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Effects of Temperature and Relative Humidity on T1100/3960 Carbon Fiber/Epoxy Prepreg

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1.0 **Introduction**

1.1 Background

It is well-established that moisture absorption can lead to degradation in composite materials with epoxy matrices. The polar nature of epoxies makes this class of materials highly susceptible to moisture absorption. As epoxies take on water, their chemical structure can be fundamentally altered, which can in turn lead to plasticization and a reduction in glass transition temperature. These adverse effects of moisture absorption have been identified in the literature as primary contributors to knockdowns in mechanical properties, including strength and fracture toughness [1–10].

Though much attention has been paid to the general issue of moisture absorption in epoxy-based composites, experimental studies in this area have typically focused on moisture absorption in cured composites [6,7,11–18]. Much less information is available in the open literature on the effects of moisture absorption while the composite material is still in the prepreg state, which is significant with respect to the manufacturing process. Temperature and relative humidity (RH) – both of which directly affect moisture diffusion rates [5,8,19] – can vary widely in the layup environment, especially in large-volume high bay facilities suitable for large-scale composites manufacturing. Changes in weather and relative performance of facility heating, ventilation, and air conditioning (HVAC) systems can lead to swings in facility temperature and RH from season-to-season and even day-to-day in extreme circumstances. It is therefore critical to understand the effects of temperature and RH during layup – while the composite material is still in the prepreg state – on the performance of composite materials.

In this study, T1100/3960 carbon fiber/epoxy prepreg is evaluated with respect to a range of temperatures and RH values prior to layup and cure. A practical approach is taken to reflect realistic temperature and RH values commonly encountered in a large-volume high bay manufacturing environment. Environmentally conditioned prepreg plies are used to fabricate a series of composite laminates which are tested for physical and mechanical performance. Results are presented and discussed with the intent of establishing an improved understanding of the effects of temperature and RH in the layup environment for an increasingly common epoxy-based prepreg.

2.0 **Experimental**

2.1 Study Design

The four test groups considered in this study are summarized in Table 1. In addition to the baseline group (where no environmental conditioning was considered), three environmentally conditioned groups were considered to evaluate low (23%), medium (50%), and high (70%) RH environments. These RH values were selected in the interest of evaluating a range of conditions commonly encountered in large-volume high bay manufacturing facilities where HVAC performance relative to local weather can be challenging. A conditioning temperature of 72°F was selected for the 50% RH group in an effort to account for "nominal" conditions as typically observed in Huntsville, Alabama, though it should be recognized that nominal RH in a given facility is a function of geographic location (along with facility size, facility design, HVAC capability, etc.). Given that the high bay facility of interest is generally capable of holding 72°F ± 5°F, the high end of this range (77°F) was selected for the low (23%) RH and high (70%) RH groups in order to achieve the driest possible environment for the low RH group (which was 23% RH in the environmental chamber available for use) and the highest possible diffusion rate for the high RH group. It should be noted that while the low RH group provides value in terms of completeness of the study, low RH environments are not typically a concern for prepreg materials in the composites manufacturing process. A duration of 14 days was selected as this represents a nominal but conservative period of time where prepreg materials may be exposed during the manufacturing process.

Table 1. Summary of test groups considered in this study.

Test Group	Conditioning Parameters			Cure Method
	Temperature (°F)	Relative Humidity (%)	Duration (days)	
Baseline	NA	NA	NA	Autoclave
RH = 23%	77	23	14	Autoclave
RH = 50%	72	50	14	Autoclave
RH = 70%	77	70	14	Autoclave

2.2 Environmental Conditioning and Panel Fabrication

For the baseline group, prepreg plies were cut, laid up, and cured within a period of 24 hours in an effort to minimize ambient environmental conditioning. For the three test groups where environmental conditioning was considered, T1100/3960 carbon fiber/epoxy prepreg (plain weave; T1100G-12K; 35% nominal resin content by weight; 196 gsm) were cut and placed in an environmental chamber as shown in Figure 1. Backing paper was left on one side of each ply to provide rigidity and prevent adjacent plies from sticking to one another, leaving one side of each ply exposed to the chamber environment. Secondary temperature and relative humidity gauges were used to ensure the accuracy of the environmental chamber's settings. Prepreg plies for each group were conditioned for a period of 14 days.

Figure 1. Prepreg plies in environmental chamber for conditioning.

A single parent panel ($[(0/90)_f]_{18}$; 16 in. x 16 in.) was manufactured for each test group. Parent panels were autoclave-cured using a 2 hour hold at 355°F and 50 psi.

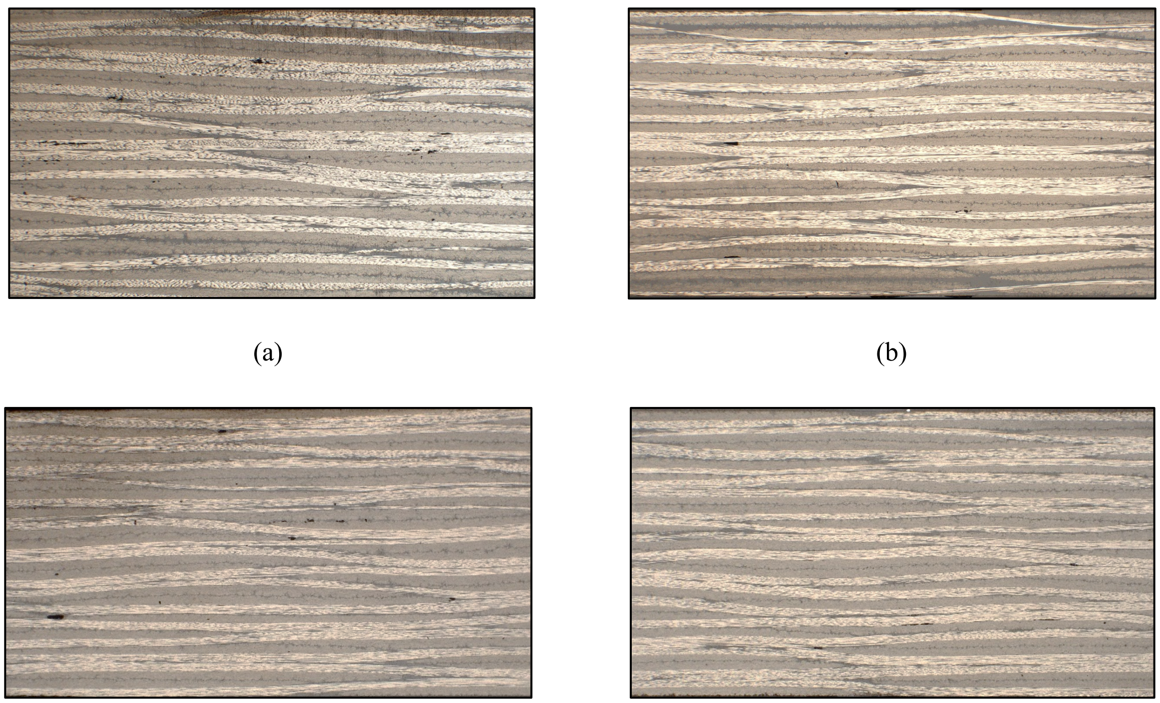
2.3 Material Characterization

In order to evaluate the effects of the previously described environmental conditioning on the T1100/3960 prepreg considered in this study, optical microscopy, mechanical testing, and dynamic mechanical analysis (DMA) was carried out for each test group. Optical microscopy and constituent content determination (via matrix burnoff per ASTM D3171, Procedure G [20]) were used to verify cured laminate quality and consistency across test groups. The open hole compression test per ASTM D6484 [21] was used to screen for strength degradation in the composite system. Note that the open hole compression test is preferred by the authors over a pristine compression test, such as the combined loading compression (CLC) test described in ASTM D6641 [22], given that the open hole compression test is much less sensitive to specimen preparation and test procedure while still providing a meaningful metric for compression strength in a composite laminate. The short beam shear test per ASTM D2344 [23] was used to screen for matrix degradation given the epoxy's aforementioned susceptibility to moisture absorption. DMA was used to screen for changes in glass transition temperature as a result of moisture absorption during environmental conditioning.

3.0 **Results and Discussion**

3.1 Optical Microscopy and Constituent Content Results

The optical micrographs included in Figure 2 show nominal laminate quality for each of the four test groups considered in this study. A minimal number of small voids are observable in each of the test groups. Beyond this, laminate quality in each of the four test groups is as-expected for an aerospace-grade carbon fiber/epoxy prepreg following autoclave cure.



(a) (b) (c) (d)

Figure 2. Optical micrographs for (a) baseline test group, (b) RH = 23% test group, (c) RH = 50% test group, and (d) RH = 70% test group.

Constituent content results are presented in detail in Table 2 and are shown graphically in Figure 3. Values are based on a minimum of 5 samples for each test group. While resin content values vary slightly among test groups, each value is within 3.5% of the nominal resin content (35%) for the T1100/3960 prepreg considered herein. These results, coupled with the qualitative comparisons carried out via optical microscopy, do not suggest any significant differences among test groups in terms of cured laminate quality.

Table 2. Constituent content results. Coefficients of variation are shown in italics.

Test Group	Resin Content by Weight (%)
Baseline	34.3 ± 0.4 *(1.2%)*
RH = 23%	35.4 ± 1.2 *(3.4%)*
RH = 50%	33.8 ± 0.3 *(0.9%)*
RH = 70%	34.4 ± 0.4 *(1.2%)*

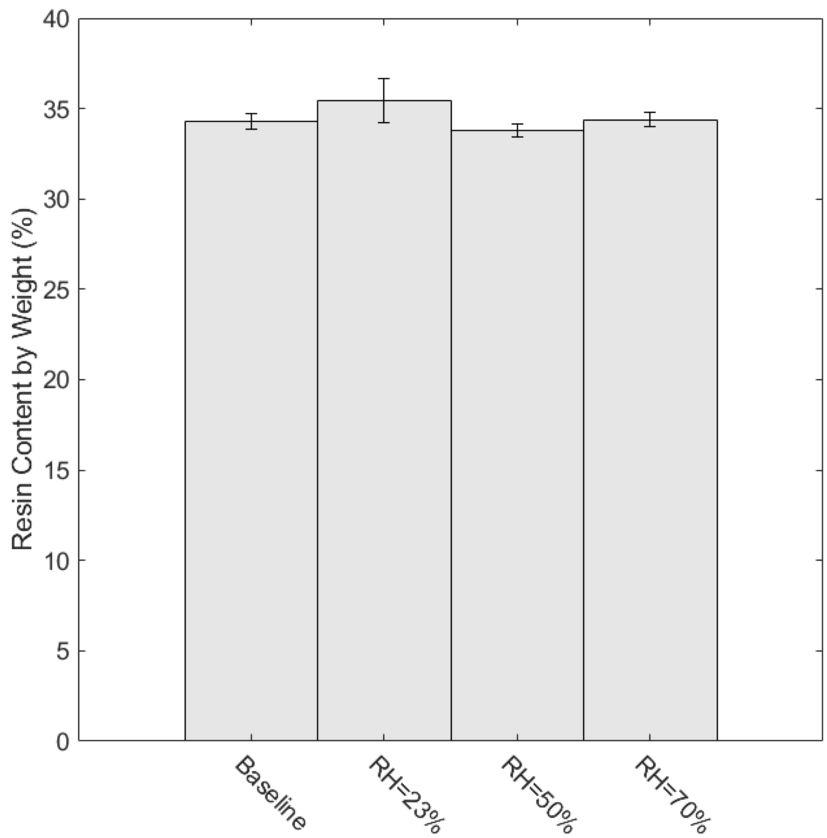


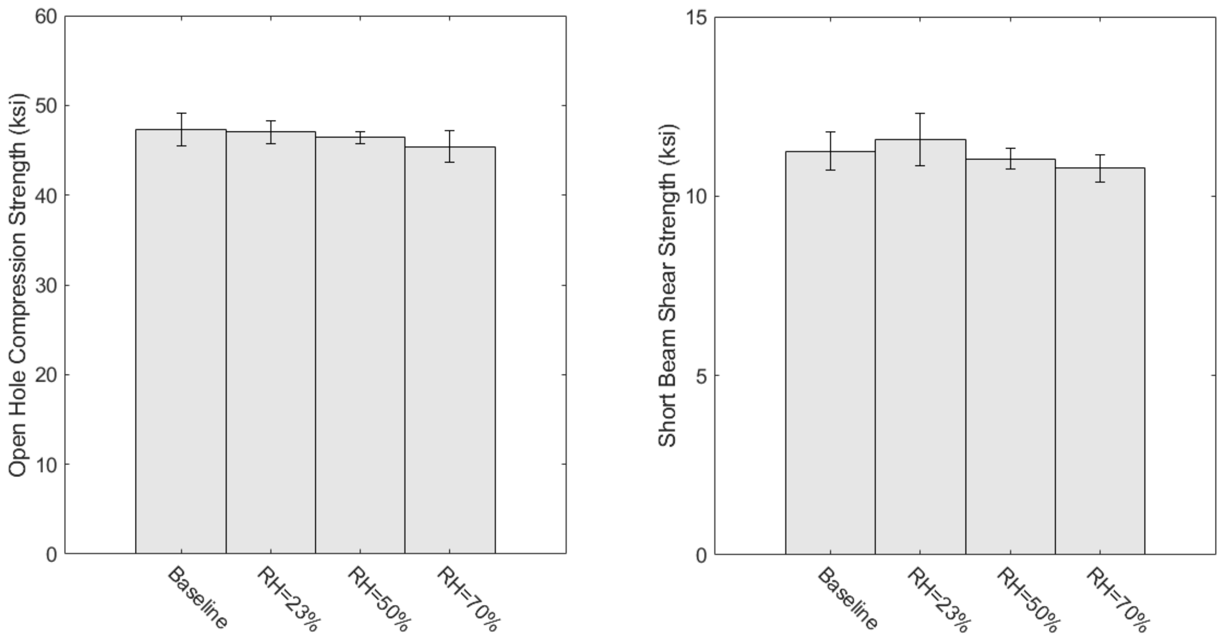
Figure 3. Graphical representation of constituent content results.

3.2 Mechanical Test Results

Open hole compression and short beam shear test results are presented in detail in Table 3 and are shown graphically in Figure 4. Values are based on a minimum of 5 samples for each test group.

Table 3. Open hole compression and short beam shear test results. Coefficients of variation are shown in italics.

Test Group	Open Hole Compression Strength (ksi)	Short Beam Shear Strength (ksi)
Baseline	47.3 ± 1.8 *(3.8%)*	11.3 ± 0.5 *(4.4%)*
RH = 23%	47.0 ± 1.3 *(2.8%)*	11.6 ± 0.7 *(6.0%)*
RH = 50%	46.5 ± 0.7 *(1.5%)*	11.0 ± 0.3 *(2.7%)*
RH = 70%	45.4 ± 1.8 *(4.0%)*	10.8 ± 0.4 *(3.7%)*



(a) (b)

Figure 4. Graphical representation of (a) open hole compression and (b) short beam shear test results.

Similar observations can be made based on open hole compression and short beam shear test results – while some variation is present, each of the four test groups performed in a similar manner. With respect to both open hole compression and short beam shear strength, the environmentally conditioned test groups performed within 5% of the baseline test group based on mean values. To complement the quantitative data gathered, failure modes were examined for each specimen tested and were consistent across the test groups considered. While open hole compression strength may be thought of as a fiber-dominated property since failure occurs as a result of the stress concentration that arises near the hole (where the severity of said stress concentration is driven by laminate stiffness), the matrix plays a significant role as well. This is best evidenced by typical trends in open hole compression test results in the room temperature ambient environment compared to the elevated temperature wet environment, including those reported in Toray's technical data sheet for the 3960 matrix system [24] (note that similar trends can be observed for IM7/8552 [25], T650/5320-1 [26], and HTS40/MTM45-1 [27]). Because fiber performance does not vary significantly within the temperature and RH ranges typically considered, the knockdowns commonly observed in open hole compression strength in the elevated temperature wet environment signify that the matrix – which is affected by both temperature and RH – is a significant driver with respect to open hole compression strength. It follows, then, that the open hole compression test results gathered herein show that exposure of T1100/3960 prepreg to high RH values prior to layup does not adversely affect matrix performance in cured laminates. This takeaway is reinforced by the short beam shear test results (given that short beam shear strength is dominated by matrix and fiber-matrix interface performance), which also show no significant declines as a result of environmental conditioning.

3.3 Dynamic Mechanical Analysis (DMA) Results

DMA results are presented in detail in Table 4 and are shown graphically in Figure 5. Values are based on a minimum of 5 samples for each test group.

Table 4. Dynamic mechanical analysis (DMA) results. Coefficients of variation are shown in italics.

	Glass Transition Temperature, T_g (°F)	
Test Group	Storage Modulus Shoulder	Tan Delta Peak
Baseline	282 ± 9.2 *(3.3%)*	374 ± 8.6 *(2.3%)*
RH = 23%	285 ± 10 *(3.5%)*	374 ± 5.8 *(1.6%)*
RH = 50%	289 ± 10 *(3.5%)*	369 ± 7.3 *(2.0%)*
RH = 70%	282 ± 7.6 *(2.7%)*	368 ± 3.3 *(0.9%)*

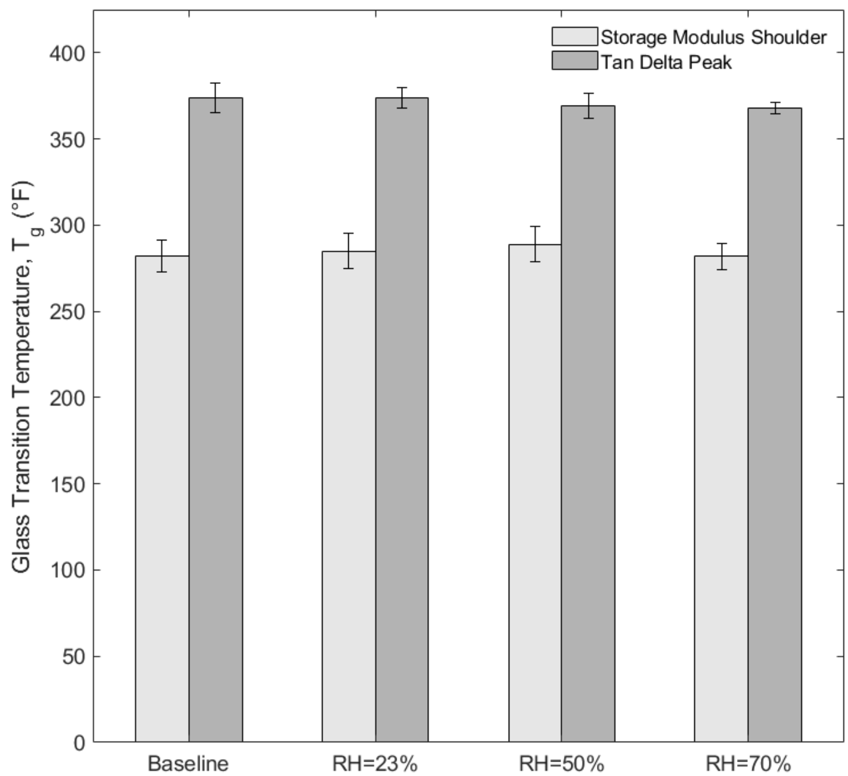


Figure 5. Graphical representation of dynamic mechanical analysis (DMA) results.

Similar to the trends (or lack thereof) observed in the mechanical test results, the four test groups considered herein performed in a similar manner with respect to glass transition temperature. Environmentally conditioned test groups exhibited mean glass transition temperatures within 3% of the baseline test group. This suggests that any plasticization of the matrix material as a result of environmental conditioning at elevated RH values for a period of 14 days prior to layup does not manifest itself in notably reduced glass transition temperatures.

In the interest of completeness, Figure 6 shows representative DMA output from each of the four test groups considered. Note that storage modulus values are normalized by the initial storage modulus value for run.

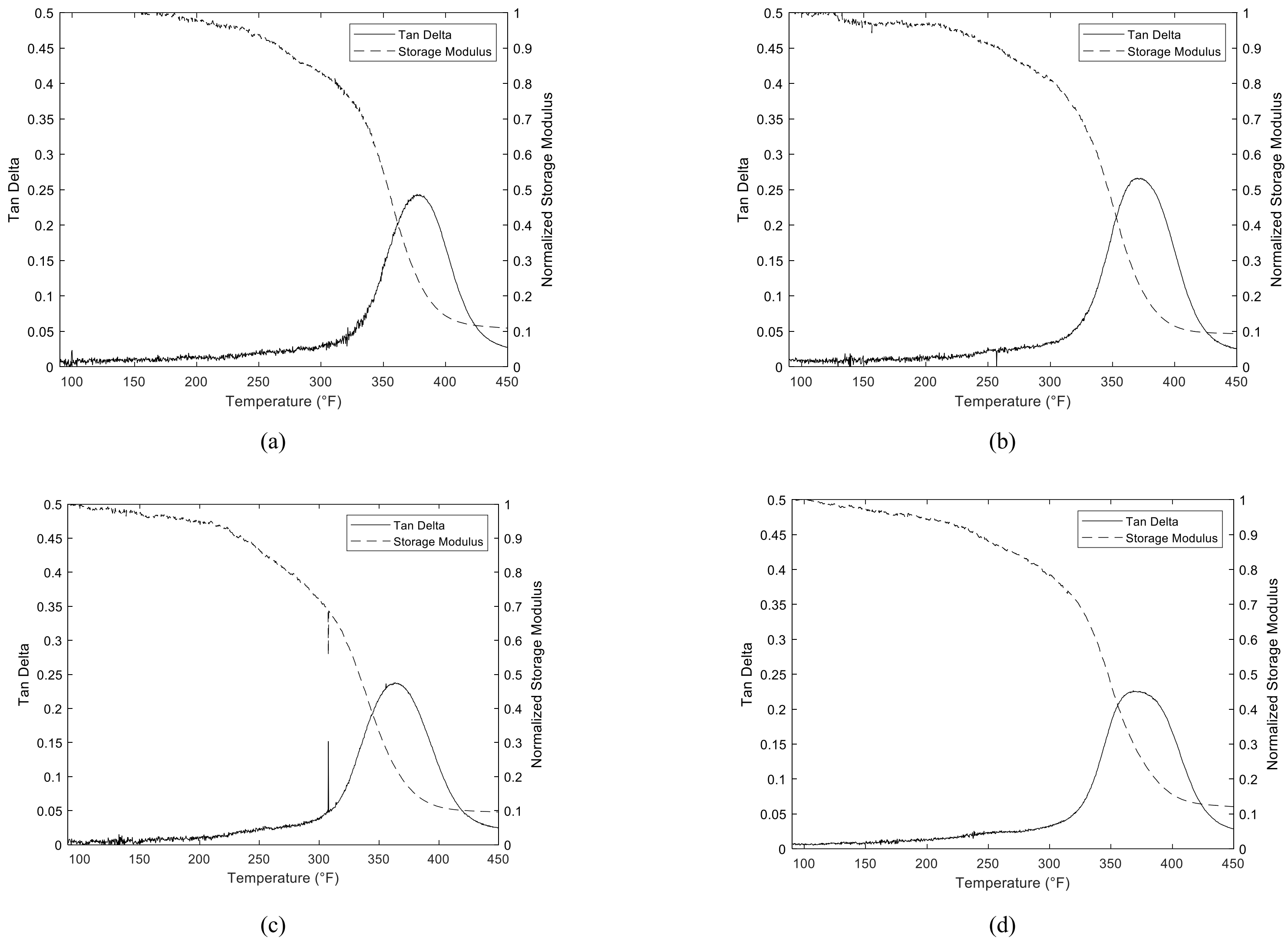


Figure 6. Representative DMA output for (a) baseline test group, (b) RH = 23% test group, (c) RH = 50% test group, and (d) RH = 70% test group.

4.0 **Conclusions**

The work carried out in this study provides for several conclusions with respect to environmental conditioning of T1100/3960 carbon fiber/epoxy prepreg prior to layup and cure. Following 14 days of exposure to a range of environmental conditions typical of large-volume high bay manufacturing facilities (up to 77°F and 70% RH), T1100/3960 prepreg was used to fabricate a series of composite laminates that were then evaluated for physical and mechanical performance. Optical microscopy and constituent content testing shows that nominal laminate quality is the same (or at least, very similar) among the four test groups considered. Open hole compression and short beam shear tests – both of which screen for matrix degradation, while open hole compression also reliably indicates fiber performance – shows that environmental conditioning of the prepreg prior to layup and cure has no significant effect on composite laminate performance. DMA shows that the environmental conditioning considered herein does not affect glass transition temperature, which signifies that any matrix plasticization that occurs as a result of extended exposure to elevated RH does not manifest itself in an adverse change in glass transition temperature in cured T1100/3960 laminates.

Taken together, these results and accompanying conclusions suggest that 77°F and 70% RH are reasonable upper bounds for a manufacturing facility where T1100/3960 prepreg is processed. While it is advisable to maintain lower RH values in the manufacturing environment whenever possible – given that the risk of moisture uptake in prepreg materials should be minimized as a best practice – the results gathered herein are clear in that 14 days of exposure at up to 77°F and 70% RH while in the prepreg form does not have an adverse effect on T1100/3960 in the cured laminate form.

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