{m}^3} \right) \left(287 \frac{\text{N} \cdot \text{m}}{\text{kg} \cdot \text{K}} \right) (294 \text{ K}) = 1.7 \times 10^{-3} \frac{\text{N}}{\text{m}^2} \quad (51)$$

where R = gas constant for air

If we assume that the remaining mole fraction ($1 - 0.56 = 0.44$) is made up of water vapor only (worst case assumption), then we have an estimate for the maximum partial pressure of water vapor. This calculates to a value of $7.5 \times 10^{-4} \text{ Pa}$ ($1.1 \times 10^{-7} \text{ psi}$).

Using experimental data that suggested M_m was independent of temperature, Springer rewrote equation (49) in the form:

$$M_m = a(\phi)^b \quad (52)$$

where:

a, b = experimentally determined fitting

coefficients (a=0.017, b=1)

ϕ = relative humidity ($p_{\text{vapor}}/p_{\text{sat}}$)

Using the form of the above equation, the last piece of information required to determine the LEO space environment we are trying to simulate is the saturation pressure at 21°C (70°F). From a Mollier diagram, the saturation pressure at 21°C (70°F) is approximately 3033 Pa (0.44 psi). This implies the equilibrium moisture content we should be trying to achieve to simulate space is roughly $4 \times 10^{-9} \%$ by dry weight of the composite test piece. This amount of moisture is undetectable by any balance, and surely undetectable by the balance chosen for the experimentation.

Appendix C - Additional Data

Table 2. Laminate Ply Thicknesses (Theoretical: 0.178 mm (0.007 in))

Panel	ANA	ANB	ASPA	ASPB	RNA	RNB	RSPA	RSPB
Ply 1 T (mm)	0.216	0.193	0.178	0.188	0.185	0.185	0.188	0.196
(in)	0.0085	0.0076	0.0070	0.0074	0.0073	0.0073	0.0074	0.0077
Ply 2 T (mm)	0.193	0.188	0.193	0.191	0.185	0.196	0.180	0.191
(in)	0.0076	0.0074	0.0076	0.0075	0.0073	0.0077	0.0071	0.0075
Ply 3 T (mm)	0.206	0.185	0.251	0.193	0.178	0.180	0.191	0.193
(in)	0.0081	0.0073	0.0099	0.0076	0.0070	0.0071	0.0075	0.0076
Ply 4 T (mm)	0.173	0.201	0.140	0.198	0.183	0.185	0.198	0.183
(in)	0.0068	0.0079	0.0055	0.0078	0.0072	0.0073	0.0078	0.0072
Ply 5 T (mm)	0.185	0.173	0.178	0.178	0.185	0.180	0.188	0.168
(in)	0.0073	0.0069	0.0070	0.0070	0.0073	0.0071	0.0074	0.0066
Ply 6 T (mm)	0.185	0.188	0.191	0.206	0.185	0.178	0.208	0.191
(in)	0.0073	0.0074	0.0075	0.0081	0.0073	0.0070	0.0082	0.0075
Ply 7 T (mm)	0.178	0.213	0.193	0.183	0.183	0.193	0.178	0.188
(in)	0.0070	0.0084	0.0076	0.0072	0.0072	0.0076	0.0070	0.0074
Ply 8 T (mm)	0.185	0.173	0.191	0.180	0.203	0.198	0.180	0.180
(in)	0.0073	0.0069	0.0075	0.0071	0.0080	0.0078	0.0071	0.0071
Average Ply Thickness:								
(mm)	0.191	0.188	0.188	0.188	0.185	0.185	0.188	0.185
(in)	0.0075	0.0074	0.0074	0.0074	0.0073	0.0073	0.0074	0.0073

Notes: Panel Code

ex: W XY Z

W: Conditioning Scheme

A = unconditioned (ambient temperature)

R = rapid conditioning (82°C (180°F))

XY: Final Use Environment

N = Nominal (50% RH)

SP = Space (0% RH)

Z: Lay-up

A = [0, ± 45, 90]_s

B = [0, ± 15, 0]_s

Table 3. Laminate Fiber Volume Results (Target Value: 57-63%)

Laminate	ANA	ANB	ASPA	ASPB	RNA	RNB	RSPA	RSPB
Fiber Volume (%)	58.2	58.6	58.2	58.7	59.3	57.9	57.7	60.4

Notes: See Panel Code in Notes to Table 2

Figure 15. Coupon Moisture Content (ANA)

Figure 16. Coupon Moisture Content (ANB)

Figure 17. Coupon Moisture Content (ASPA)

Figure 18. Coupon Moisture Content (ASPB)

Figure 19. Coupon Moisture Content (RNA)

Figure 20. Coupon Moisture Content (RNB)

Figure 21. Coupon Moisture Content (RSPA)

Figure 22. Coupon Moisture Content (RSPB)

Figure 23. Experimental and Theoretical Dimensional Change for $[0, \pm 45, 90]_s$ Lay-up Exposed to 0% RH at 21°C (70°F) (ASPA)

Figure 24. Experimental and Theoretical Dimensional Change for $[0, \pm 15, 0]_s$ Lay-up Exposed to 0% RH at 21°C (70°F) (ASPB)

Figure 25. Experimental and Theoretical Dimensional Change After Conditioning for $[0, \pm 45, 90]_s$ Lay-up Exposed to 50% RH at 82°C (180°F) (Note: The First 46 days of Exposure Were Spent in the Equilibration Chamber) (RNA)

Figure 26. Experimental and Theoretical Dimensional Change After Conditioning for $[0, \pm 15, 0]_s$ Lay-up Exposed to 50% RH at 82°C (180°F) (Note: The First 46 days of Exposure Were Spent in the Equilibration Chamber) (RNB)

Figure 27. Experimental and Theoretical Dimensional Change After Conditioning for [0, ± 45, 90]_s Lay-up Exposed to 0% RH at 82°C (180°F) (Note: The First 46 days of Exposure Were Spent in the Equilibration Chamber) (RSPA)

Figure 28. Experimental and Theoretical Dimensional Change After Conditioning for [0, ± 15, 0]_s Lay-up Exposed to 0% RH at 82°C (180°F) (Note: The First 46 Days of Exposure Were Spent in the Equilibration Chamber) (RSPB)

Figure 29. Comparison of Experimental and Theoretical Y Axis Dimensional Change for Ambient Panels Exposed to 50% RH at 21°C (70°F)

Figure 30. Moisture Content for Panel RNA

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