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Determination of Temperature/ Moisture Sensitive Composite Properties

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16. Abstract

An investigation of the temperature and moisture sensitive composite properties was conducted. Results provide an evaluation of the degree of conservatism of the 50°F margin from glass transition values commonly used to determine a material system's operational limit (MOL). A series of dynamic mechanical analysis (DMA) and static mechanical tests were performed using specimens conditioned at three relative humidity levels and tested at six different temperatures for two commonly used 270°F cure prepreg systems. This report documents the results obtained from the DMA and static mechanical tests from which moisture and temperature sensitive properties can be determined. Limitations of the scope of this project are addressed as a means of providing recommendations for future research.

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EXECUTIVE SUMMARY

The 50°F margin commonly used to determine a given composite system's material operational limit (MOL) from glass transition temperature values (T_g) was evaluated. Dynamic mechanical analysis (DMA) and static mechanical tests were the major tests used to evaluate this rule as applied to 270°F cured composite materials. The mechanical tests performed included compression strength and modulus, in-plane shear strength and modulus, and open-hole compression tests. These tests were performed at 70° ±10°F, 150° ±5°F, 180° ±5°F, 210° ±5°F, 240° ±5°F, and 270° ±5°F with specimens conditioned at 0% relative humidity (RH), 68% RH, and 85% RH. The DMA tests were conducted using two different specimen thickness values and two different temperature ramp-up rates (1°C/min and 5°C/min) with specimens that were also conditioned at 0% RH, 68% RH, and 85% RH. The data from these tests provided a detailed source of information to determine the moisture and temperature sensitive composite properties and to re-evaluate the 50°F margin.

The results of the DMA tests indicated that for both material systems the lowest T_g values were obtained at 85% RH, followed those obtained at 68% RH, and finally, the dry T_g values. The operational limit of both material systems was determined using the 50°F margin method at 85% RH. For Newport 7781/NB321 glass fabric material system, the MOL was found to be 169°F. For the FiberCote E 765/T300 3KPW carbon fabric material system, the MOL was found to be 179°F, which is very close to 180°F, a typical maximum operating temperature. This indicates that if the 50°F rule was relaxed by 10° to 40°F, both the 270°F cure materials would pass the modified rule. Such a change in the rule is reasonable, as the mechanical properties are not reduced drastically at higher temperatures but decrease in a controlled manner.

For the mechanical tests performed, with the exception of compression modulus, all the properties degraded as a function of temperature regardless of the moisture level. It was also found that moisture increased the property degradation due to the increase in temperature. For both material systems, in-plane shear modulus was found to be the most sensitive property to moisture and temperature.

The degree of conservatism of the 50°F margin method, which is based on T_g values, was assessed using the 2/3 retention method. This method evaluates the drop in the mechanical properties between 180° and 230°F (50° above 180°F). A drop of more than 1/3 or less is acceptable, suggesting that the value of the MOL used was appropriate. A drop of more than 1/3 of the mechanical property is not acceptable, suggesting that the MOL value used was not appropriate since significant property degradations were induced. At 0% RH, all the mechanical properties satisfied the 2/3 rule for both material systems. At 68% RH, in-plane shear modulus was the only property that failed the 2/3 rule specifications for both material systems. At 85% RH, the in-plane shear modulus and the open-hole compression strength failed the 2/3 rule for the Newport glass fabric material system. For the FiberCote carbon fabric material system, all the properties failed the specifications of the 2/3 rule with the exception of in-plane shear strength. However it should be noted that the lowest retention value at 85% RH was 56%.

It is recommended that the 50°F margin method based on T_g values should be retained for the 270°F cured materials. If there is difficulty meeting this rule, 2/3 retention rule of mechanical properties should be used, but it will require additional testing at temperatures above MOL.

The results obtained were based on static tests. More extensive research needs to be performed to determine a realistic method of finding the MOL. The method should consider results of time dependent properties such as creep, a probabilistic study, that would determine the likelihood of simultaneous occurrence of high temperature and moisture content, and a thermomodeling analysis that would determine realistic usage temperatures.

1. INTRODUCTION.

1.1 PURPOSE.

This program has two major purposes related to the properties of composite materials: first to investigate the effect of humidity on the mechanical properties and the material operational limit (MOL) and second to re-evaluate the validity of the 50°F margin used to determine the MOL from glass transition temperature (T_g) values.

1.2 BACKGROUND.

A 50°F margin between the wet T_g (obtained at 85% RH) and the allowable operating temperature has been used to avoid the use of composite materials in the mechanical property response area where large rates of property degradation exist (primarily for properties that are matrix dependent) [1]. This 50°F margin has been effective and did not cause significant economic penalties for composite materials cured at 350°F. However, with the increased usage of 250° and 200°F vacuum bag cured composite materials and adhesives, there is a need to re-examine the method used to determine the MOL.

The main purpose of this project was to characterize the temperature and moisture dependent properties for the appropriate materials not only at extreme levels of temperature and moisture content, but also at other levels of moisture content and temperature to determine functional dependance. There were indications that the use of 85% RH moisture content in design as the criteria for each of life moisture content is unrealistic [2]. More realistic moisture contents were obtained [3] from worldwide observations. These corresponded to saturation levels consistent with 68% RH. There are also applications and material types in which some violation of the 50°F margin may be permitted, while maintaining sufficient life and static strength margins of safety.

The first task to be performed was to re-examine the hot and wet combination used to generate design allowables. In fact, the hot and wet condition, as assumed in the National Aeronautics and Space Administration (NASA) AGATE material qualification program [4], was based on the assumption that an aircraft which has reached equilibrium moisture content at 85% RH may experience a corresponding hot condition at 180°F (i.e., hot-wet). The 85% RH was a realistic relative humidity level for some parts of the world such as in Southeast Asia. At that location, diurnal moisture levels range between the high 90s in the early morning to around 60% in the mid-afternoon. Mean value is 84%. During prolonged heavy rain, relative humidity often reaches 100% [5]. Collings suggests that the worldwide worst environment might best be simulated by a constant humidity of 84% [6]. Furthermore, relative humidity data was collected over a period of 16 to 30 years by Brunei [7], yielding the following values:

- Mean RH in January: 86%
- Mean RH in February: 86%
- Mean RH in March: 85%
- Mean RH in April: 85%
- Mean RH in May: 85%

- Mean RH in June: 85%
- Mean RH in July: 85%
- Mean RH in August: 84%
- Mean RH in September: 85%
- Mean RH in October: 86%
- Mean RH in November: 87%
- Mean RH in December: 87%

According to the meteorological services of Malaysia [8], the mean monthly relative humidity falls between 70% to 90%, varying from place to place and from month to month. In fact, the minimum range is found in Sitiawan where the mean relative humidity varies from a low of 84% in February to a high of only 88% in November. The maximum range is found in the northwest area of the Malaysian peninsula (Alor Setar) where the mean relative humidity varies from a low of 72% to a high of 87%. The minimum relative humidity is normally found in the months of January and February except for the east coast states of Kelantan and Terengganu, which have the minimum in March. The maximum is, however, generally found in the month of November.

As in the case of temperature, the diurnal variation of relative humidity is much greater as compared to the annual variation. The mean daily minimum can be as low as 42% during the dry months and reaches as high as 70% during the wet months. The mean daily maximum, however, does not vary much from place to place and is at no place below 94%. It may reach as high as nearly 100%. Again, the northwest states of Kedah and Perlis have the largest diurnal variation of relative humidity.

The indications that show that an 85% RH is unrealistic were based on investigations performed in areas with average annual relative humidity level of 80% or less [9]. "The moisture contents of four graphite/epoxy and two Kevlar/epoxy material systems after seven years of exposure at six exposure sites… NASA Langley in Hampton, VA; San Diego, CA; Honolulu, HA; Wellington, New Zealand; Sao Paulo, Brazil; and Frankfurt, W. Germany" [9]. "In general, the specimens exposed at Sao Paulo, Brazil, had the highest moisture content. This result is somewhat expected since the average annual relative humidity level is about 80 percent at Sao Paulo" [9].

However, the combination of hot and wet (180ºF and 85% RH) as assumed in the AGATE material qualification program may be unrealistic since:

a. humid places such as Southeast Asia rarely experience temperature higher than 105°F

b. warm and dry places such as the deserts of Arizona will likely to dry out the moisture in the composite and rarely experience humidity levels as high as 85% RH.

The second purpose was to re-evaluate the validity of the 50°F margin used to determine the MOL from glass transition temperature values. Figure 1 indicates that the MOL determined from the 50°F margin may not always corroborate with mechanical test data.

In the first instance, the mechanical data corroborates the chosen T_g. In the second case, the mechanical data suggests that the MOL predicted from the T_g is conservative. In the third case, the mechanical test data indicates that the MOL predicted from the T_g is not conservative.

MIL-HDBK-17-1E, DOD Coordination Working Draft

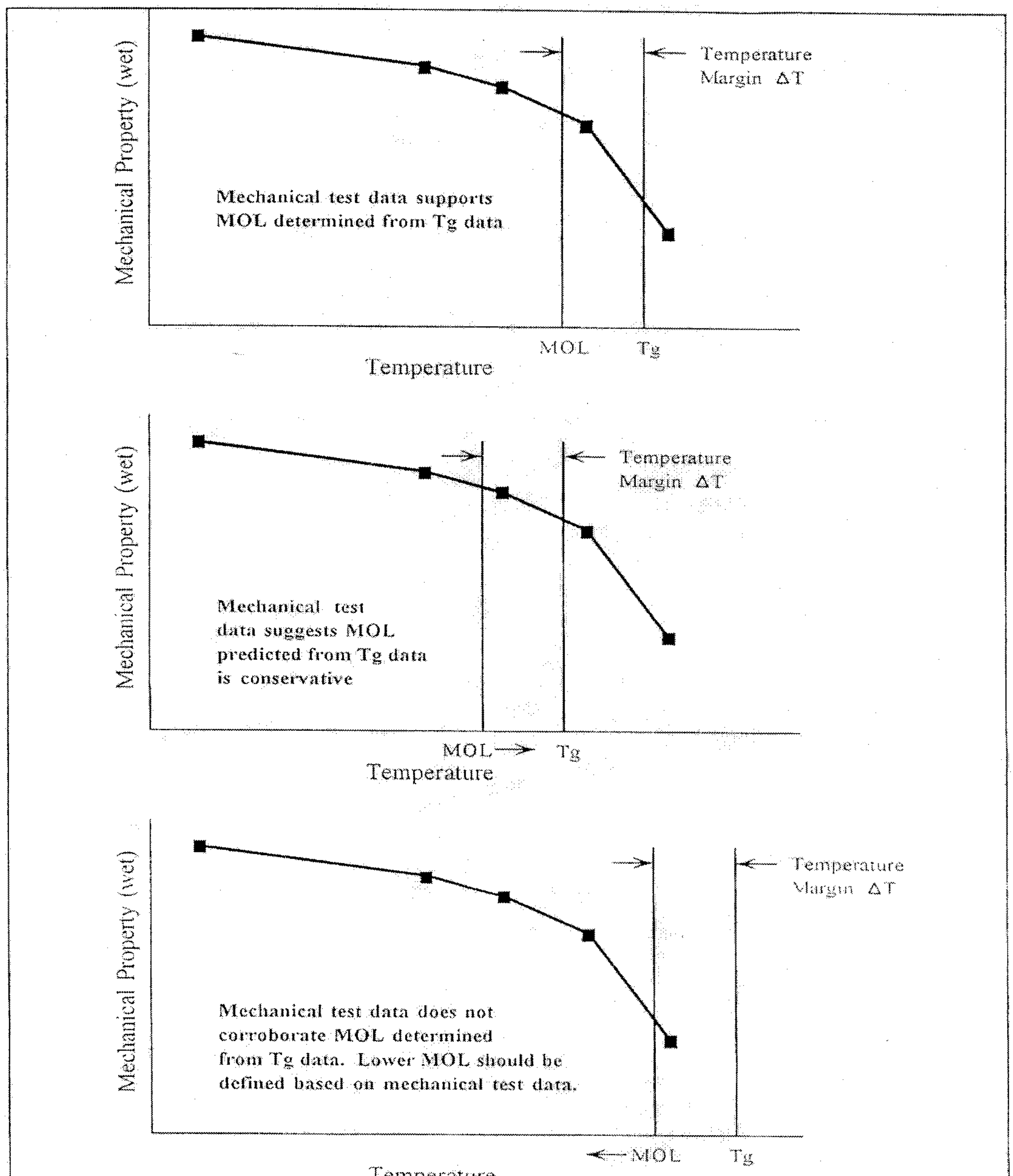

FIGURE 2.2.8(c) *Use of mechanical and T_g data todetermine MOL.*

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FIGURE 1. EVALUATION OF THE MOL USING T_g AND MECHANICAL DATA [10]

2. TECHNICAL PROCEDURE.

Two material systems were selected for the purpose of this study:

- Graphite plain weave/epoxy (FiberCote E 765/T300 3KPW)
- Fiberglass/epoxy (Newport 7781/NB321)

The average cured per ply thicknesses are 0.0087 in and 0.009 in, respectively. These two material systems are presently being used by certified general aviation aircraft.

Table 1 shows the number of plies per panel as well as the lay-up used for each test method.

TABLE 1. PANEL SPECIFICATIONS FOR EACH TEST METHOD

Panel Test Method	Code	Number of Plies	Lay-up
Compression Strength and Modulus	ASTM D695-96 or SRM 1R-94	14	$[0]_{14}$
In-plane shear strength and modulus	ASTM D5379	16	$[0/90]_{4S}$
Open-hole compression strength	SRM 3R-94	16	$[+45/0/-45/90]_{2S}$
Dynamic mechanical analysis	ASTM D5418-95 SRM 18R-94	5 14	$[0]_5$ $[0]_{14}$

After all panels were laid up, they were vacuum bagged and cured in the oven at 270°F for 100 minutes.

Three relative humidity levels were considered for the purpose of this study: 0%, 68%, and 85% RH. For the 0% (dry) relative humidity level investigated, the specimens were dried in a vacuum oven until equilibrium was reached. For the 68% and the 85% RH, the specimens were conditioned in an environmental chamber at 145°F until the equilibrium moisture content was achieved. Moisture equilibrium was achieved when the average moisture content of the specimen changed by less than 0.05% for two consecutive readings within a span of 7 ±0.5 days.

Dynamic mechanical analysis (DMA) and mechanical tests were performed at all three relative humidity levels. Mechanical tests performed included compression strength and modulus, in-plane shear strength and modulus, and open-hole compression strength. These mechanical properties were obtained for six different temperatures: room temperature, 150°, 180°, 210°, 240°, and 270°F. At each humidity level and temperature, three tests were performed for both DMA and mechanical tests for each material system. For DMA tests the coefficients of variation (COV) of the three tests were below 7% for the FiberCote E765/T300 3KPW and below 4% for Newport 7781/NB321. Mechanical tests experienced COVs below 10%, except at 270°F for compression and shear moduli when the COV was in the 20% range.

Glass transition temperature values were obtained using DMA equipment (Perkin Elmer DMA 7e) with a 20-millimeter span, three-point bend fixture. DMA is a test method capable of determining the viscoelasticity behavior of a material. The term "viscoelasticity" is the derived from "viscosity" and "elasticity." It is the dual response of a material under an applied load where part of the material returns to its original shape when the load is removed (i.e., elastic portion of the material), while the other part undergoes permanent deformation (i.e., viscous portion of the material). Under minimal stress, metallic materials such as aluminum and steel have negligible viscous behavior and their behavior is mainly elastic. For this reason, Hooke's Law adequately models their behavior. On the other hand, materials such as liquid or fluid are often modeled using Newton's Law for their viscous behavior. Materials such as amorphous polymer (e.g., cured epoxy) exhibit both elastic and viscous behaviors, hence, viscoelasticity. DMA is capable of separating the two responses. Storage modulus is related to the elastic portion of the material; one that fully recovers when an applied load is removed. A fairly popular method of interpreting T_g is through the onset of storage modulus curve. Loss modulus, on the other hand, is related to the viscous portion of the material; one that sustains permanent deformation. The peak of loss modulus curve is sometimes interpreted as the T_g of a material, however, this method is not used in this report as it is usually higher than the storage modulus. Another method of interpreting T_g is known as the peak of tangent delta (δ). Tangent δ is the ratio of loss modulus to storage modulus. Appendix A provides a more detailed explanation of these definitions.

Two different temperature ramp-up rates (5°C/min and 1°C/min) and two different specimen thickness values were considered. The different thicknesses and ramp-up rates were used to find out the differences in the two standards prevalent in the industry. ASTM E1640 specifies 0.04 inch (1 mm) thickness and 1C/min ramp-up rate. Dissapointingly, there were no specific trends. Hence, an average value for all test configurations was used to determine T_g. The specimens were loaded under a fixed frequency of 1 Hz. The equipment calibration was performed according to the manufacturer's recommended procedures.

Mechanical test results were plotted to show the property variation with respect to temperature. Curve fits of the raw data were obtained using Grapher™ 2.0 software.

3. RESULTS.

3.1 ZERO PERCENT RELATIVE HUMIDITY.

3.1.1 Mechanical Test Results.

Figures 2 to 11 summarize the results obtained from the mechanical tests performed for both material systems at 0% relative humidity. At this relative humidity, the specimens are assumed 0% moisture content, as they were dried in the oven. On each of the graphs, a curve fit of the test data is shown by a solid line, while the actual test data is illustrated using symbols which represent averages of three tests. Stress values are shown in ksi while modulus values are shown in msi.

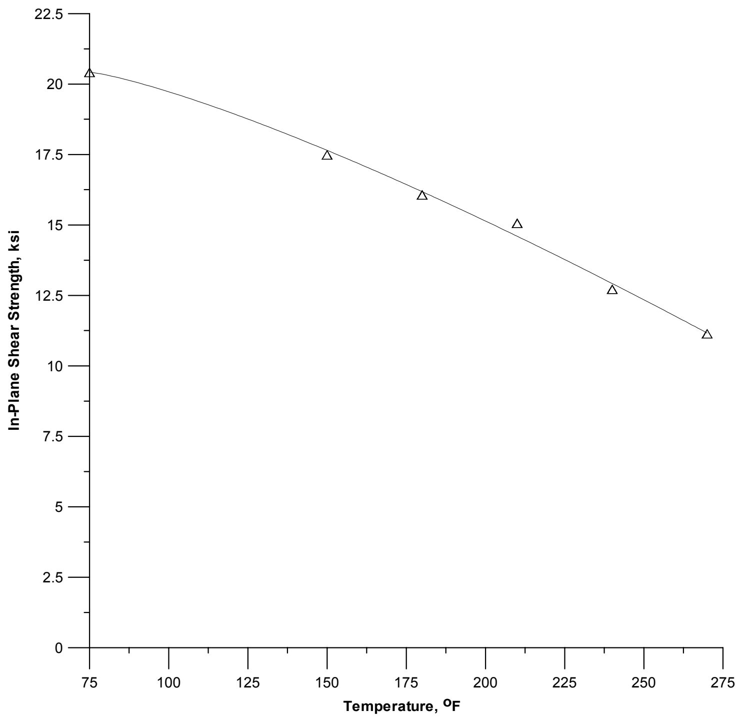

FIGURE 2. VARIATION OF IN-PLANE SHEAR STRENGTH VALUES AS A FUNCTION OF TEMPERATURE FOR NEWPORT 7781/NB321 GLASS FABRIC MATERIAL SYSTEM (0% RH)

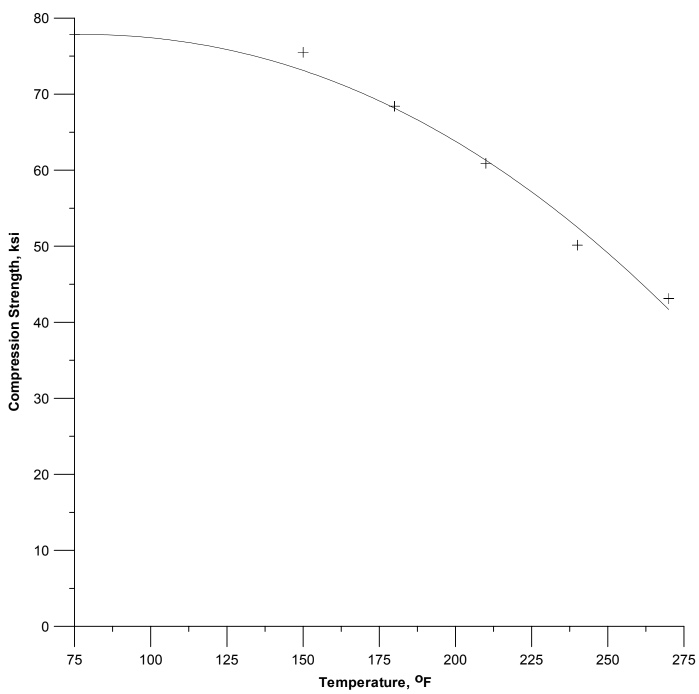

FIGURE 3. VARIATION OF COMPRESSION STRENGTH VALUES AS A FUNCTION OF TEMPERATURE FOR NEWPORT 7781/NB321 GLASS FABRIC MATERIAL SYSTEM (0% RH)

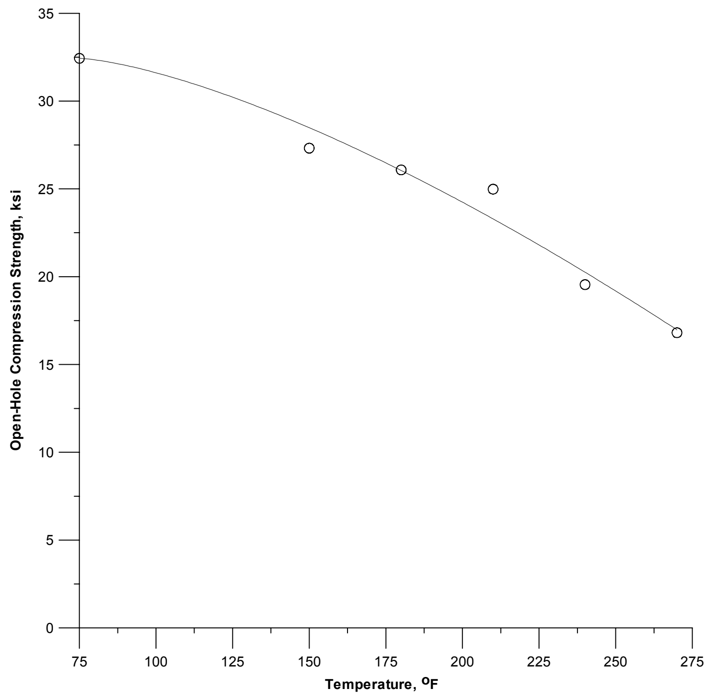

FIGURE 4. VARIATION OF OPEN-HOLE COMPRESSION STRENGTH VALUES AS A FUNCTION OF TEMPERATURE FOR NEWPORT 7781/NB321 GLASS FABRIC MATERIAL SYSTEM (0% RH)

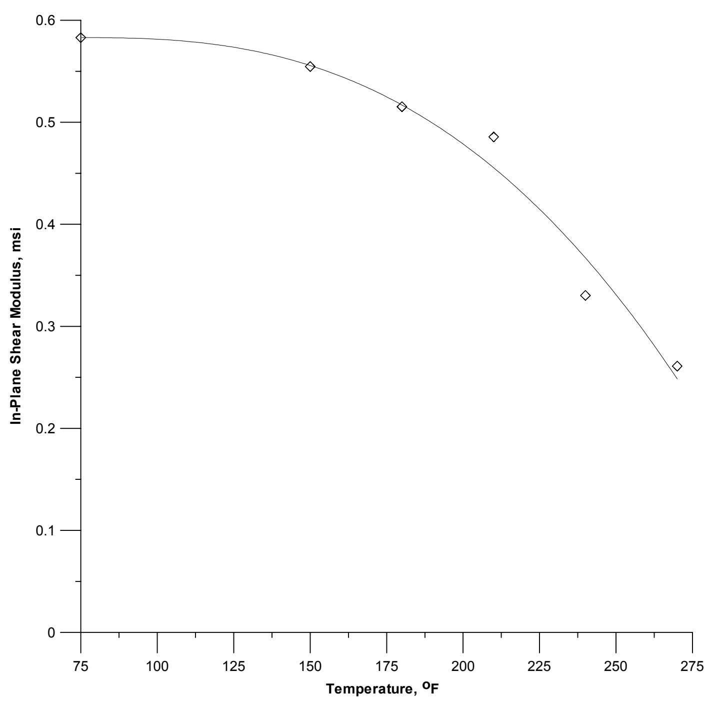

FIGURE 5. VARIATION OF IN-PLANE SHEAR MODULUS VALUES AS A FUNCTION OF TEMPERATURE FOR NEWPORT 7781/NB321 GLASS FABRIC MATERIAL SYSTEM (0% RH)

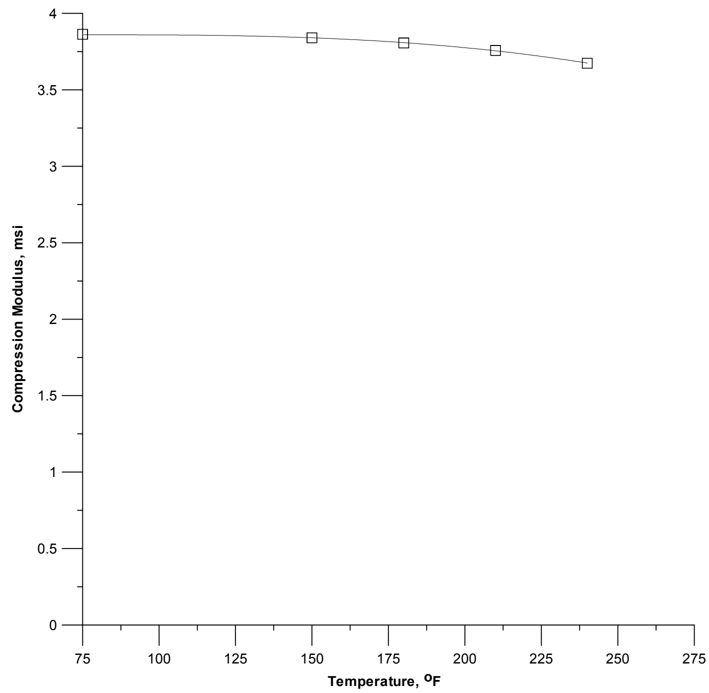

FIGURE 6. VARIATION OF COMPRESSION MODULUS VALUES AS A FUNCTION OF TEMPERATURE FOR NEWPORT 7781/NB321 GLASS FABRIC MATERIAL SYSTEM (0% RH)

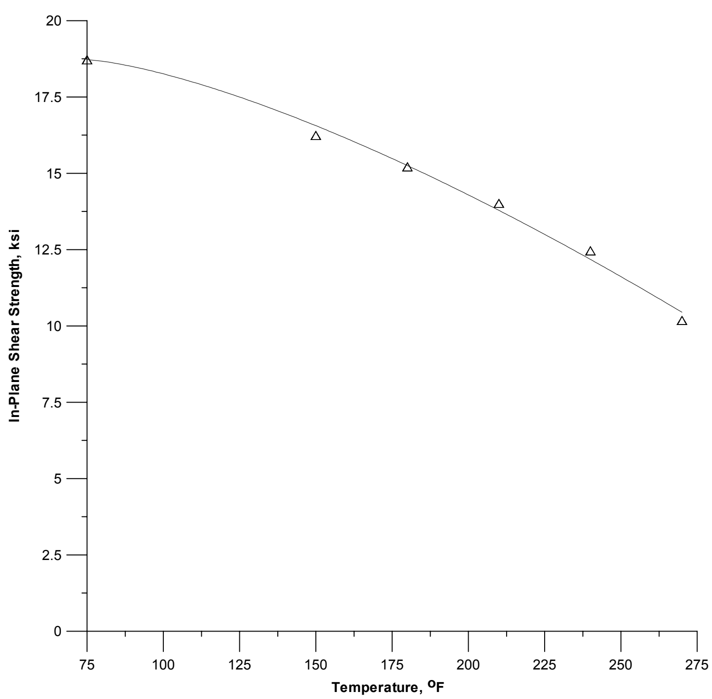

FIGURE 7. VARIATION OF IN-PLANE SHEAR STRENGTH VALUES AS A FUNCTION OF TEMPERATURE FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC MATERIAL SYSTEM (0% RH)

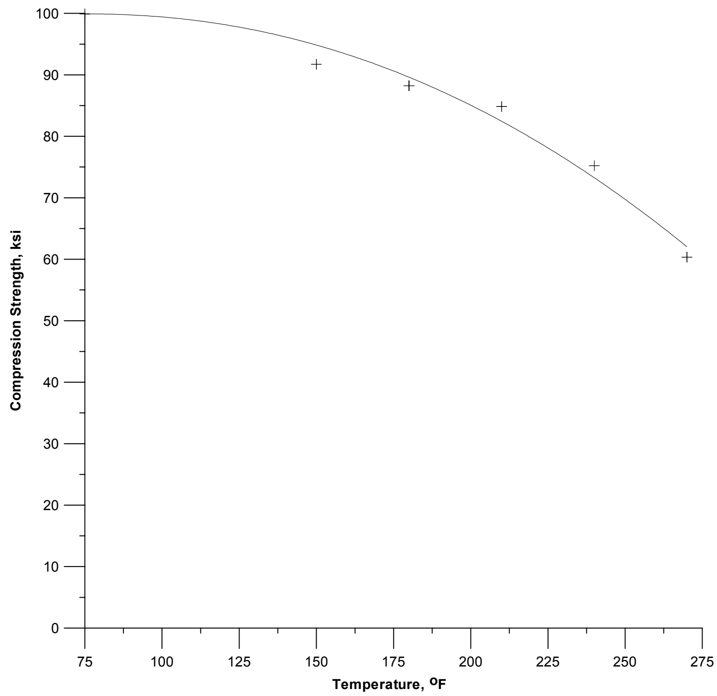

FIGURE 8. VARIATION OF COMPRESSION STRENGTH VALUES AS A FUNCTION OF TEMPERATURE FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC MATERIAL SYSTEM (0% RH)

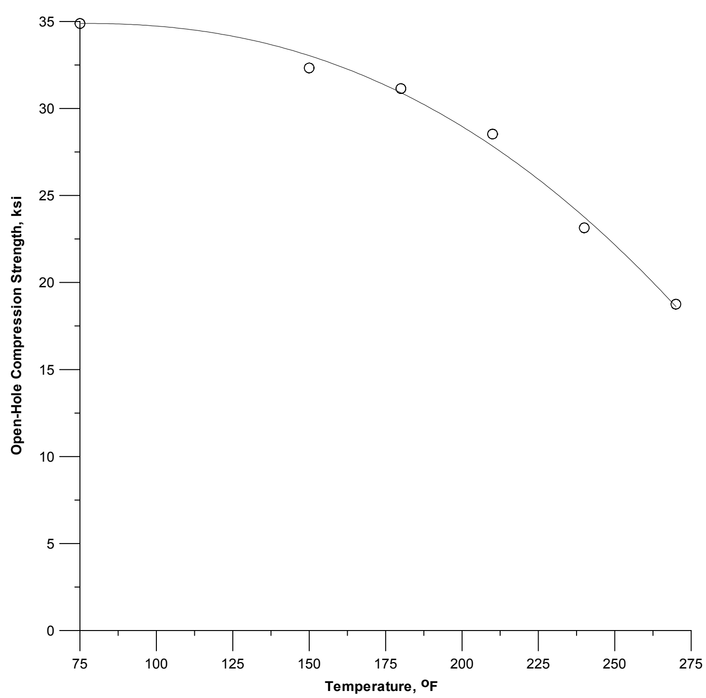

FIGURE 9. VARIATION OF OPEN-HOLE COMPRESSION STRENGTH VALUES AS A FUNCTION OF TEMPERATURE FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC MATERIAL SYSTEM (0% RH)

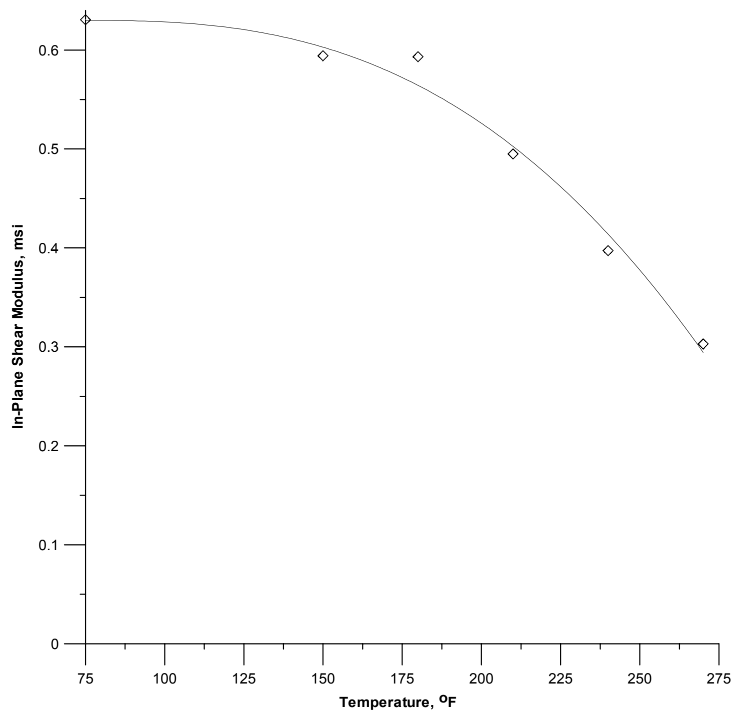

FIGURE 10. VARIATION OF IN-PLANE SHEAR MODULUS VALUES AS A FUNCTION OF TEMPERATURE FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC MATERIAL SYSTEM (0% RH)

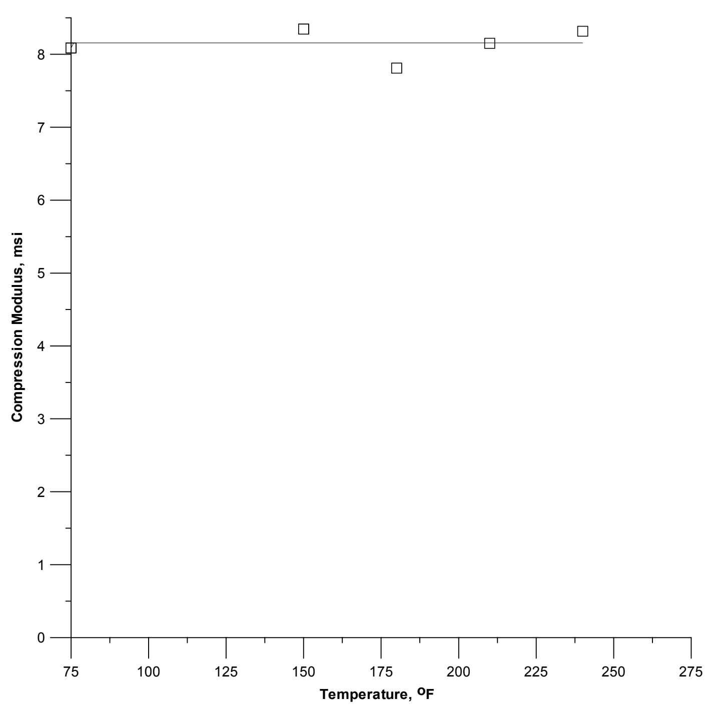

FIGURE 11. VARIATION OF COMPRESSION MODULUS VALUES AS A FUNCTION OF TEMPERATURE FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC MATERIAL SYSTEM (0% RH)

3.1.2 DMA Test Results.

Tables 2 and 3 list the average glass transition temperature values obtained for both material systems at 0% relative humidity. The tables also show the T_g values corresponding to the different temperature ramp-up rates and specimen thickness values considered.

TABLE 2. DYNAMIC MECHANICAL ANALYSIS TEST RESULTS FOR NEWPORT 7781/NB321 GLASS FABRIC MATERIAL SYSTEM (0% RH)

	T_g (Onset of Storage Modulus) (°F)	T_g (Peak of Tangent Delta) (°F)
Thick Specimens		
5°C/min	326	358
1°C/min	323	358
Thin Specimens		
5°C/min	312	362
1°C/min	339	370

TABLE 3. DYNAMIC MECHANICAL ANALYSIS TEST RESULTS FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC MATERIAL SYSTEM (0% RH)

	T_g (Onset of Storage Modulus) (°F)	T_g (Peak of Tangent Delta) (°F)
Thick Specimens		
5°C/min	305	365
1°C/min	319	377
Thin Specimens		
5°C/min	315	365
1°C/min	345	380

3.2 SIXTY-EIGHT PERCENT RELATIVE HUMIDITY.

3.2.1 Mechanical Test Results.

Figures 12 to 21 summarize the results obtained from the mechanical tests performed for both material systems conditioned at 68% relative humidity. The equilibrium moisture content at 68% RH of Newport 7781/NB321 material was 0.62% and FiberCote E 765/T300 3KPW material was 0.85%. The specimens were not dried prior to environmental conditioning, so the actual moisture content may be slightly higher than the values reported above (approximately 0.1%-0.2%). On each of the graphs, a curve fit of the test data is shown by a solid line, while the actual test data (averages of three tests) are illustrated using symbols. Stress values are shown in ksi while modulus values are shown in msi.

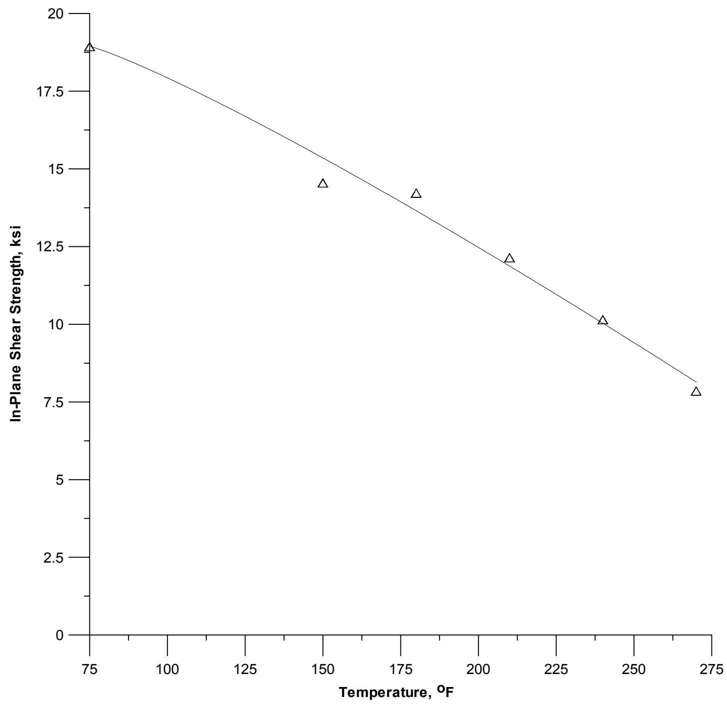

FIGURE 12. VARIATION OF IN-PLANE SHEAR STRENGTH VALUES AS A FUNCTION OF TEMPERATURE FOR NEWPORT 7781/NB321 GLASS FABRIC MATERIAL SYSTEM (68% RH)

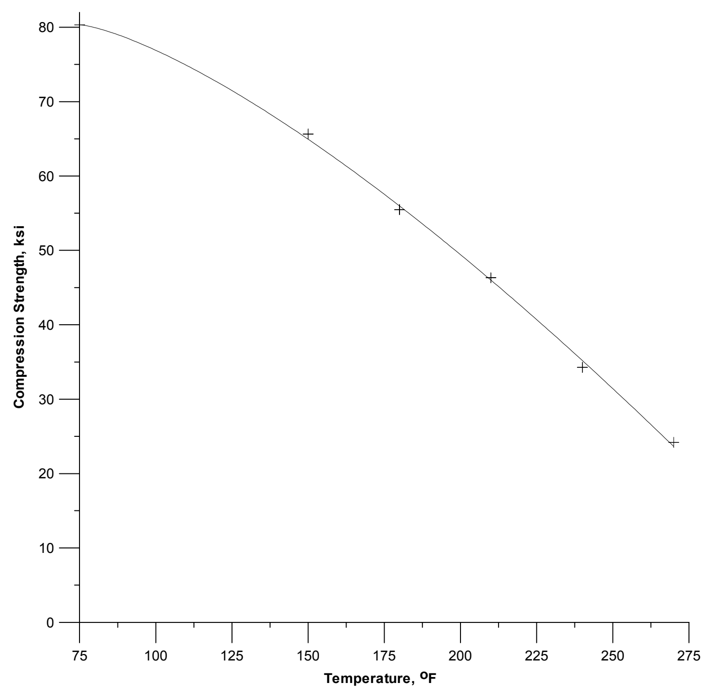

FIGURE 13. VARIATION OF COMPRESSION STRENGTH VALUES AS A FUNCTION OF TEMPERATURE FOR NEWPORT 7781/NB321 GLASS FABRIC MATERIAL SYSTEM (68% RH)

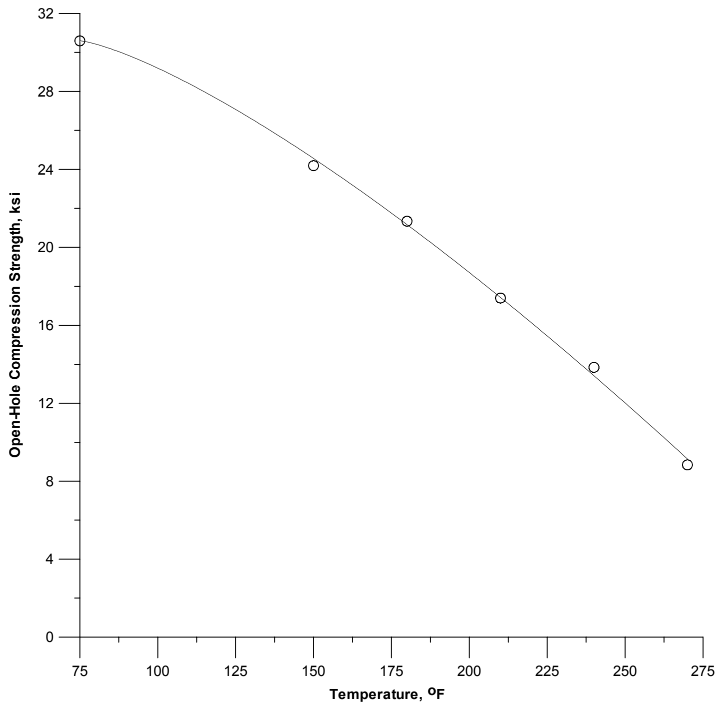

FIGURE 14. VARIATION OF OPEN-HOLE COMPRESSION STRENGTH VALUES AS A FUNCTION OF TEMPERATURE FOR NEWPORT 7781/NB321 GLASS FABRIC MATERIAL SYSTEM (68% RH)

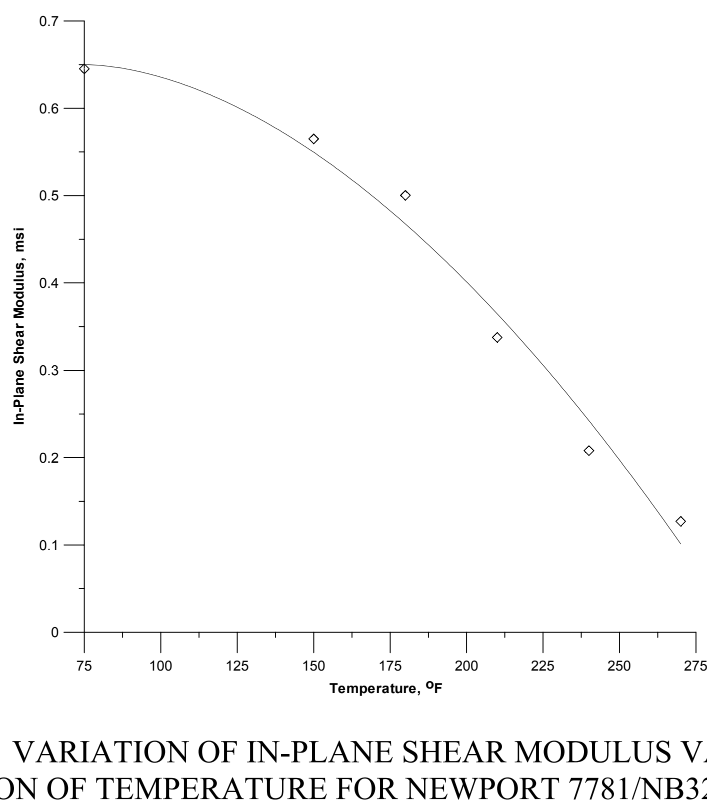

FIGURE 15. VARIATION OF IN-PLANE SHEAR MODULUS VALUES AS A FUNCTION OF TEMPERATURE FOR NEWPORT 7781/NB321 GLASS FABRIC MATERIAL SYSTEM (68% RH)

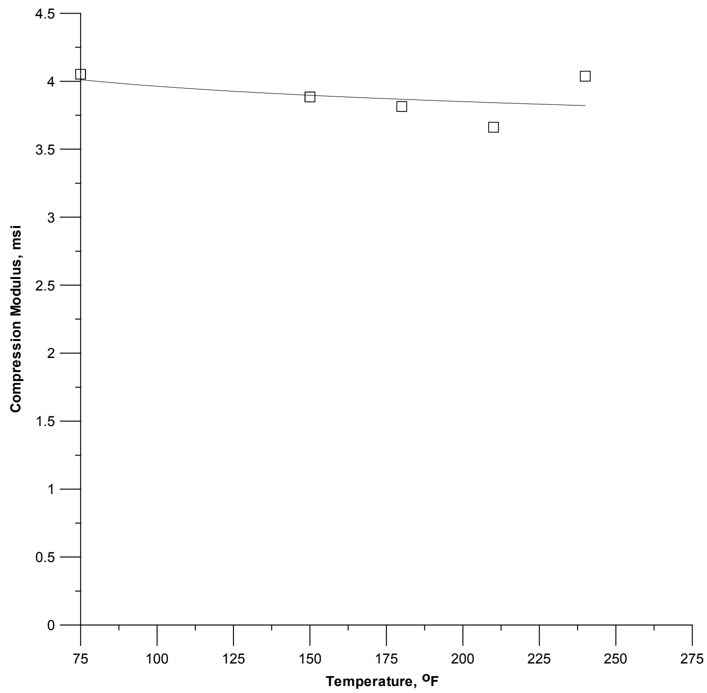

FIGURE 16. VARIATION OF COMPRESSION MODULUS VALUES AS A FUNCTION OF TEMPERATURE FOR NEWPORT 7781/NB321 GLASS FABRIC MATERIAL SYSTEM (68% RH)

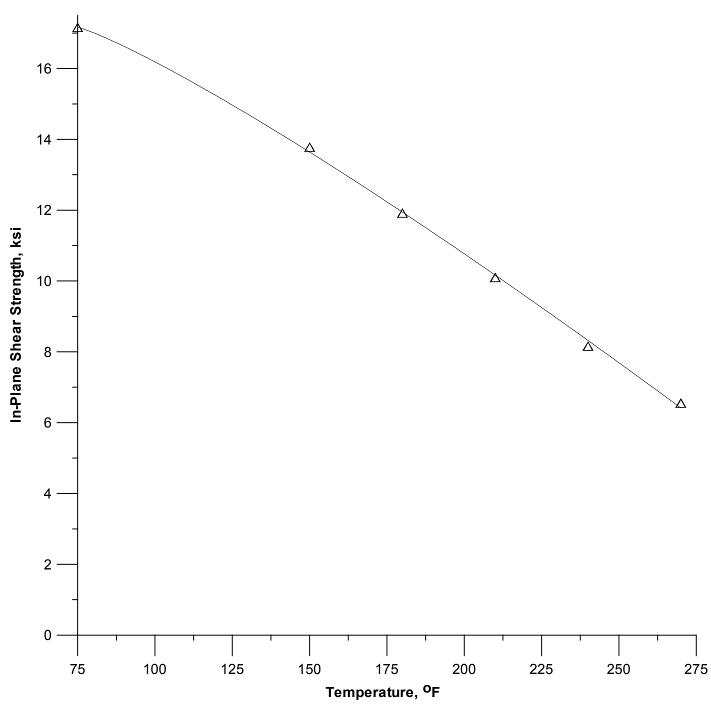

FIGURE 17. VARIATION OF IN-PLANE SHEAR STRENGTH VALUES AS A FUNCTION OF TEMPERATURE FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC MATERIAL SYSTEM (68% RH)

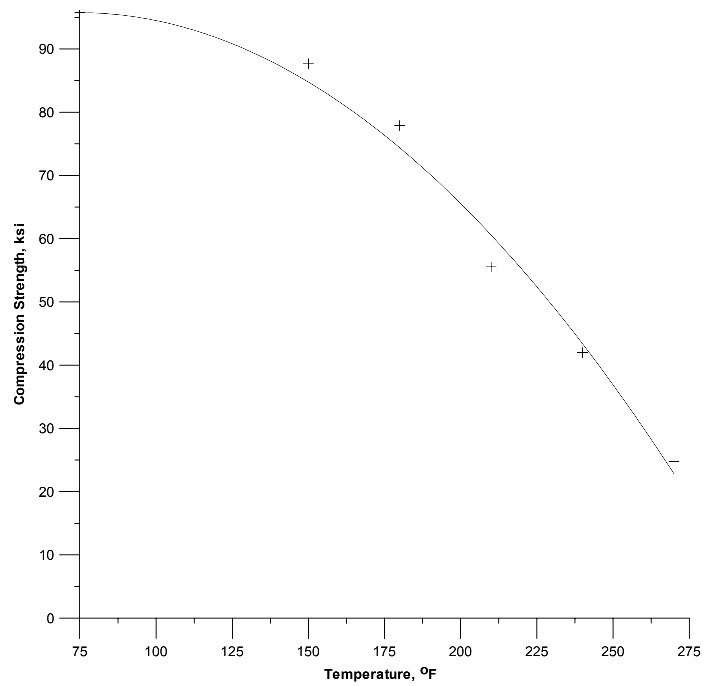

FIGURE 18. VARIATION OF COMPRESSION STRENGTH VALUES AS A FUNCTION OF TEMPERATURE FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC MATERIAL SYSTEM (68% RH)

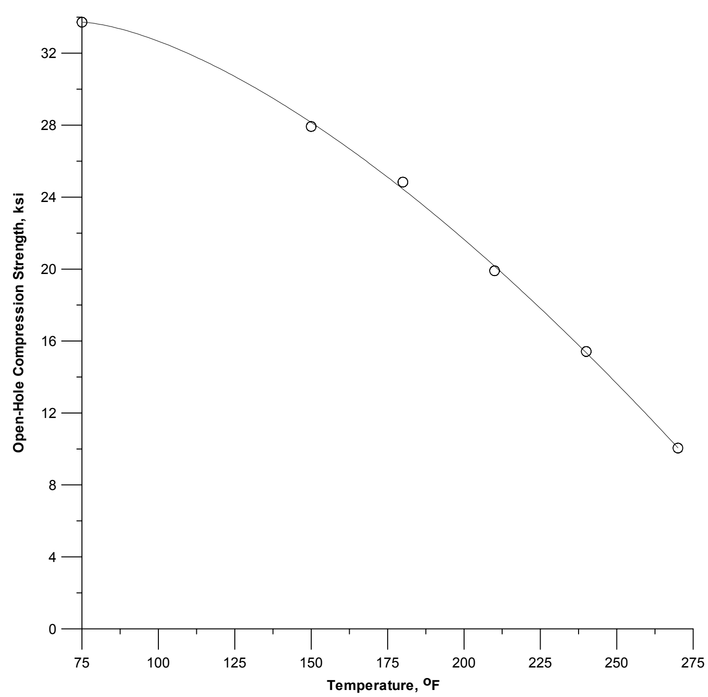

FIGURE 19. VARIATION OF OPEN-HOLE COMPRESSION STRENGTH VALUES AS A FUNCTION OF TEMPERATURE FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC MATERIAL SYSTEM (68% RH)

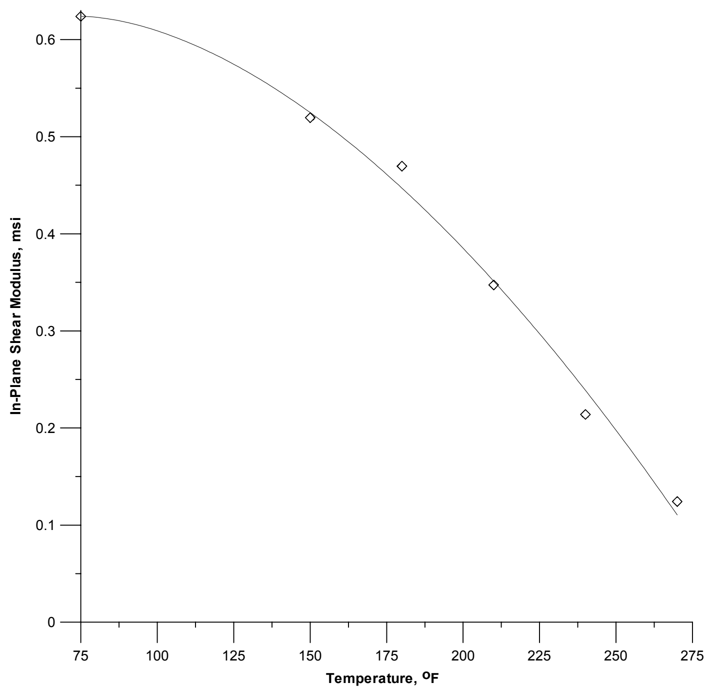

FIGURE 20. VARIATION OF IN-PLANE SHEAR MODULUS VALUES AS A FUNCTION OF TEMPERATURE FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC MATERIAL SYSTEM (68% RH)

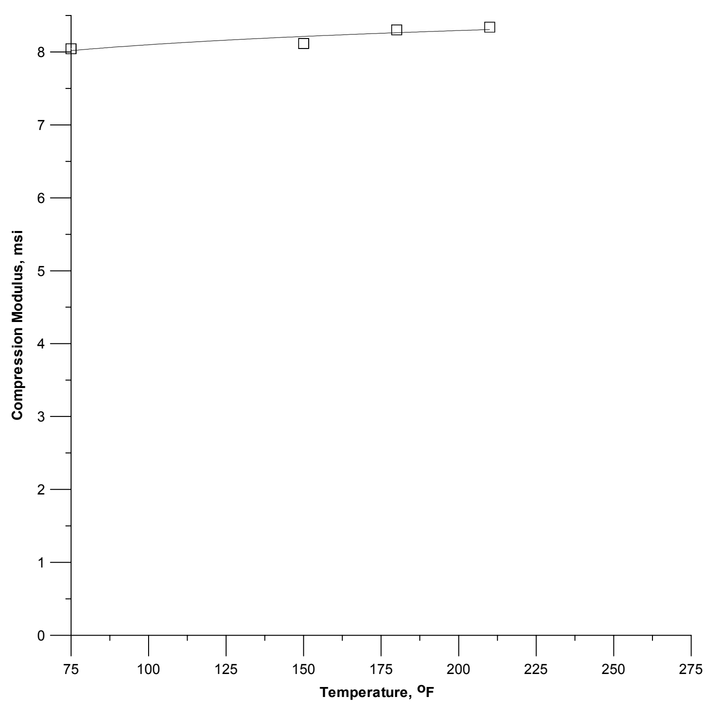

FIGURE 21. VARIATION OF COMPRESSION MODULUS VALUES AS A FUNCTION OF TEMPERATURE FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC MATERIAL SYSTEM (68% RH)

3.2.2 DMA Test Results.

Tables 4 and 5 list the average glass transition temperature values obtained for both material systems conditioned at 68% relative humidity. The tables also show the T_g values corresponding to the different temperature ramp-up rates and specimen thickness values investigated.

TABLE 4. DYNAMIC MECHANICAL ANALYSIS TEST RESULTS FOR NEWPORT 7781/NB321 GLASS FABRIC MATERIAL SYSTEM (68% RH)

	T_g (Onset of Storage Modulus) (°F)	T_g (Peak of Tangent Delta) (°F)
Thick Specimens		
5°C/min	250	291
1°C/min	263	291
Thin Specimens		
5°C/min	242	294
1°C/min	261	300

TABLE 5. DYNAMIC MECHANICAL ANALYSIS TEST RESULTS FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC MATERIAL SYSTEM (68% RH)

	T_g (Storage Modulus) (°F)	T_g (Tangent Delta) (°F)
Thick Specimens		
5°C/min	223	273
1°C/min	248	291
Thin Specimens		
5°C/min	252	285
1°C/min	251	303

3.3 EIGHTY-FIVE PERCENT RELATIVE HUMIDITY.

3.3.1 Mechanical Test Results.

Figures 22 to 31 summarize the results obtained from the mechanical tests performed for both material systems conditioned at 85% relative humidity. The equilibrium moisture content at the 85% RH of the Newport 7781/NB321 material was 1.01% and FiberCote E 765/T300 3KPW material was 1.31%. The specimens were not dried prior to environmental conditioning, so the actual moisture content may be slightly higher than the values reported above (approximately 0.1%-0.2%). On each of the graphs, a curve fit of the test data is shown by a solid line, while the actual test data is illustrated using symbols which represent averages of three tests. Stress values are shown in ksi while modulus values are shown in msi.

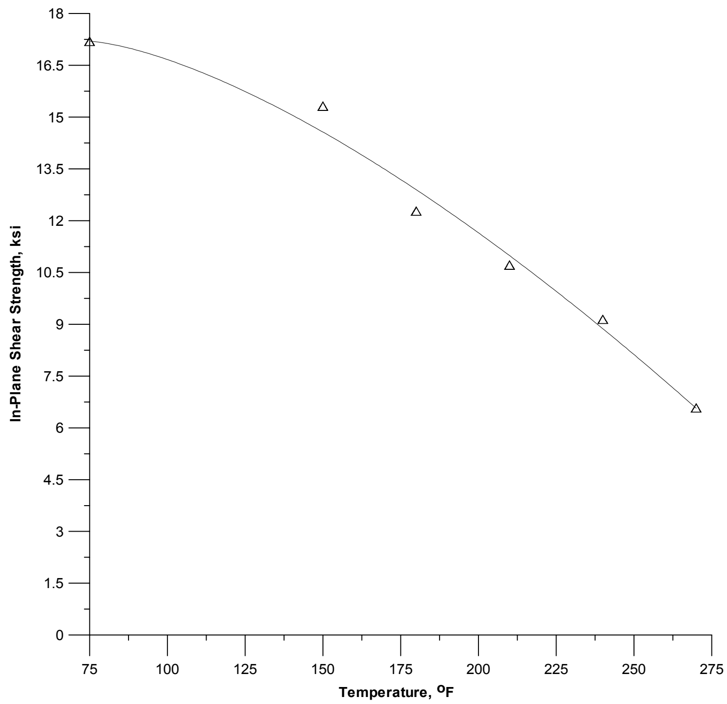

FIGURE 22. VARIATION OF IN-PLANE SHEAR STRENGTH VALUES AS A FUNCTION OF TEMPERATURE FOR NEWPORT 7781/NB321 GLASS FABRIC MATERIAL SYSTEM (85% RH)

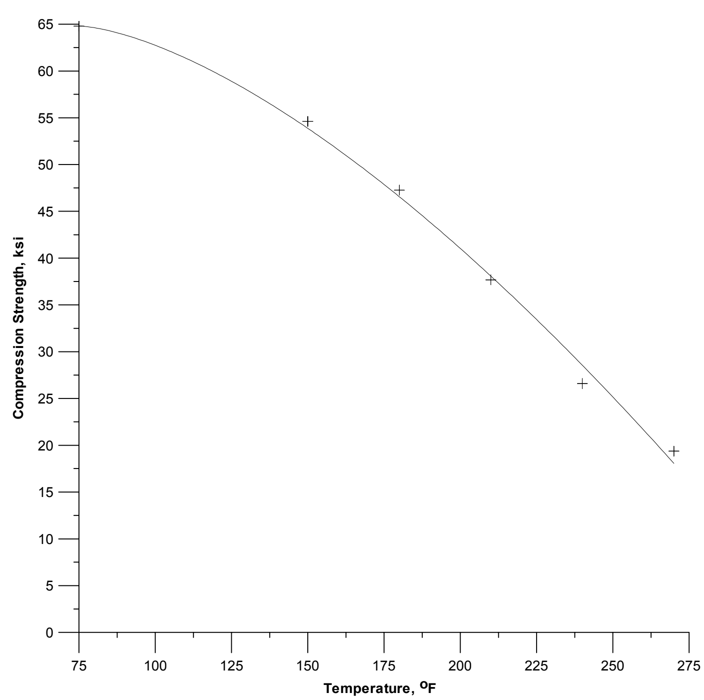

FIGURE 23. VARIATION OF COMPRESSION STRENGTH VALUES AS A FUNCTION OF TEMPERATURE FOR NEWPORT 7781/NB321 GLASS FABRIC MATERIAL SYSTEM (85% RH)

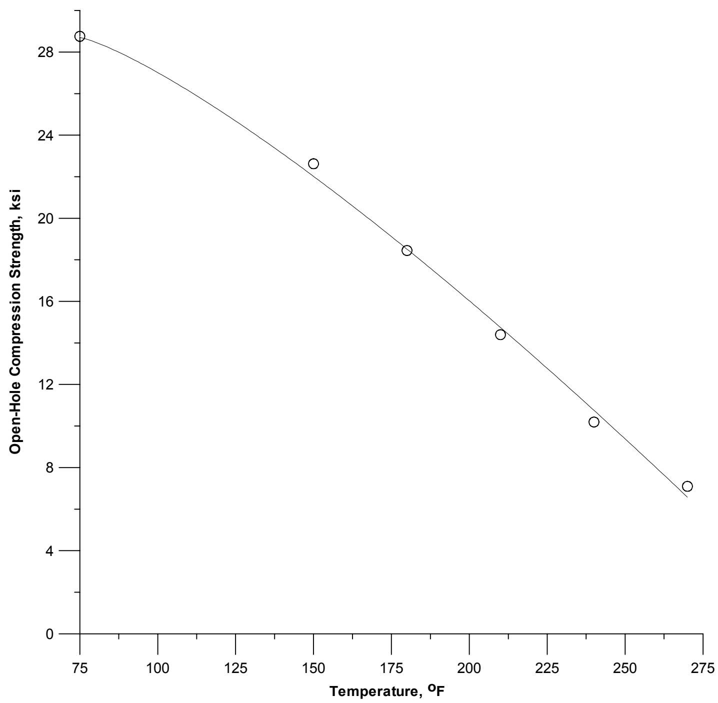

FIGURE 24. VARIATION OF OPEN-HOLE COMPRESSION STRENGTH VALUES AS A FUNCTION OF TEMPERATURE FOR NEWPORT 7781/NB321 GLASS FABRIC MATERIAL SYSTEM (85% RH)

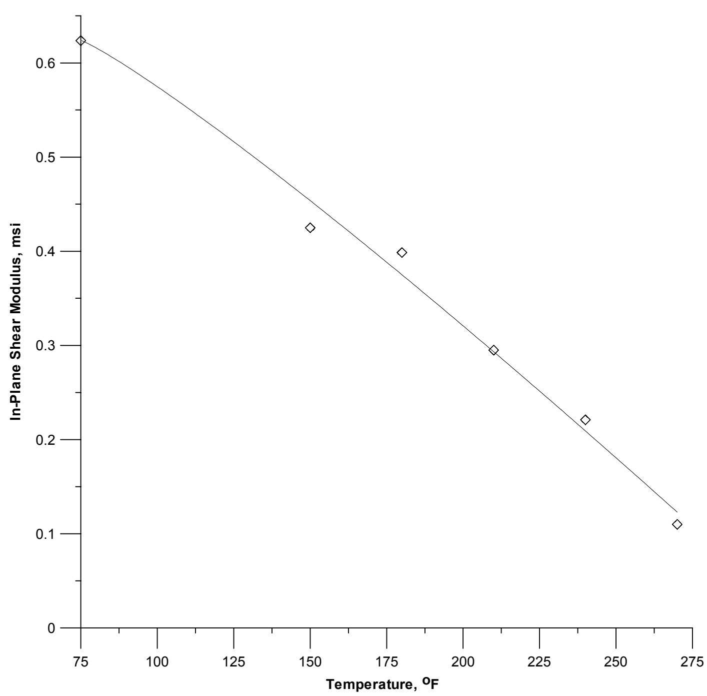

FIGURE 25. VARIATION OF IN-PLANE SHEAR MODULUS VALUES AS A FUNCTION OF TEMPERATURE FOR NEWPORT 7781/NB321 GLASS FABRIC MATERIAL SYSTEM (85% RH)

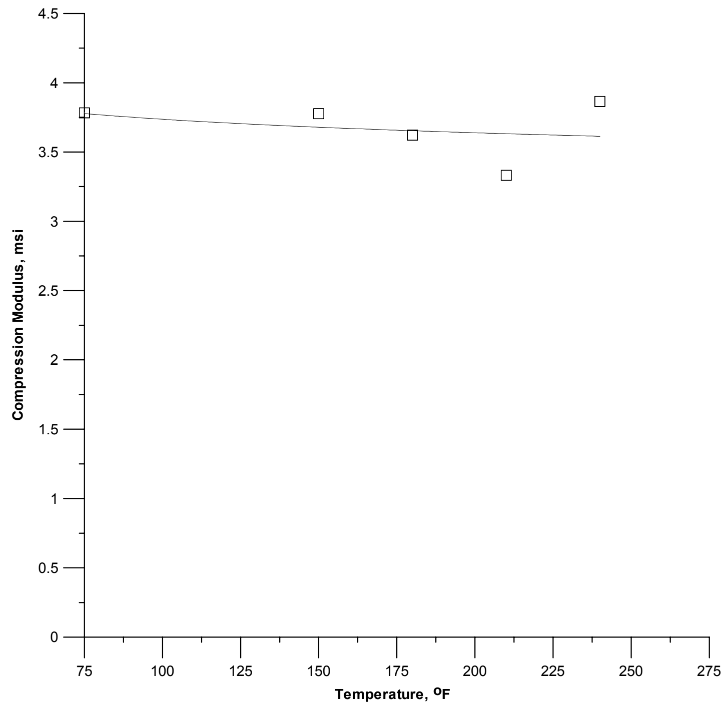

FIGURE 26. VARIATION OF COMPRESSION MODULUS VALUES AS A FUNCTION OF TEMPERATURE FOR NEWPORT 7781/NB321 GLASS FABRIC MATERIAL SYSTEM (85% RH)

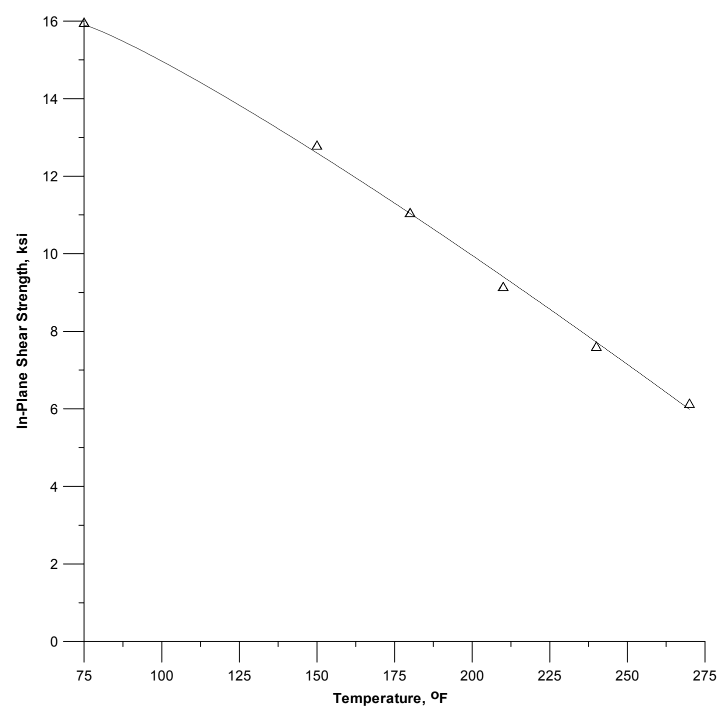

FIGURE 27. VARIATION OF IN-PLANE SHEAR STRENGTH VALUES AS A FUNCTION OF TEMPERATURE FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC MATERIAL SYSTEM (85% RH)

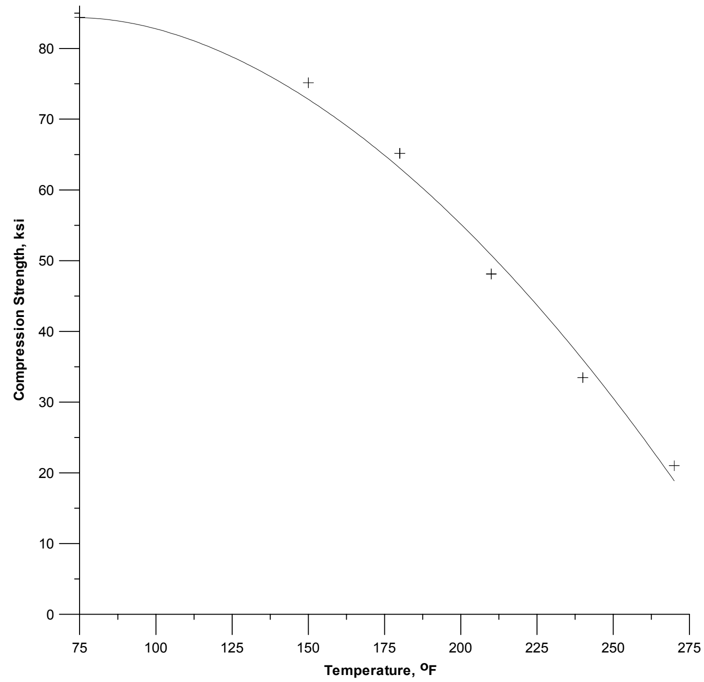

FIGURE 28. VARIATION OF COMPRESSION STRENGTH VALUES AS A FUNCTION OF TEMPERATURE FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC MATERIAL SYSTEM (85% RH)

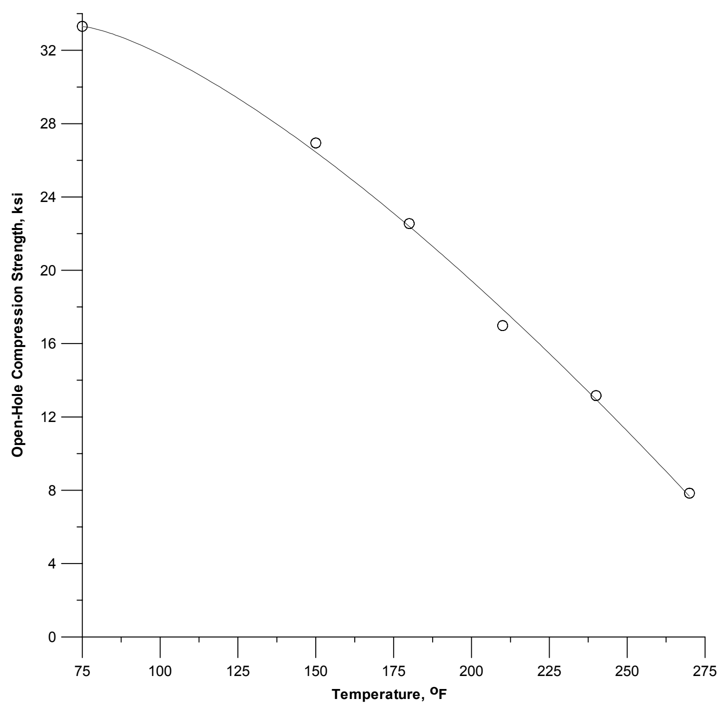

FIGURE 29. VARIATION OF OPEN-HOLE COMPRESSION STRENGTH VALUES AS A FUNCTION OF TEMPERATURE FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC MATERIAL SYSTEM (85% RH)

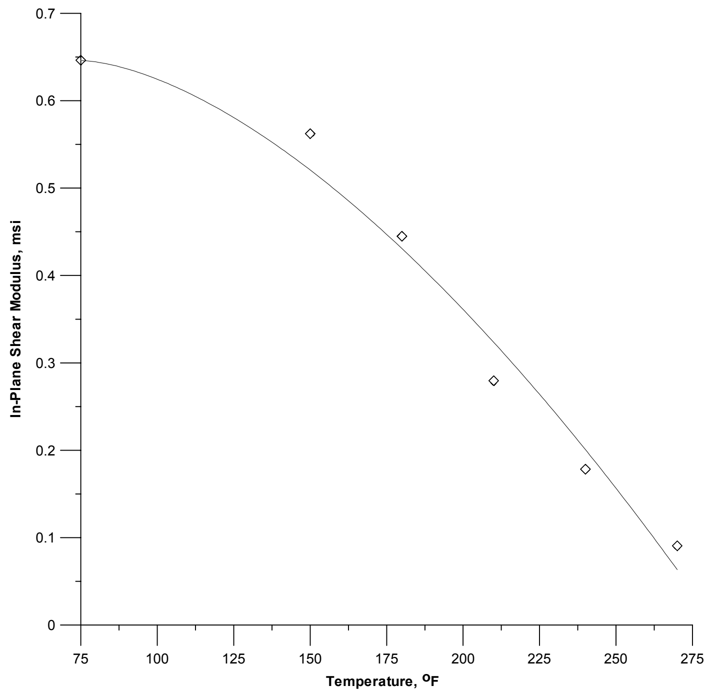

FIGURE 30. VARIATION OF IN-PLANE SHEAR MODULUS VALUES AS A FUNCTION OF TEMPERATURE FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC MATERIAL SYSTEM (85% RH)

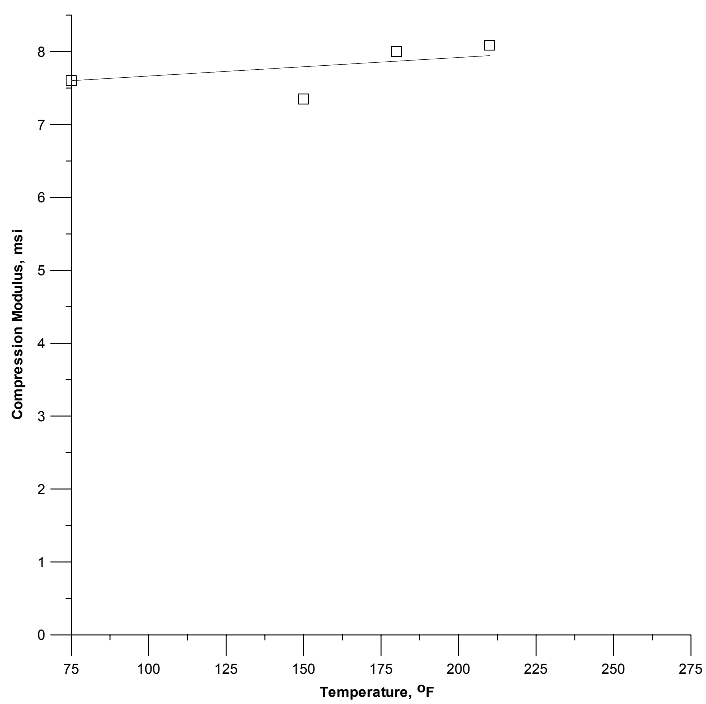

FIGURE 31. VARIATION OF COMPRESSION MODULUS VALUES AS A FUNCTION OF TEMPERATURE FOR FIBERCOTE E 765/T300 3KPW MATERIAL SYSTEM (85% RH)

3.3.2 DMA Test Results.

Tables 6 and 7 list the average glass transition temperature values obtained for both material systems conditioned at 85% relative humidity. The tables also show the T_g values corresponding to the different temperature ramp-up rates and specimen thickness values investigated.

TABLE 6. DYNAMIC MECHANICAL ANALYSIS TEST RESULTS FOR NEWPORT 7781/NB321 GLASS FABRIC MATERIAL SYSTEM (85% RH)

	T_g (Onset of Storage Modulus) (°F)	T_g (Peak of Tangent Delta) (°F)
Thick Specimens		
5°C/min	213	268
1°C/min	232	261
Thin Specimens		
5°C/min	211	261
1°C/min	219	268

TABLE 7. DYNAMIC MECHANICAL ANALYSIS TEST RESULTS FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC MATERIAL SYSTEM (85% RH)

	T_g (Onset of Storage Modulus) (°F)	T_g (Peak of Tangent Delta) (°F)
Thick Specimens		
5°C/min	228	272
1°C/min	221	261
Thin Specimens		
5°C/min	238	277
1°C/min	224	277

3.4 SUMMARY.

From figures 2 through 31, it can be concluded that matrix-dominated mechanical property values were markedly influenced by temperature and moisture. In fact, all of the mechanical properties considered, with the exception of the 0° (warp) compression modulus, exhibited a similar behavior with respect to temperature, regardless of the moisture level. This behavior could be described as a dramatic reduction in the mechanical property values as temperature was increased. “This degradation in composite properties is attributed to the plasticizing effect of moisture on the resin system which reduces the resin moduli over a wide range of temperature” [11].

This severe degradation of the mechanical properties followed either a linear or a parabolic trend. However “the knee” observed in the property degradation curves for materials cured at 350°F as shown in MIL-HDBK-17 (figure 1) was not evident for the two material systems considered which were cured at 270°F.

Curve fits of the experimental data were obtained using Grapher™ 2.0 software. A family of polynomial equations was used to generate these fits. All of these equations were of the same form described as follows:

$$Y = A + B * (X - 75)^C$$

Where A, B, and C are constants.

as previously mentioned, two specimen thickness values and two different temperature ramp-up rates were investigated for dynamic mechanical analysis. For both material systems, it was found that the glass transition temperature dropped drastically as the specimens' relative humidity level was increased.

In general, for the Newport 7781/NB321 thin specimens, the tests performed at 5°C/min yielded more conservative results than the ones performed at 1°C/min for the three relative humidity levels considered. The results did not follow any specific trend when the specimen thickness was varied and the difference in the results obtained did not exceed a value of 16°F.

For the FiberCote E 765/T300 3KPW material system, it was found that the results yielded by the thick specimens were more conservative than those obtained from the thin specimens, regardless of the test rate.

4. TEMPERATURE EFFECTS ON THE MECHANICAL PROPERTIES.

For comparison purposes, all of the mechanical test data was converted to percentage values where the property at room temperature corresponded to 100%, and the property values at the higher temperatures were converted to a percentage relative to the value at room temperature. For both material systems considered, three graphs were generated with each corresponding to one of the three relative humidity levels investigated. Each graph contains curve fits of the data obtained from the five mechanical tests performed: compression strength, compression modulus, in-plane shear strength, in-plane shear modulus, and open-hole compression strength. Figures 32 to 37 emphasize the behavior of the different properties and their sensitivity to temperature.

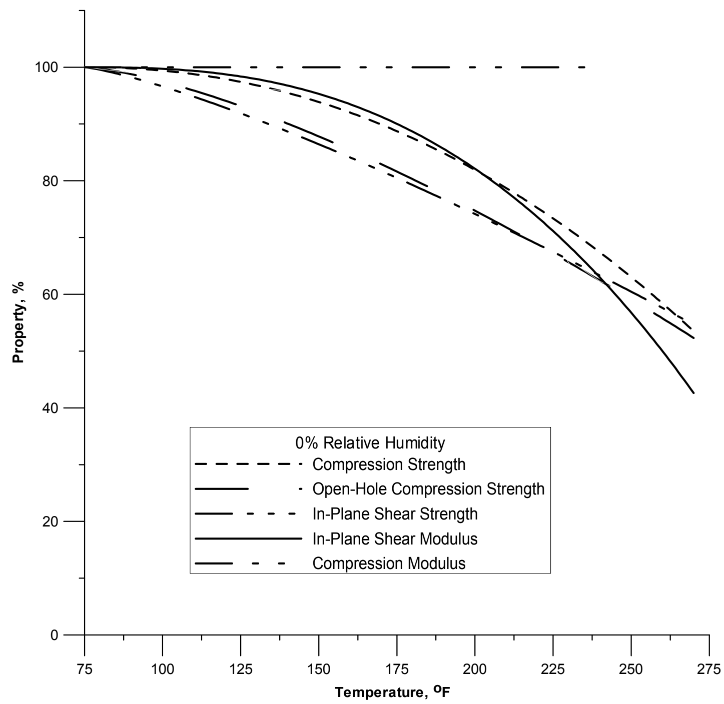

FIGURE 32. VARIATION IN THE PERCENTAGE OF MECHANICAL PROPERTIES AS A FUNCTION OF TEMPERATURE FOR NEWPORT 7781/NB321 GLASS FABRIC DRY SPECIMENS

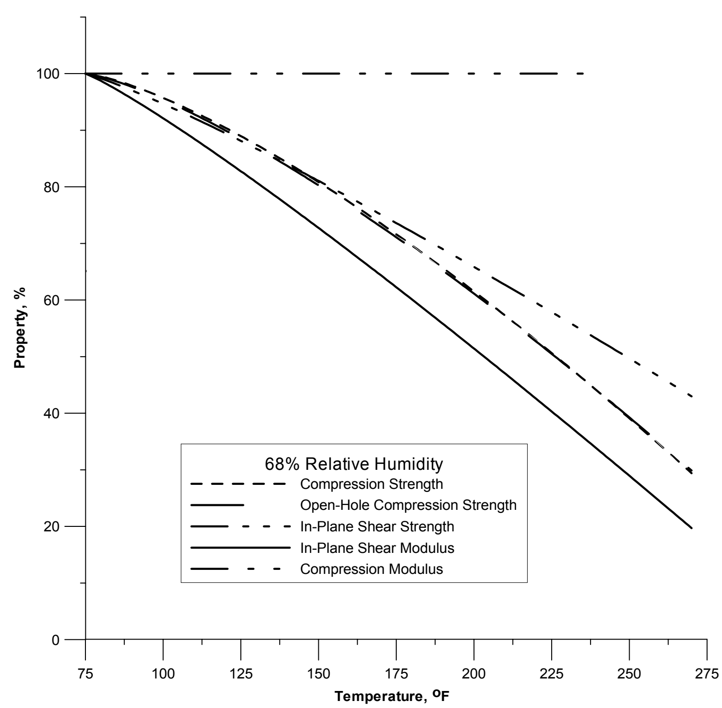

FIGURE 33. VARIATION IN THE PERCENTAGE OF MECHANICAL PROPERTIES AS A FUNCTION OF TEMPERATURE FOR NEWPORT 7781/NB321 GLASS FABRIC SPECIMENS CONDITIONED AT 68% RH

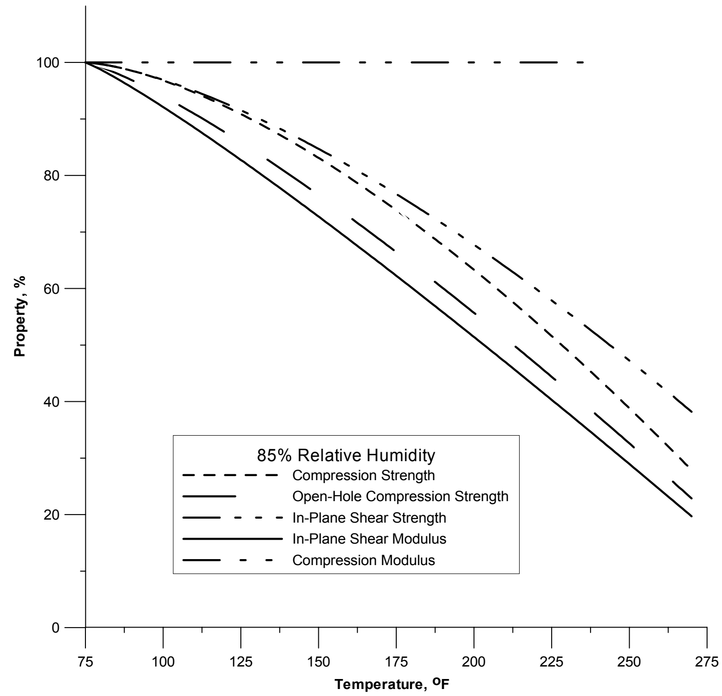

FIGURE 34. VARIATION IN THE PERCENTAGE OF MECHANICAL PROPERTIES AS A FUNCTION OF TEMPERATURE FOR NEWPORT 7781/NB321 GLASS FABRIC SPECIMENS CONDITIONED AT 85% RH

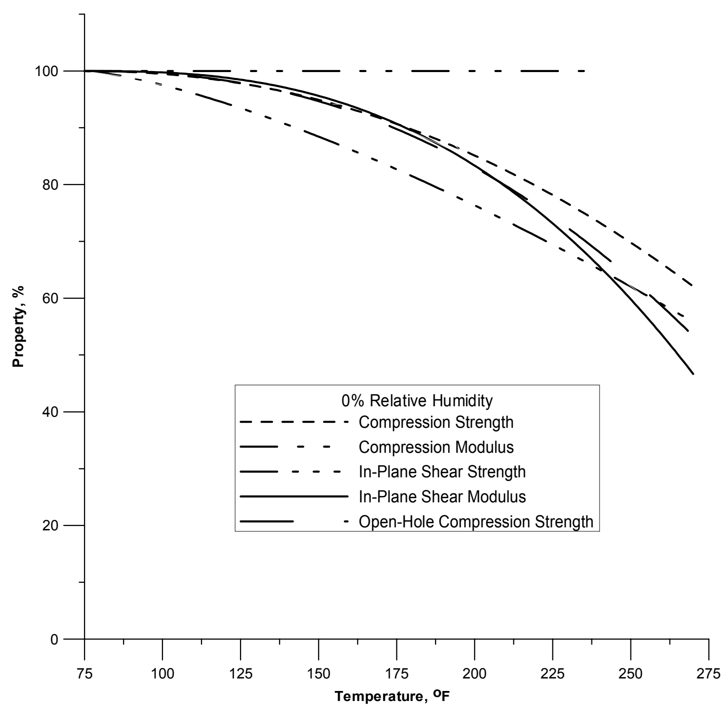

FIGURE 35. VARIATION IN THE PERCENTAGE OF MECHANICAL PROPERTIES AS A FUNCTION OF TEMPERATURE FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC SPECIMENS CONDITIONED AT 0% RH

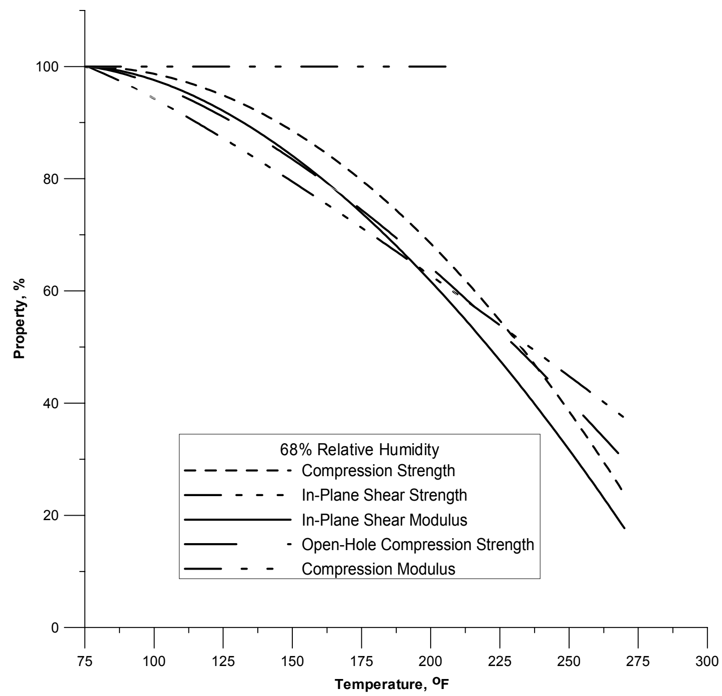

FIGURE 36. VARIATION IN THE PERCENTAGE OF MECHANICAL PROPERTIES AS A FUNCTION OF TEMPERATURE FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC SPECIMENS CONDITIONED AT 68% RH

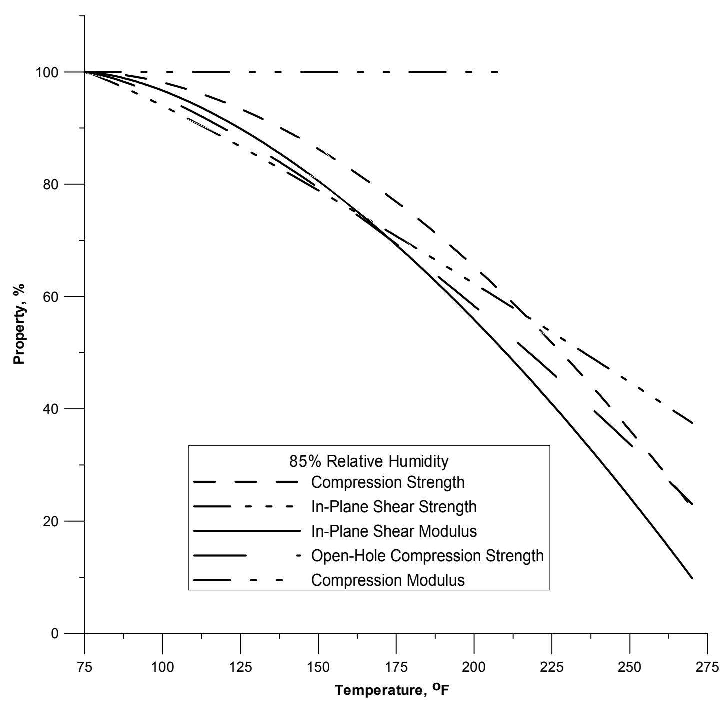

FIGURE 37. VARIATION IN THE PERCENTAGE OF MECHANICAL PROPERTIES AS A FUNCTION OF TEMPERATURE FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC SPECIMENS CONDITIONED AT 85% RH

From figures 32 to 37, it can be noted that for matrix-dominated properties (i.e., shear strength and modulus, open-hole compression strength, and compression strength), regardless of the relative humidity level, the degradation of the mechanical property values was significant as temperature was increased. Reductions of the property values as temperature was increased did not occur for (0° Warp) compression modulus, a fiber-dominated property.

From the same figures, it can also be seen that for both material systems the most sensitive property to temperature at all relative humidity levels considered was in-plane shear modulus. In fact, at 0% RH, the Newport 7781/NB321 material system retained about 40% of its room temperature in-plane shear modulus value at 270°F. At the same temperature and at 68% and 85% RH levels, the material system retained about 20% of its in-plane shear modulus value at room temperature. At 0% RH, the FiberCote E 765/T300 3KPW carbon fabric material system retained about 46% of its room temperature in-plane shear modulus value at 270°F. At the same temperature and at 68% RH level, the material system retained about 18% of its in-plane shear modulus value at room temperature, and finally, at 85% RH it retained only 9% of its value at room temperature.

The least sensitive property to temperature was found to be compression modulus. It should be noted that at the highest temperatures, it was not possible to get reliable values for compression modulus due to the fact that the tested specimens failed before enough strain data, corresponding to the range of 0.001-0.003 in/in, was obtained.

5. MOISTURE EFFECTS ON THE MECHANICAL PROPERTIES.

To determine the effect of moisture on the mechanical properties, a series of graphs were plotted showing the variation in the percentage of the mechanical property values with respect to relative humidity level for the six different temperatures considered. Each of the graphs in figures 38 to 47 show a given test method and contains six curves, each representing a given temperature. All property values are normalized to 0% RH value in terms of percentage. Thus, the 0% RH values are shown as 100%. For example, in figure 38 the actual dry in-plane shear strength value obtained at room temperature was different from the one obtained at 270°F, however, for the purpose of comparison these values were normalized to 100% so that the effect of moisture could be separated.

From figures 38 to 42, it can be seen that overall, as the relative humidity level was increased, there was a definite reduction in the mechanical property values for the Newport 7781/NB321 material system. The effect of moisture was enhanced as the temperature was increased, and the most sensitive property to moisture was found to be in-plane shear modulus. In fact, for tests performed at room temperature, the in-plane shear modulus value obtained at 85% RH retained about 96% of its value at 0% RH. This 4% reduction in the property was mainly due to the increase in the relative humidity level. At 270°F, however, the in-plane shear modulus value obtained at 85% RH retained only 42% of its value at 0% RH. This 58% reduction in the property value is a combination of the effect of moisture enhanced by the added effect of temperature.

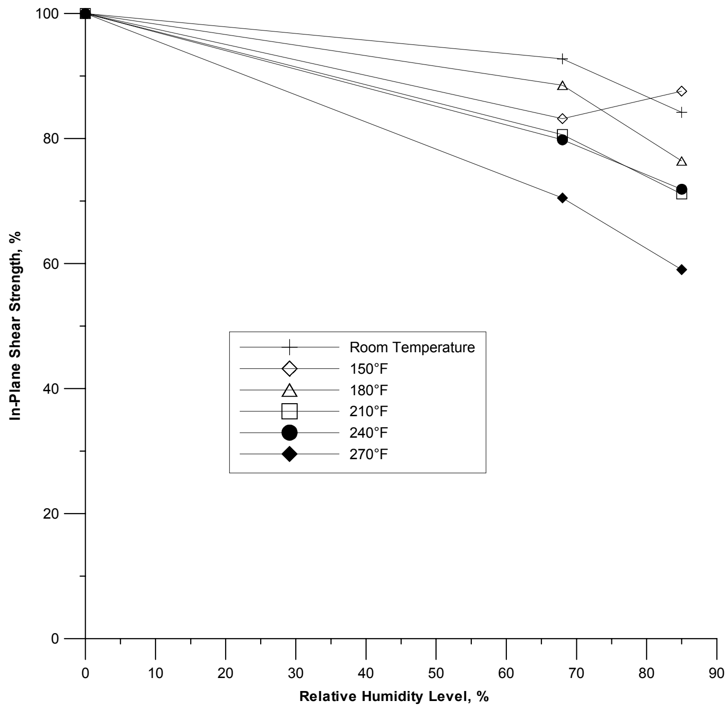

FIGURE 38. VARIATION IN THE PERCENTAGE OF IN-PLANE SHEAR STRENGTH VALUES AS A FUNCTION OF RELATIVE HUMIDITY LEVEL FOR THE SIX TEMPERATURES CONSIDERED FOR NEWPORT 7781/NB321 MATERIAL SYSTEM

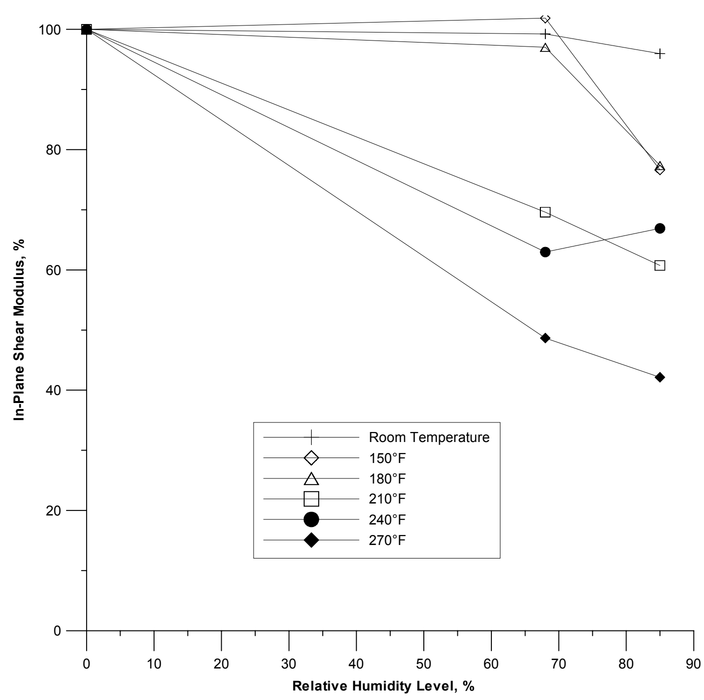

FIGURE 39. VARIATION IN THE PERCENTAGE OF IN-PLANE SHEAR MODULUS VALUES AS A FUNCTION OF RELATIVE HUMIDITY LEVEL FOR THE SIX TEMPERATURES CONSIDERED FOR NEWPORT 7781/NB321 MATERIAL SYSTEM

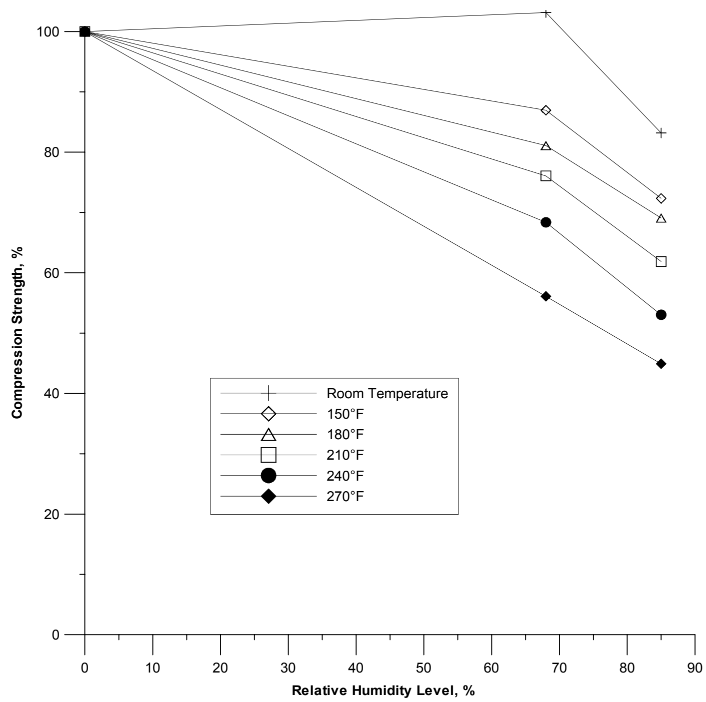

FIGURE 40. VARIATION IN THE PERCENTAGE OF COMPRESSION STRENGTH VALUES AS A FUNCTION OF RELATIVE HUMIDITY LEVEL FOR THE SIX TEMPERATURES CONSIDERED FOR NEWPORT 7781/NB321 MATERIAL SYSTEM

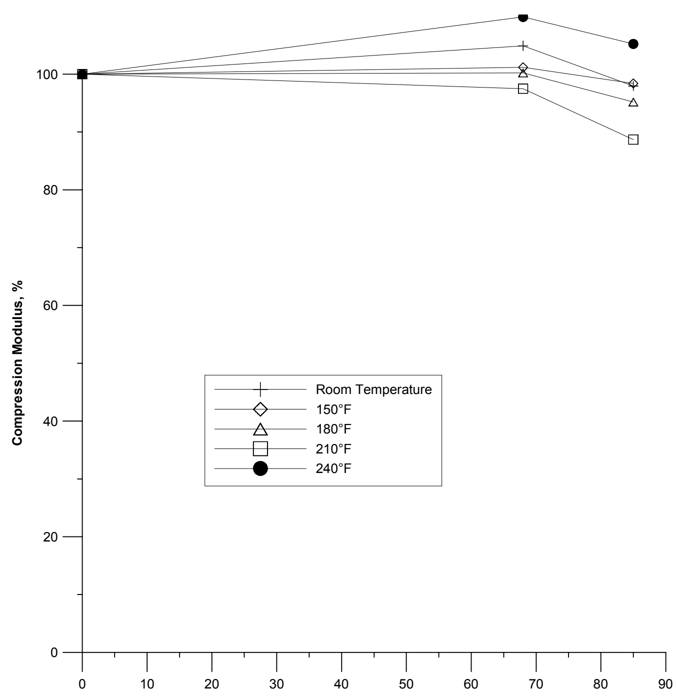

FIGURE 41. VARIATION IN THE PERCENTAGE OF COMPRESSION MODULUS VALUES AS A FUNCTION OF RELATIVE HUMIDITY LEVEL FOR THE TEMPERATURES CONSIDERED FOR NEWPORT 7781/NB321 MATERIAL SYSTEM

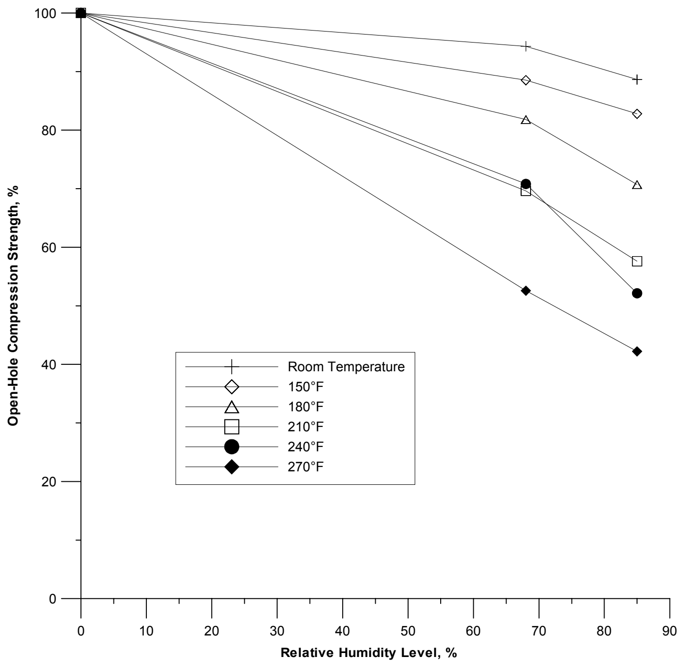

FIGURE 42. VARIATION IN THE PERCENTAGE OF OPEN-HOLE COMPRESSION STRENGTH VALUES AS A FUNCTION OF RELATIVE HUMIDITY LEVEL FOR THE SIX TEMPERATURES CONSIDERED FOR NEWPORT 7781/NB321 MATERIAL SYSTEM

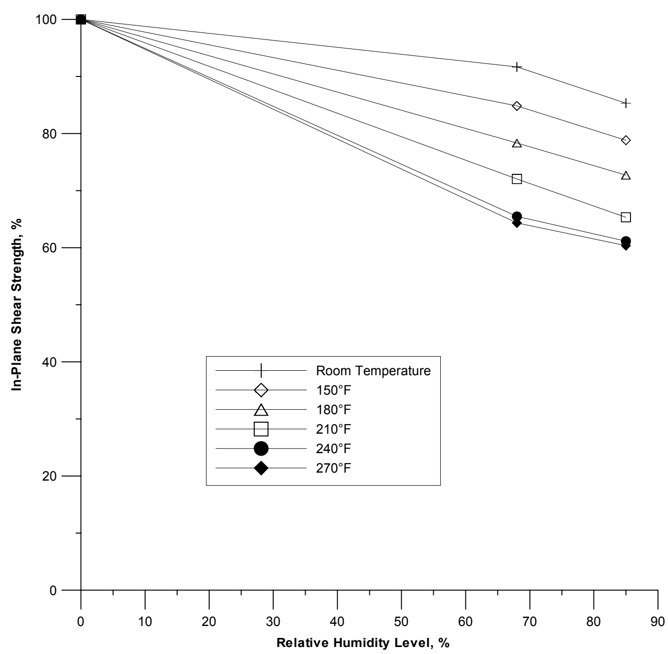

FIGURE 43. VARIATION IN THE PERCENTAGE OF IN-PLANE SHEAR STRENGTH VALUES AS A FUNCTION OF RELATIVE HUMIDITY LEVEL FOR THE SIX TEMPERATURES CONSIDERED FOR FIBERCOTE E 765/T300 3KPW MATERIAL SYSTEM

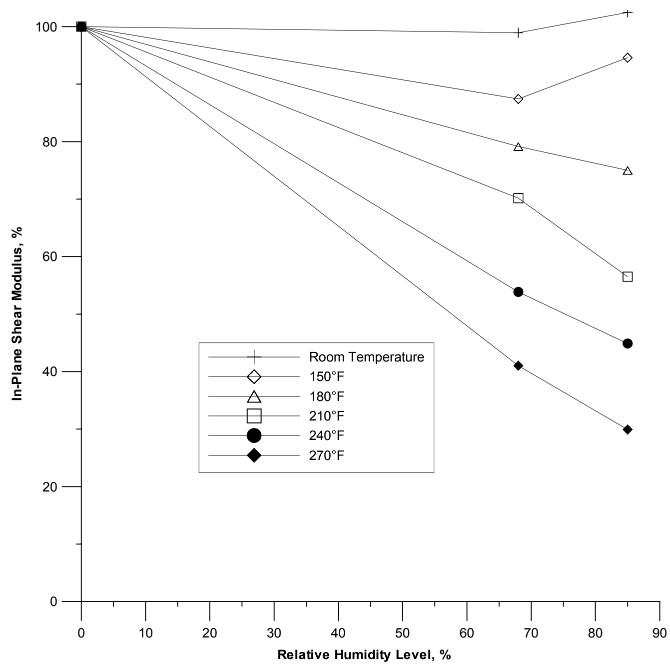

FIGURE 44. VARIATION IN THE PERCENTAGE OF IN-PLANE SHEAR MODULUS VALUES AS A FUNCTION OF RELATIVE HUMIDITY LEVEL FOR THE SIX TEMPERATURES CONSIDERED FOR FIBERCOTE E 765/T300 3KPW MATERIAL SYSTEM

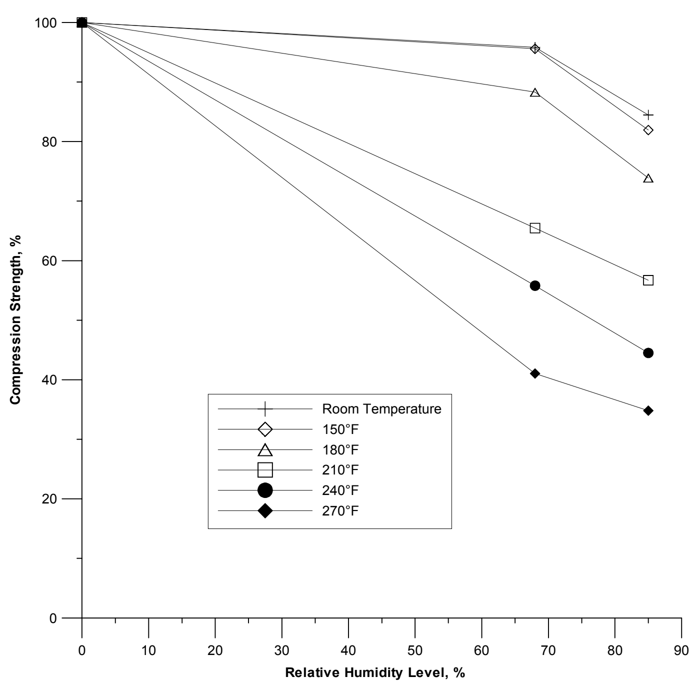

FIGURE 45. VARIATION IN THE PERCENTAGE OF COMPRESSION TRENGTH VALUES AS A FUNCTION OF RELATIVE HUMIDITY LEVEL FOR THE SIX TEMPERATURES CONSIDERED FOR FIBERCOTE E 765/T300 3KPW MATERIAL SYSTEM

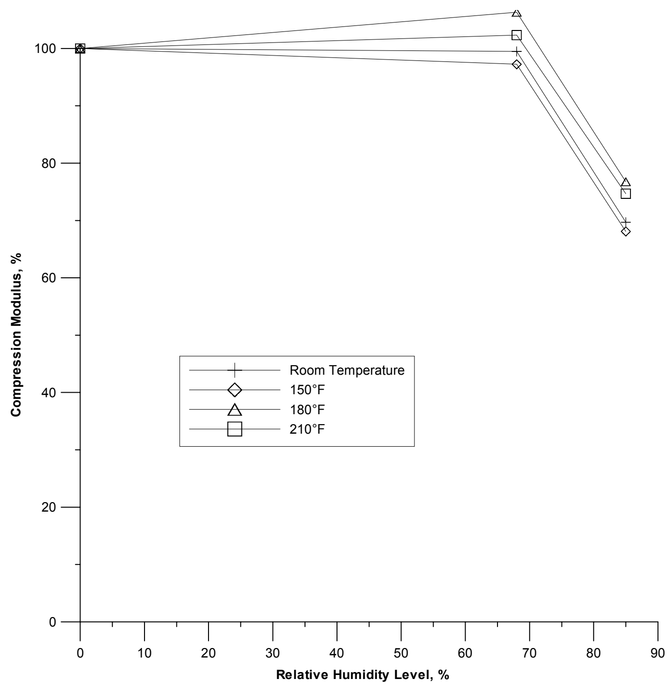

FIGURE 46. VARIATION IN THE PERCENTAGE OF COMPRESSION MODULUS VALUES AS A FUNCTION OF RELATIVE HUMIDITY LEVEL FOR THE FOUR TEMPERATURES CONSIDERED FOR FIBERCOTE E 765/T300 3KPW MATERIAL SYSTEM

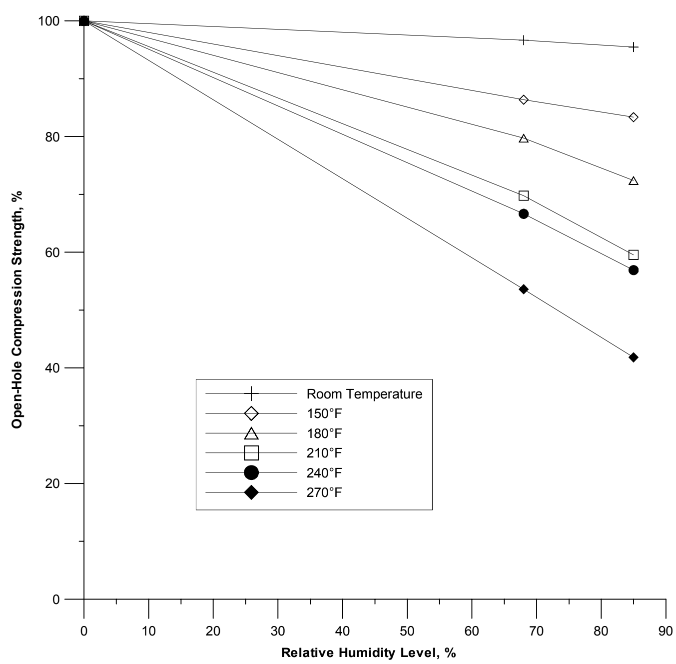

FIGURE 47. VARIATION IN THE PERCENTAGE OF OPEN-HOLE COMPRESSION STRENGTH VALUES AS A FUNCTION OF RELATIVE HUMIDITY LEVEL FOR THE SIX TEMPERATURES CONSIDERED FOR FIBERCOTE E 765/T300 3KPW MATERIAL SYSTEM

As shown in figures 43 to 47, there was a significant degradation of mechanical properties as the relative humidity level was increased for the FiberCote E 765/T300 3KPW material system. The property reduction became even more severe as the temperature was increased. The most sensitive property to moisture was found to be in-plane shear modulus. In fact, at room temperature, the in-plane shear modulus property value obtained did not vary significantly as the moisture level was increased. At 270°F, however, the in-plane shear modulus value obtained at 85% RH retained only about 30% of its value at 0% RH. This 70% reduction in the property value is a combination of the effect of moisture enhanced by the added effects of temperature.

As was previously mentioned, increasing the moisture level yielded a severe reduction in the mechanical property values for both material systems considered. Similarly, a significant drop in the glass transition values occurred when the moisture level was increased as is illustrated in figures 48 to 51.

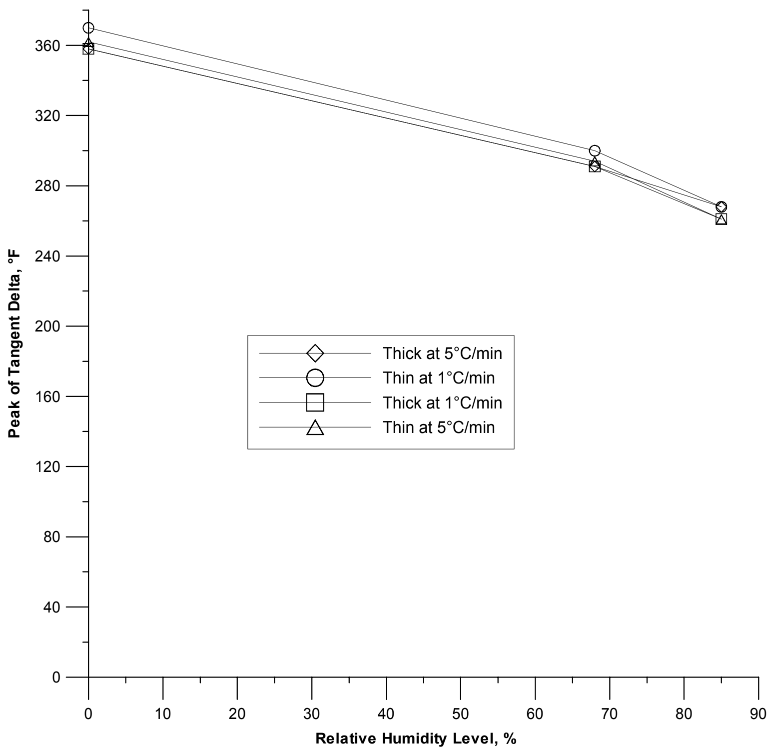

FIGURE 48. VARIATION IN THE PEAK OF TANGENT DELTA VALUES AS A FUNCTION OF RELATIVE HUMIDITY LEVEL FOR NEWPORT 7781/NB321 MATERIAL SYSTEM

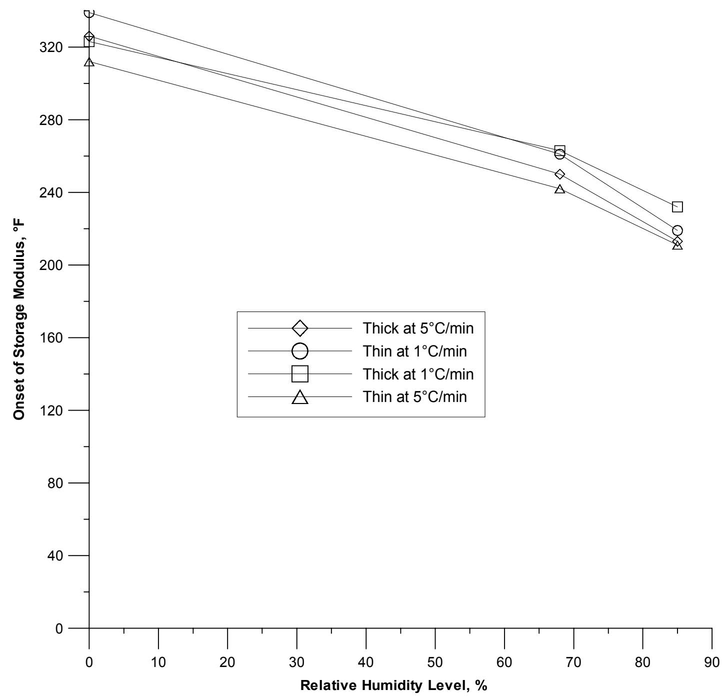

FIGURE 49. VARIATION IN THE ONSET OF STORAGE MODULUS VALUES AS A FUNCTION OF RELATIVE HUMIDITY LEVEL FOR NEWPORT 7781/NB321 MATERIAL SYSTEM

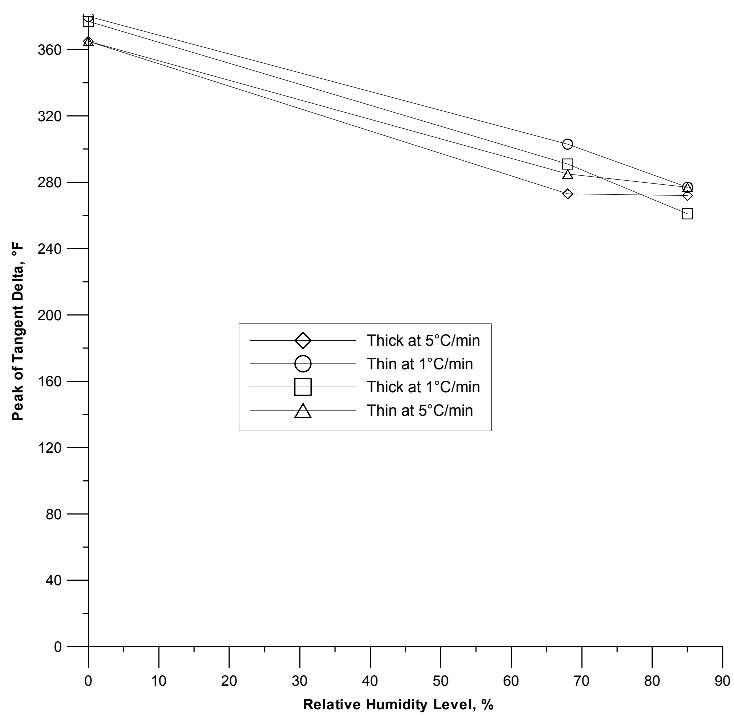

FIGURE 50. VARIATION IN THE PEAK OF TANGENT DELTA VALUES AS A FUNCTION OF RELATIVE HUMIDITY LEVEL FOR FIBERCOTE E 765/T300 3KPW MATERIAL SYSTEM

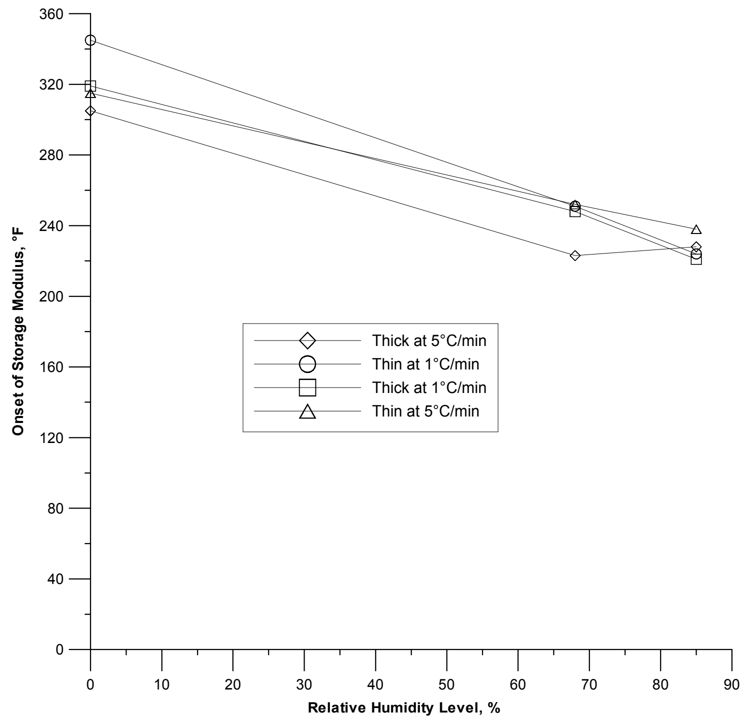

FIGURE 51. VARIATION IN THE ONSET OF STORAGE MODULUS VALUES AS A FUNCTION OF RELATIVE HUMIDITY LEVEL FOR FIBERCOTE E 765/T300 3KPW MATERIAL SYSTEM

6. GLASS TRANSITION TEMPERATURE AND 50°F MARGIN.

In practice, the maximum operating temperature at which a composite material is used in aircraft structure has been determined as 50°F below its most conservative T_g value. This practice avoided the use of a material system at temperatures where a significant degradation of its mechanical properties occurs. Figures 52 to 57 show the operational limit of both material systems considered, based on the most conservative T_g value obtained, which corresponds to the onset of the storage modulus value obtained at 85% RH. In this study two different thicknesses and heat-up rates were used to determine T_g. As there were no clear trends and the test specifications are not clear which parameters should be used, average T_g as determined by storage modules of the four tests were used in this calculation. For the Newport 7781/NB321 glass fabric specimens, the MOL was found to be 169°F, whereas for the FiberCote E765/T300 3KPW, the MOL was found to be 179°F, which is almost equal to 180°F, a typical operating temperature. If 68% RH was chosen as the more reasonable RH level, the MOL would then be 254°F for Newport 7781/NB321 and 245°F for the FiberCote E765/T300 3KPW.

From figure 52, it can be noted that up to the MOL predicted by the 50°F margin at 85% RH, the most sensitive property to temperature was in-plane shear strength. The least sensitive property to temperature was compression modulus. At the MOL, in-plane shear strength retained about 81% its value at room temperature, whereas compression modulus retained about 100% of its room temperature value.

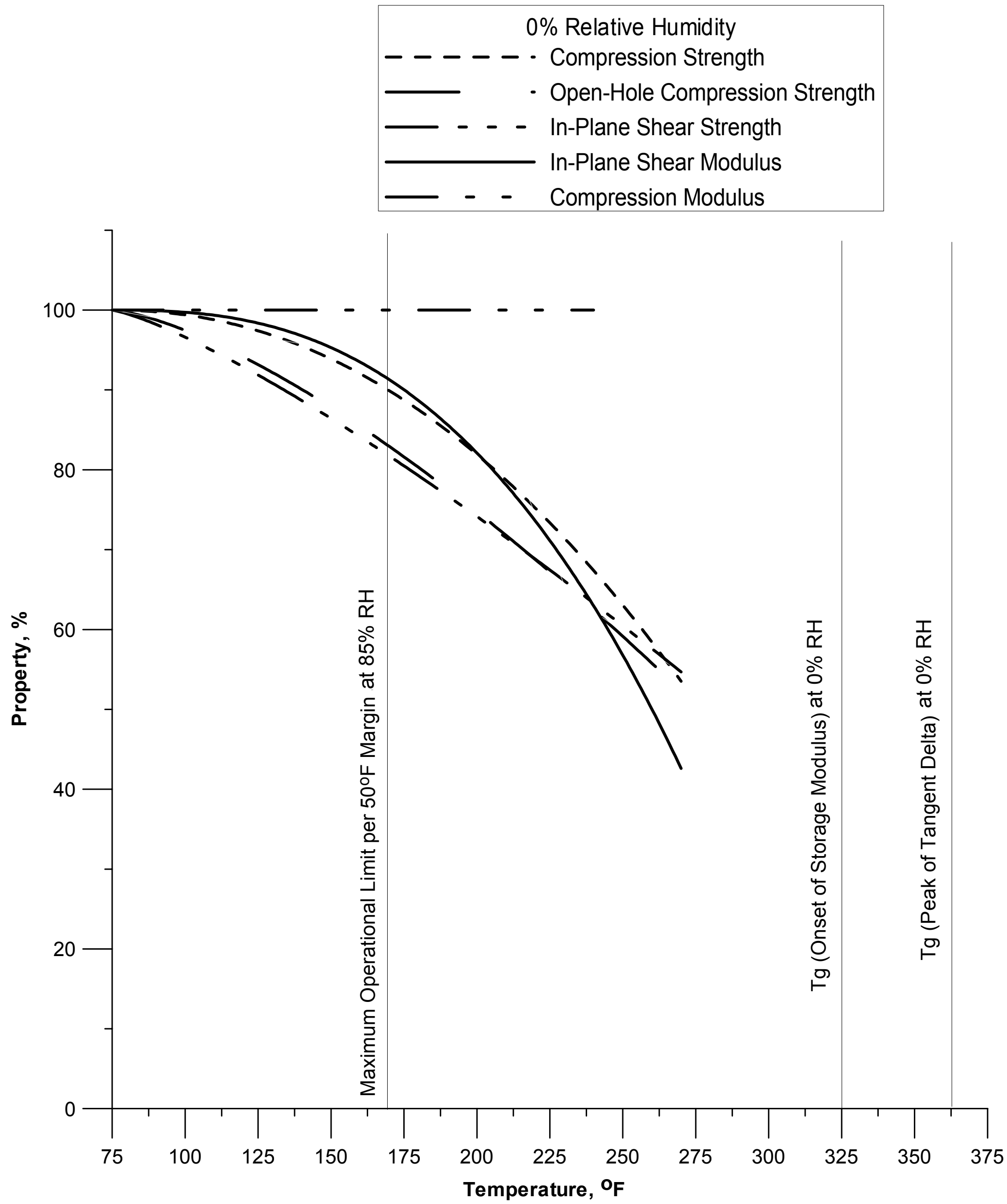

FIGURE 52. MATERIAL OPERATIONAL LIMIT AS PREDICTED BY THE 50°F MARGIN METHOD AT 85% RH BASED ON THE RESULTS FOR NEWPORT 7781/NB321 GLASS FABRIC DRY SPECIMENS

From figure 53, it can be seen that as the humidity level was increased to 68%, the most sensitive property to temperature at the MOL was in-plane shear modulus. The compressive properties (i.e., compression strength and open-hole compression strength) behaved in a similar way to temperature/moisture and were the next most sensitive properties to temperature after in-plane shear modulus. In-plane shear strength followed, and finally, the least sensitive mechanical property was compression modulus. At the MOL, the most critical mechanical property retained 65% of its value at room temperature, whereas the least critical property remained equal to its value at room temperature.

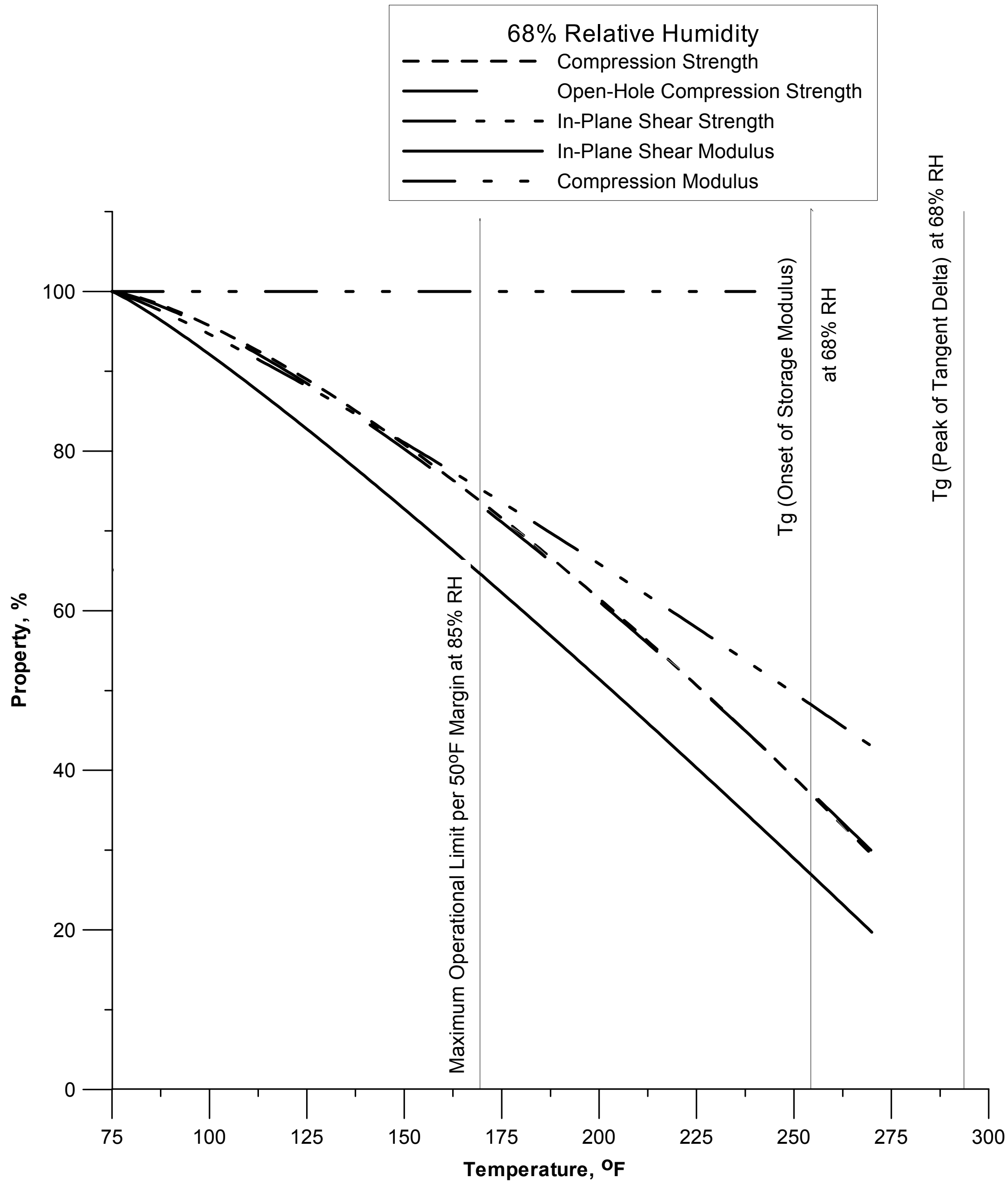

FIGURE 53. MATERIAL OPERATIONAL LIMIT AS PREDICTED BY THE 50°F MARGIN METHOD AT 85% RH BASED ON THE RESULTS FOR NEWPORT 7781/NB321 GLASS FABRIC SPECIMENS CONDITIONED AT 68% RH

As the humidity level was increased to 85% (see figure 52), in-plane shear modulus remained the most sensitive property to temperature at the MOL. The least sensitive property to temperature was compression modulus. At the MOL, the most sensitive property retained 64% of its value at room temperature, whereas the least critical property remained unchanged. Compression modulus was found to be the least sensitive property to temperature at the MOL.

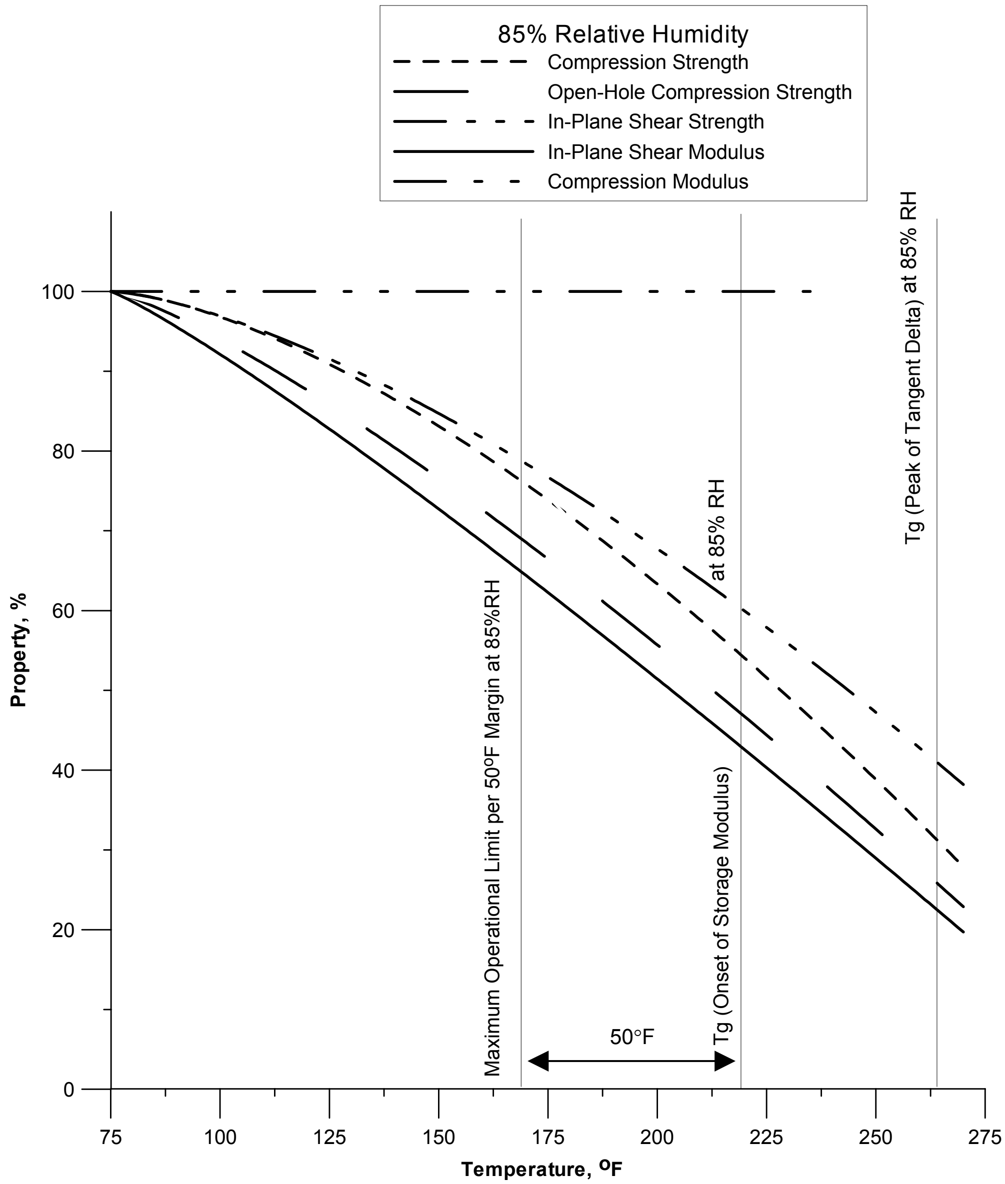

FIGURE 54. MATERIAL OPERATIONAL LIMIT AS PREDICTED BY THE 50°F MARGIN METHOD AT 85% RH BASED ON THE RESULTS FOR NEWPORT 7781/NB321 GLASS FABRIC SPECIMENS CONDITIONED AT 85% RH

Moisture increased the degradation of the material properties due to temperature. In fact, at 0% RH, the Newport 7781/NB321 material system retained about 40% of its room temperature in-plane shear modulus value at 270°F. At the same temperature and at 68% and 85% RH levels, the material system retained about 20% of its in-plane shear modulus value at room temperature.

From figure 55, it can be seen that at the MOL, the most sensitive property to temperature for the FiberCote E 765/T300 3KPW material system was in-plane shear strength, whereas the least sensitive property to temperature was compression modulus. At the MOL, in-plane shear strength retained about 82% of its value at room temperature, whereas compression modulus hardly varied from its value at room temperature.

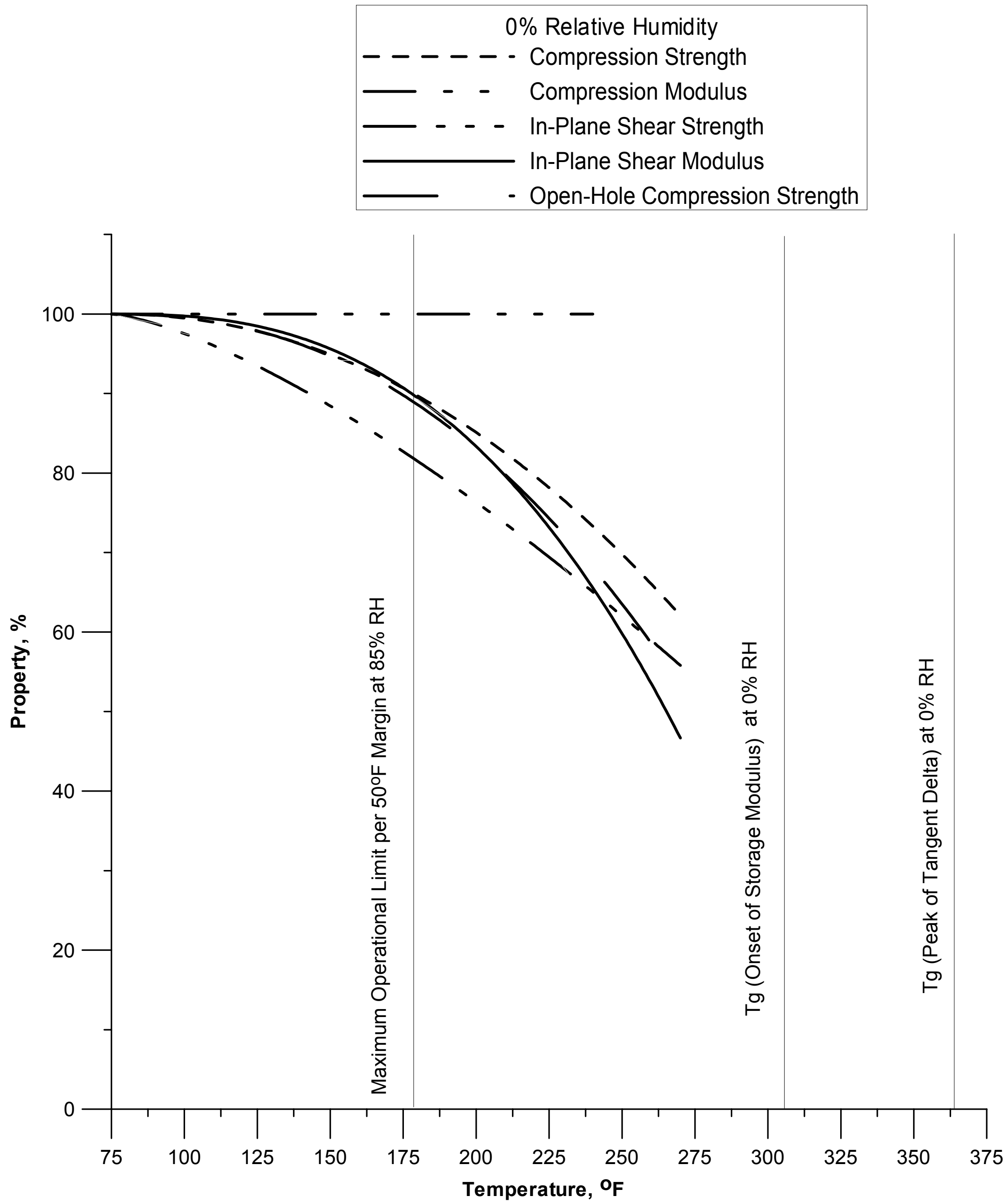

FIGURE 55. MATERIAL OPERATIONAL LIMIT AS PREDICTED BY THE 50°F MARGIN METHOD BASED ON THE RESULTS FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC DRY SPECIMENS

As shown in figure 56, at a moisture level of 68%, the most sensitive property to temperature was in-plane shear strength. The least sensitive property to moisture and temperature was compression modulus. At the MOL, in-plane shear strength was about 70% of its value at room temperature, whereas compression modulus remained the same as its initial value.

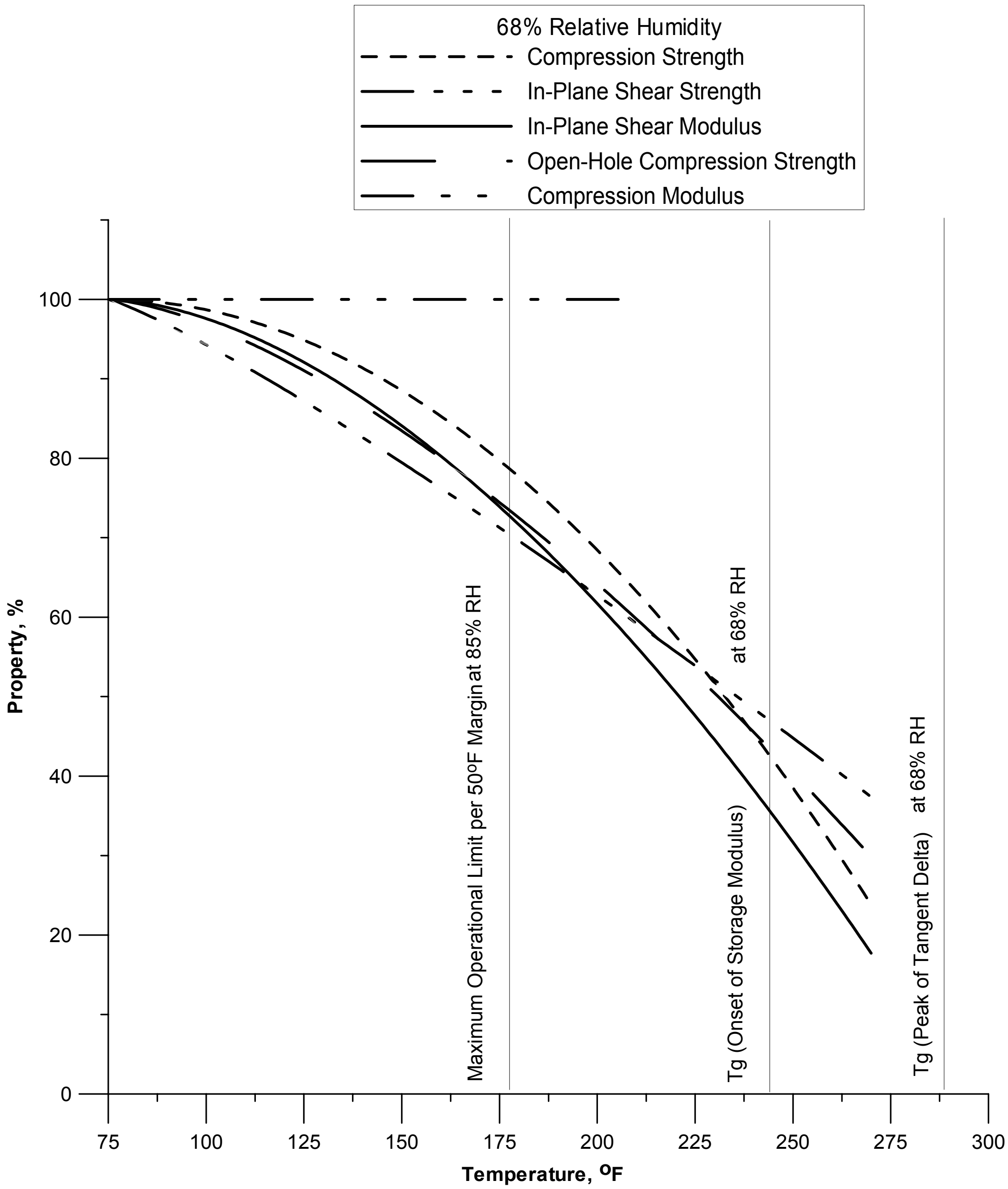

FIGURE 56. MATERIAL OPERATIONAL LIMIT AS PREDICTED BY THE 50°F MARGIN METHOD BASED ON THE RESULTS FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC SPECIMENS CONDITIONED AT 68% RH

As the humidity level was increased to 85% (see figure 57), it can be seen that at the MOL, the most sensitive property to temperature was in-plane shear modulus. The least sensitive property to temperature was compression modulus. At the MOL, the most sensitive property retained about 68% of its value at room temperature, whereas the least sensitive property remained unchanged.

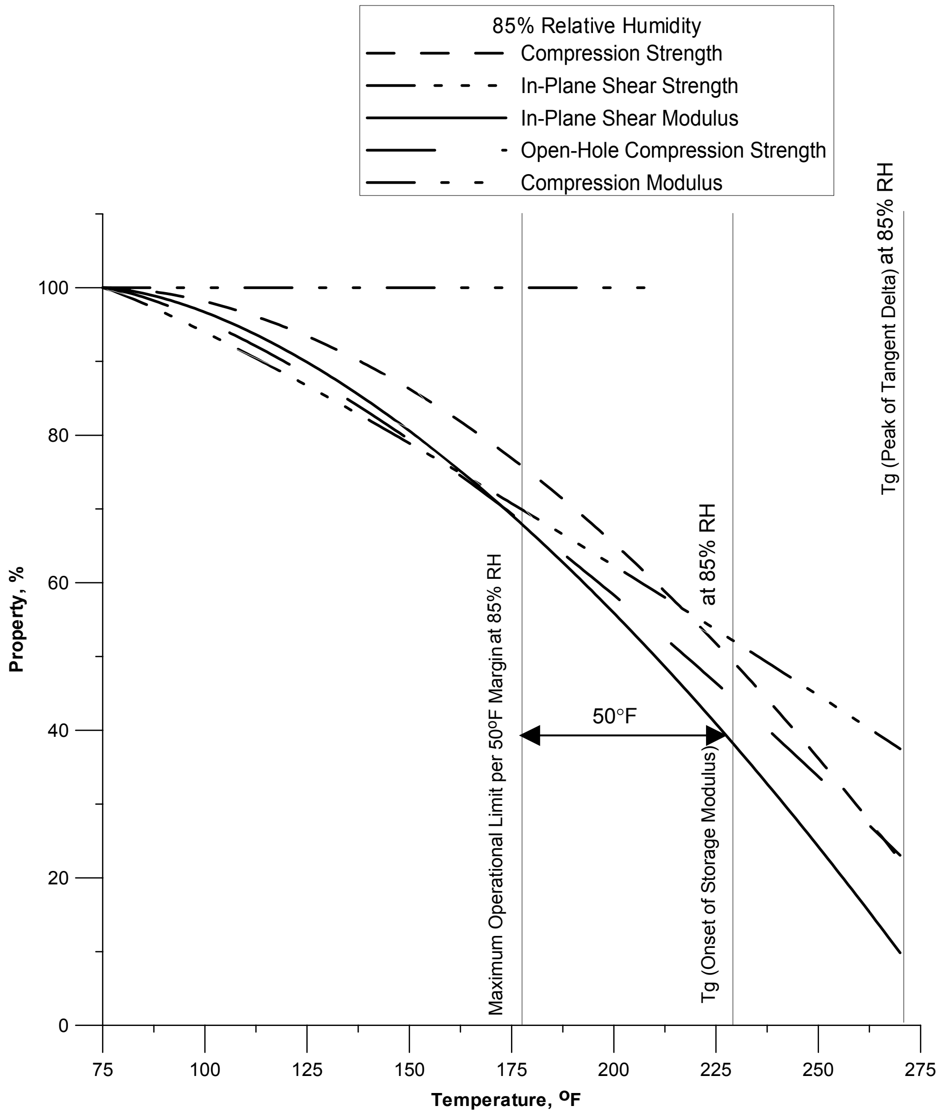

FIGURE 57. MATERIAL OPERATIONAL LIMIT AS PREDICTED BY THE 50°F MARGIN BASED ON THE RESULTS FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC SPECIMENS CONDITIONED AT 85% RH

As was noted for the Newport 7781/NB321 glass fabric material system, moisture increased the degradation of the material properties due to temperature. In fact, at 0% RH, the FiberCote E765/T300 3KPW carbon fabric material system retained about 46% of its room temperature in-plane shear modulus value at 270°F. At the same temperature and at a 68% RH level, the material system retained about 18% of its in-plane shear modulus value at room temperature, and finally, at 85% RH it retained only 9% of its value at room temperature.

7. TWO-THIRD RETENTION METHOD.

In order to assess the validity of the 50°F "rule," the 2/3 retention method illustrated in figures 58 to 81 was considered. For this purpose, a hypothetical MOL value was chosen. This value corresponds to 180°F, a typical maximum operating temperature.

The 2/3 retention method is based on mechanical properties unlike the 50°F rule, which is based on T_g values obtained from DMA tests. This method evaluates the drop in the mechanical properties between 180°F and 230°F (50°F above 180°F). A drop of 1/3 or less is acceptable, suggesting that the value of the MOL used was appropriate. A drop of more than 1/3 of the mechanical property is not acceptable, suggesting that the MOL value used was not appropriate since significant property degradations were induced. Because the 2/3 retention method is based on mechanical properties collected on a larger scale, an argument can be made that it has more meaning to structural performance.

As shown on figures 58 to 65, the mechanical property reduction between the MOL and 230°F is less than 1/3, suggesting that all the properties satisfy the 2/3 rule at 0% RH for both material systems considered.

As shown on figures 66 to 73, the property reduction between the MOL and 230°F is more than 1/3 for in-plane shear modulus for both material systems at 68% RH. All the other properties considered, however, satisfy the 2/3 rule.

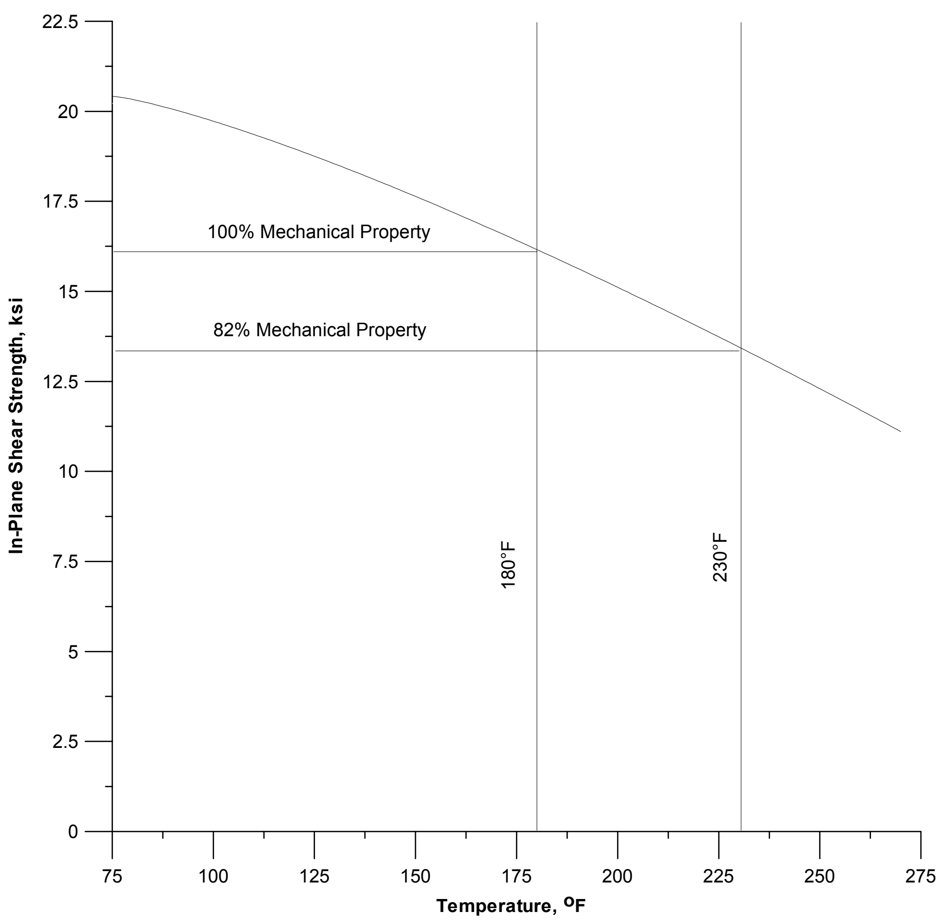

FIGURE 58. TWO-THIRD RETENTION METHOD SHOWN FOR NEWPORT 7781/NB321 GLASS FABRIC IN-PLANE SHEAR STRENGTH (0% RH)

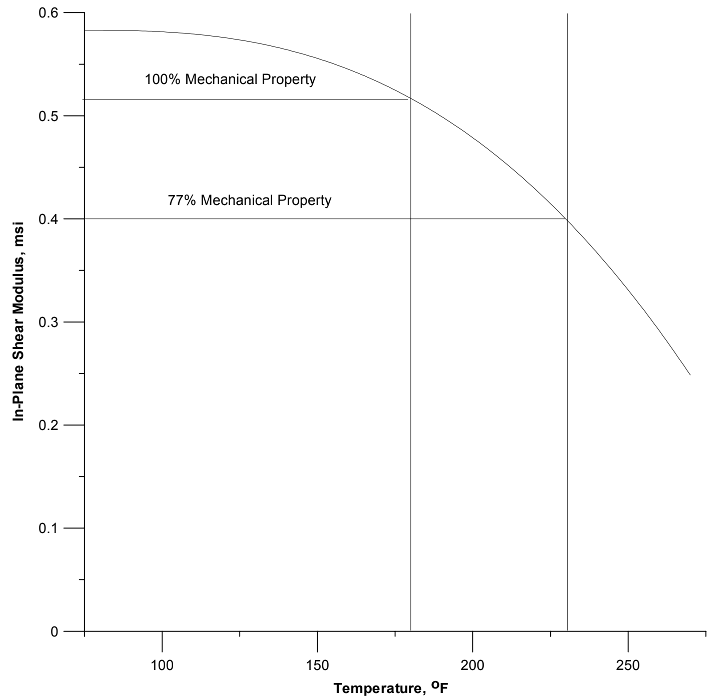

FIGURE 59. TWO-THIRD RETENTION METHOD SHOWN FOR NEWPORT 7781/NB321 GLASS FABRIC IN-PLANE SHEAR MODULUS (0% RH)

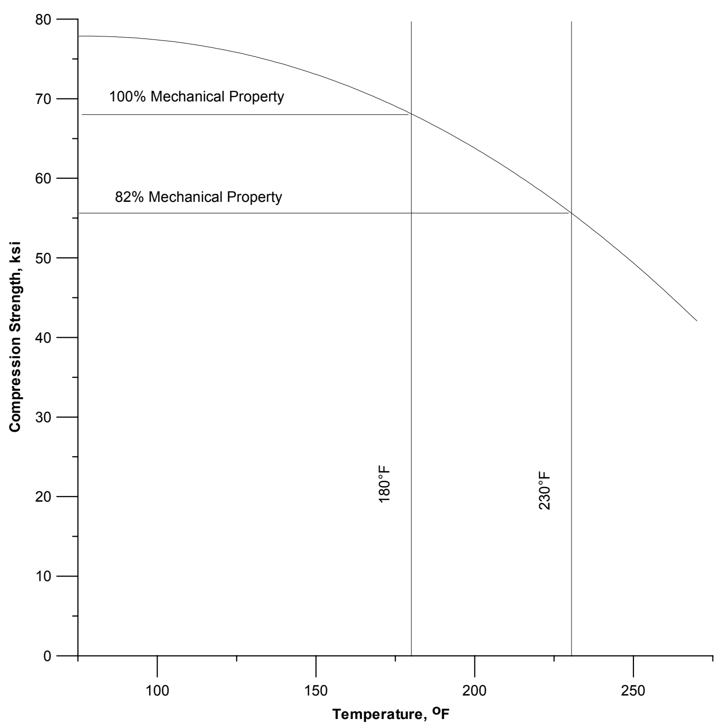

FIGURE 60. TWO-THIRD RETENTION METHOD SHOWN FOR NEWPORT 7781/NB321 GLASS FABRIC COMPRESSION STRENGTH (0% RH)

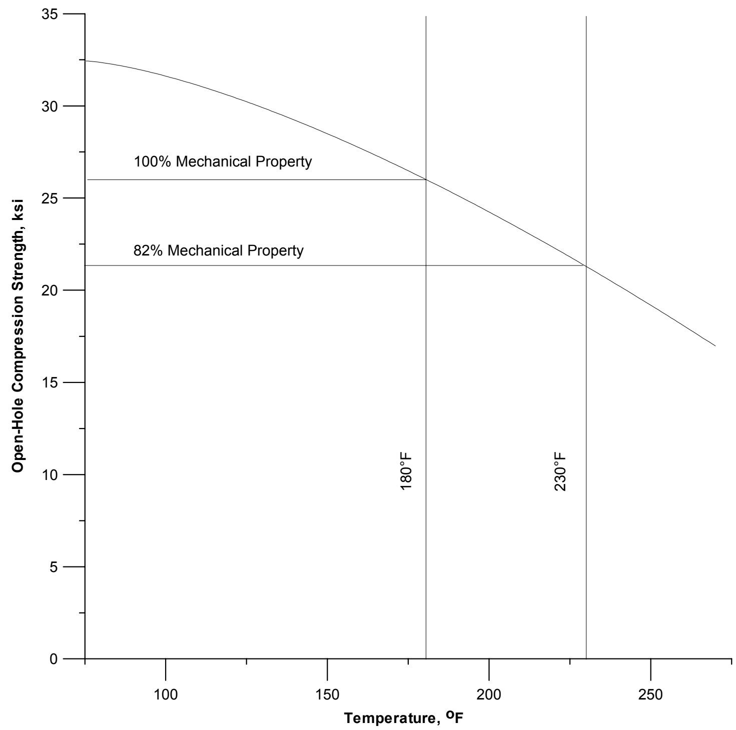

FIGURE 61. TWO-THIRD RETENTION METHOD SHOWN FOR NEWPORT 7781/NB321 GLASS FABRIC OPEN-HOLE COMPRESSION STRENGTH (0% RH)

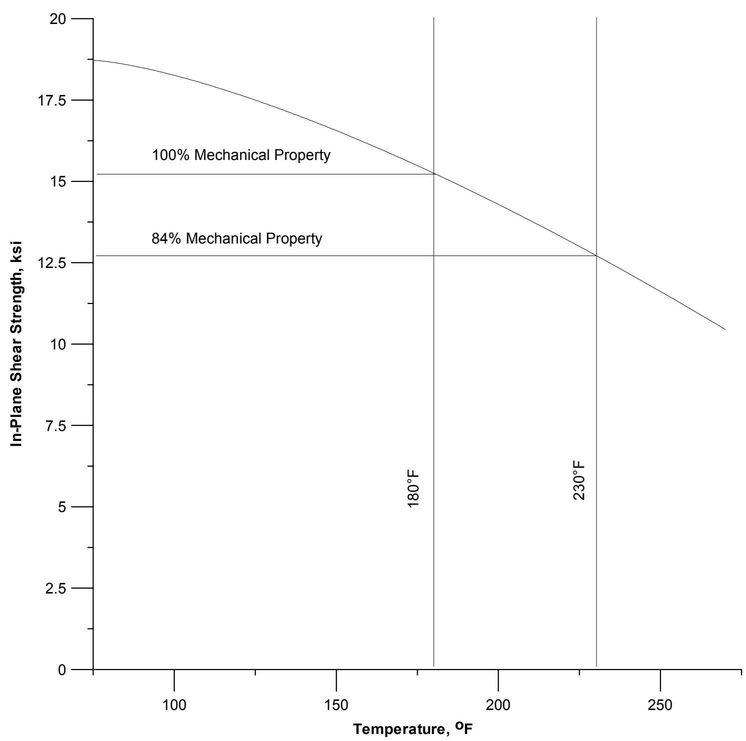

FIGURE 62. TWO-THIRD RETENTION METHOD SHOWN FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC IN-PLANE SHEAR STRENGTH (0% RH)

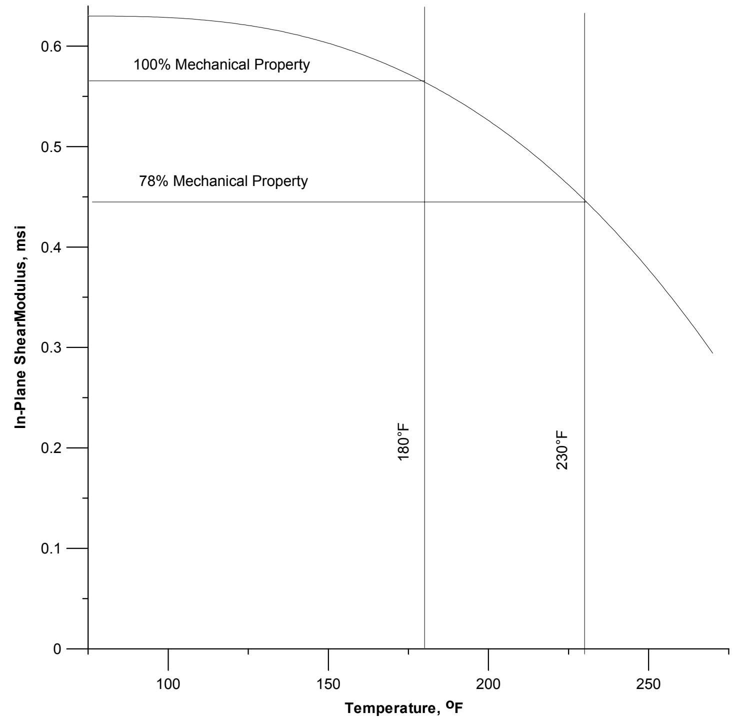

FIGURE 63. TWO-THIRD RETENTION METHOD SHOWN FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC IN-PLANE SHEAR MODULUS (0% RH)

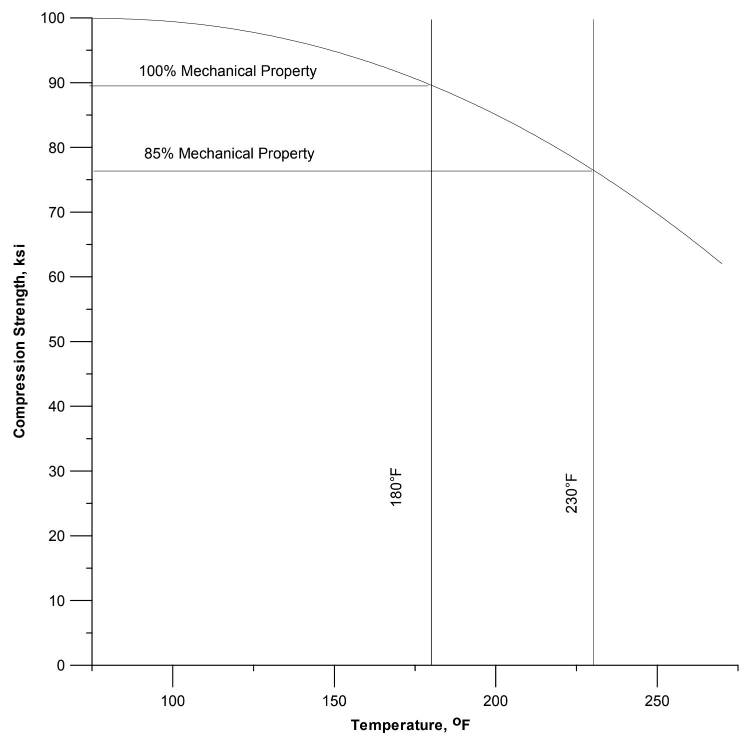

FIGURE 64. TWO-THIRD RETENTION METHOD SHOWN FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC COMPRESSION STRENGTH (0% RH)

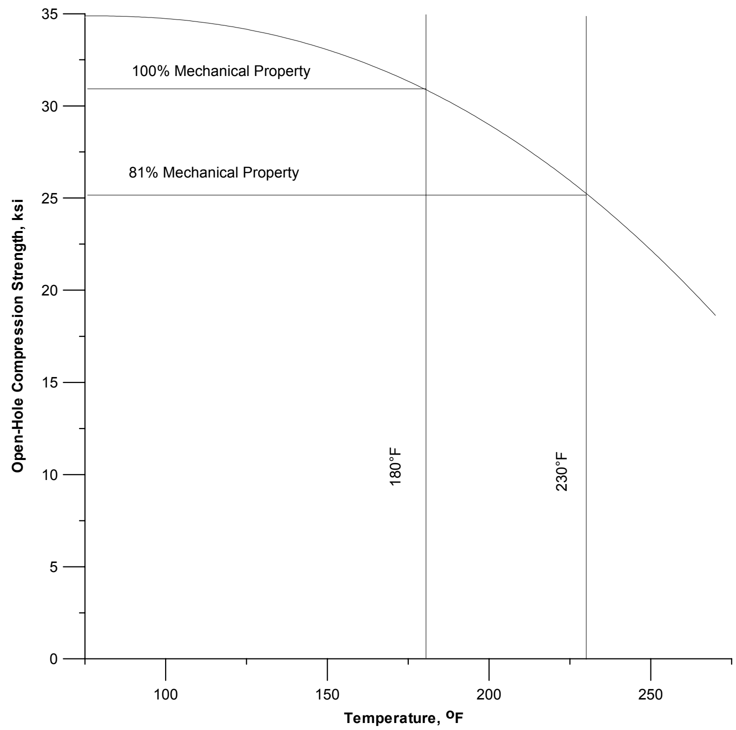

FIGURE 65. TWO-THIRD RETENTION METHOD SHOWN FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC OPEN-HOLE COMPRESSION STRENGTH (0% RH)

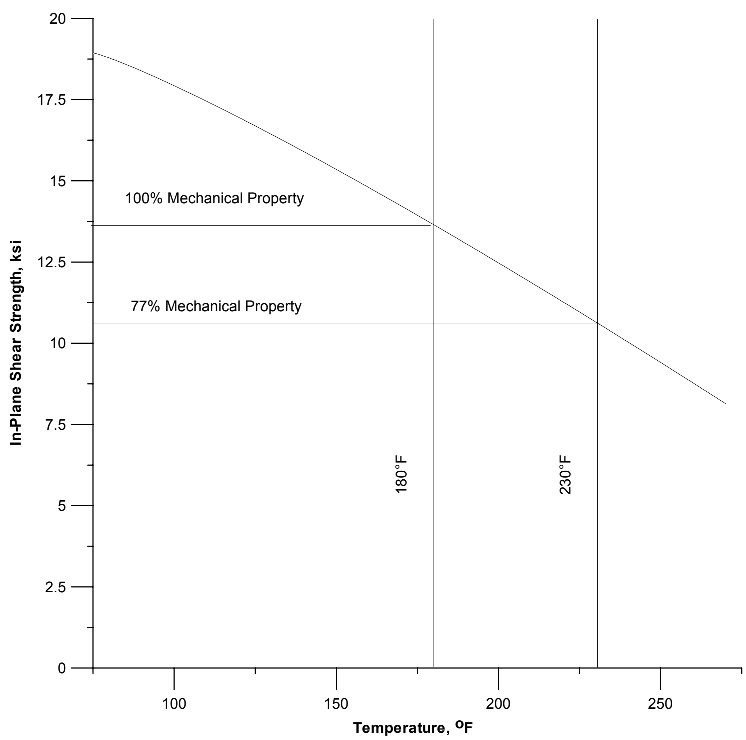

FIGURE 66. TWO-THIRD RETENTION METHOD SHOWN FOR NEWPORT 7781/NB321 GLASS FABRIC IN-PLANE SHEAR STRENGTH (68% RH)

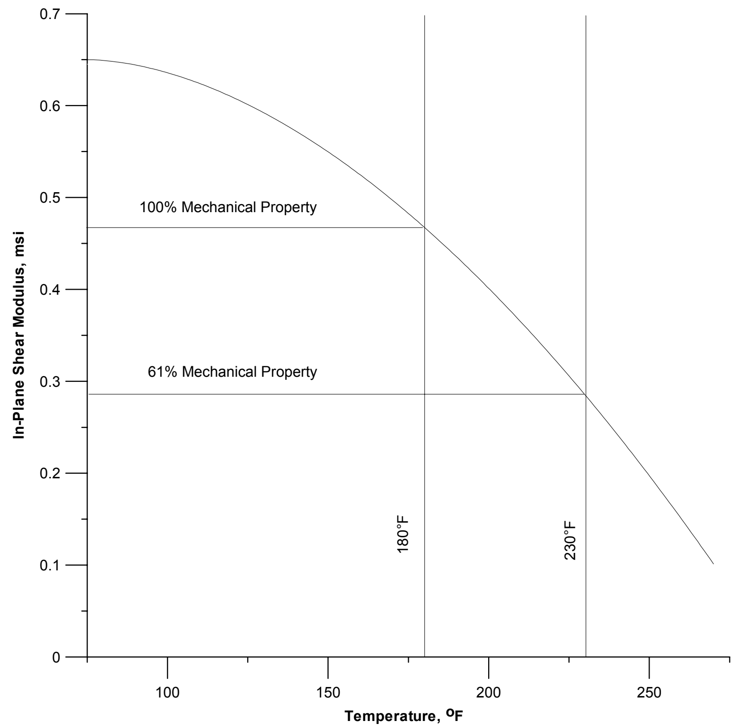

FIGURE 67. TWO-THIRD RETENTION METHOD SHOWN FOR NEWPORT 7781/NB321 GLASS FABRIC IN-PLANE SHEAR MODULUS (68% RH)

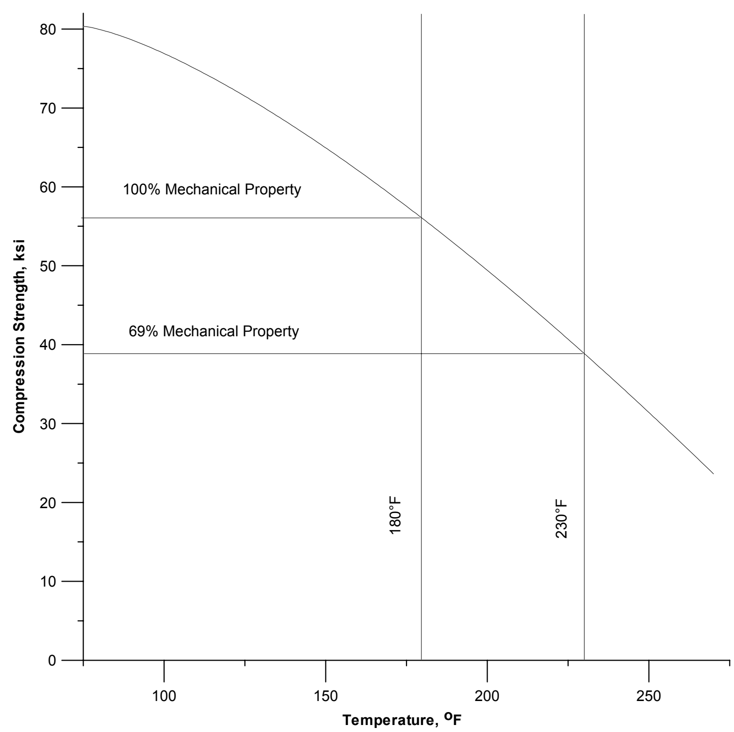

FIGURE 68. TWO-THIRD RETENTION METHOD SHOWN FOR NEWPORT 7781/NB321 GLASS FABRIC COMPRESSION STRENGTH (68% RH)

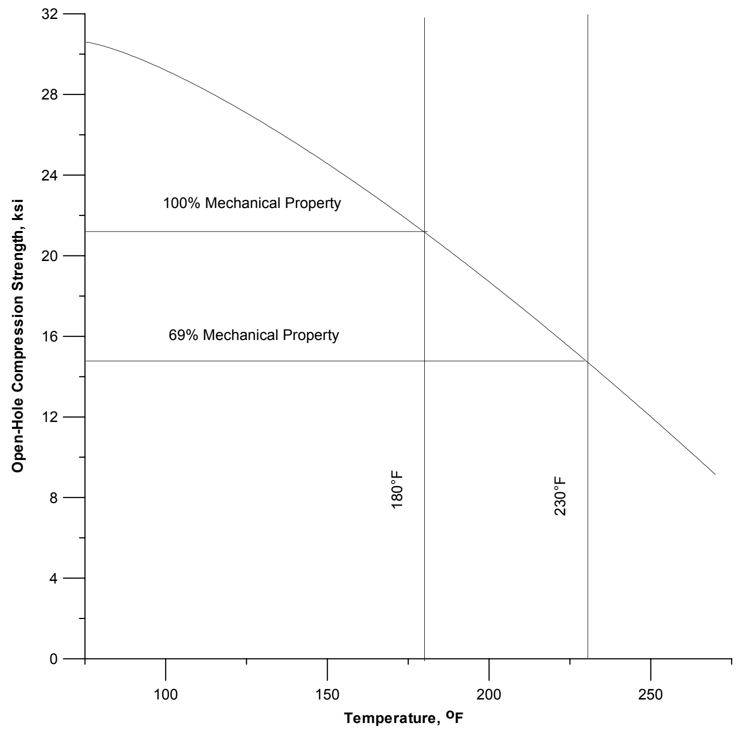

FIGURE 69. TWO-THIRD RETENTION METHOD SHOWN FOR NEWPORT 7781/NB321 GLASS FABRIC OPEN-HOLE COMPRESSION STRENGTH (68% RH)

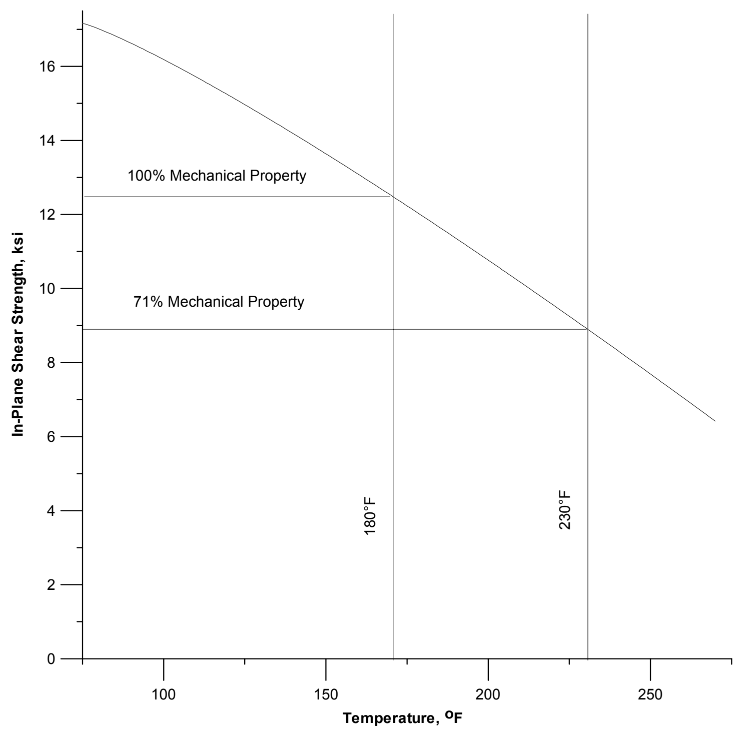

FIGURE 70. TWO-THIRD RETENTION METHOD SHOWN FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC IN-PLANE SHEAR STRENGTH (68% RH)

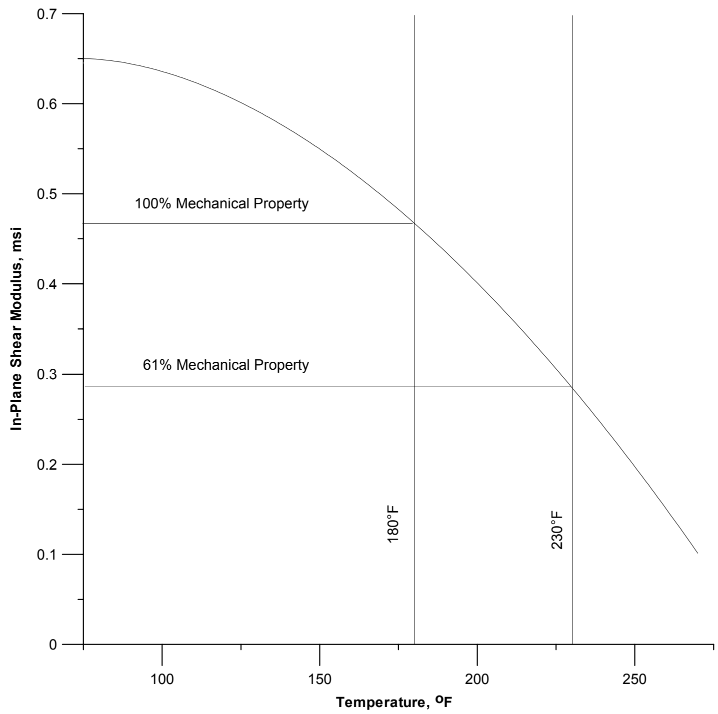

FIGURE 71. TWO-THIRD RETENTION METHOD SHOWN FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC IN-PLANE SHEAR MODULUS (68% RH)

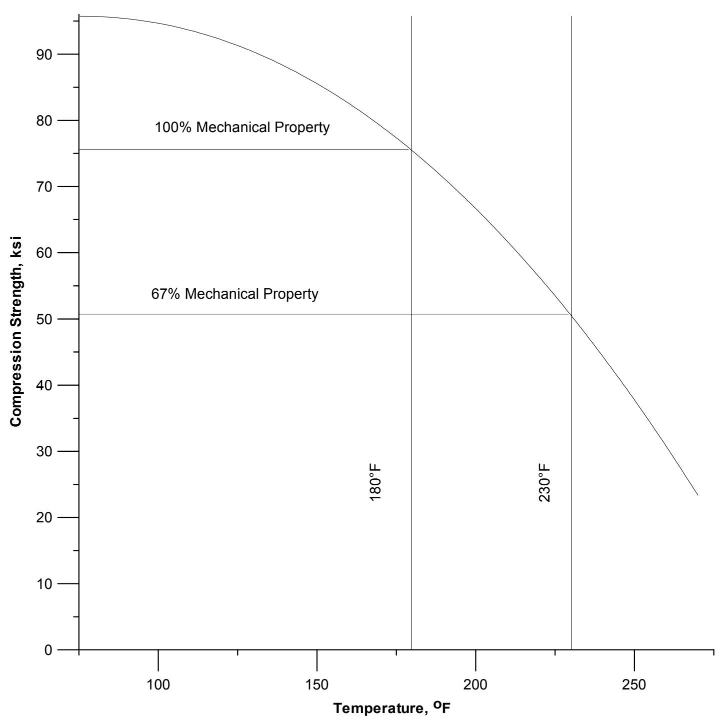

FIGURE 72. TWO-THIRD RETENTION METHOD SHOWN FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC COMPRESSION STRENGTH (68% RH)

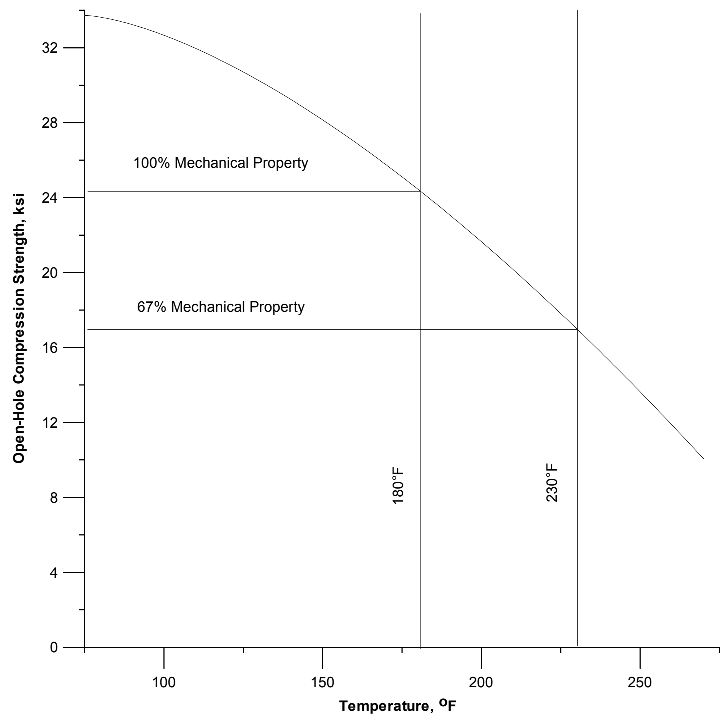

FIGURE 73. TWO-THIRD RETENTION METHOD SHOWN FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC OPEN-HOLE COMPRESSION STRENGTH (68% RH)

Moisture content of laminates increases the property degradation due to temperature. As shown on figures 74 to 77, at 85% RH, the property reduction between the MOL and 230°F is more than 1/3 for in-plane shear modulus and open-hole compression strength for the Newport glass fabric material system. For the FiberCote carbon fabric material system, all the properties failed the specifications of the 2/3 rule with the exception of in-plane shear strength.

It should be noted that the lowest retention value at 85% RH was 56%. If the maximum usage RH was chosen as 80% RH, all properties would have passed the 2/3 rule.

The disadvantage of the 2/3 rule is that additional testing is required at a temperature 50°F above the MOL. However, if the material system fails the 50°F T_g rule, it may still pass the 2/3 retention rule and be acceptable.

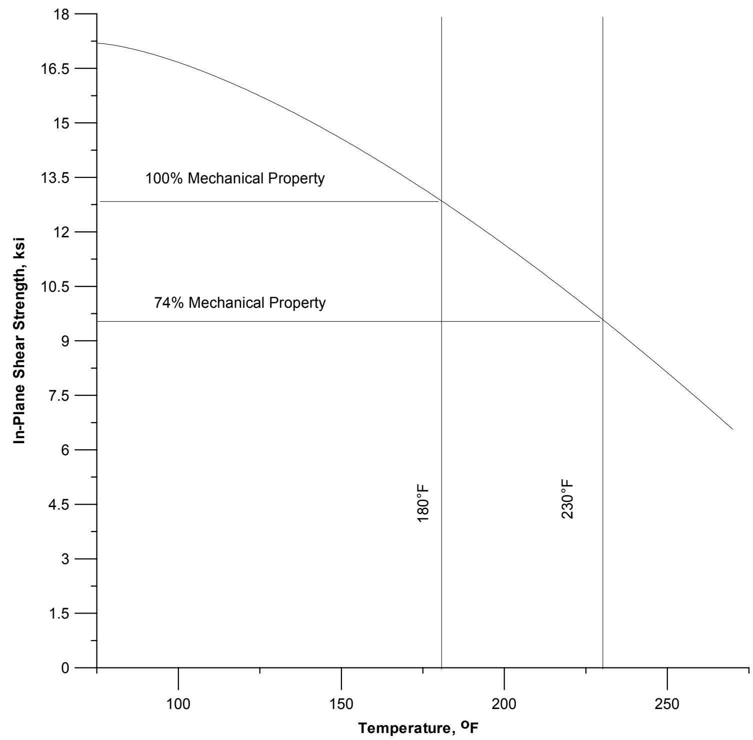

FIGURE 74. TWO-THIRD RETENTION METHOD SHOWN FOR NEWPORT 7781/NB321 GLASS FABRIC IN-PLANE SHEAR STRENGTH (85% RH)

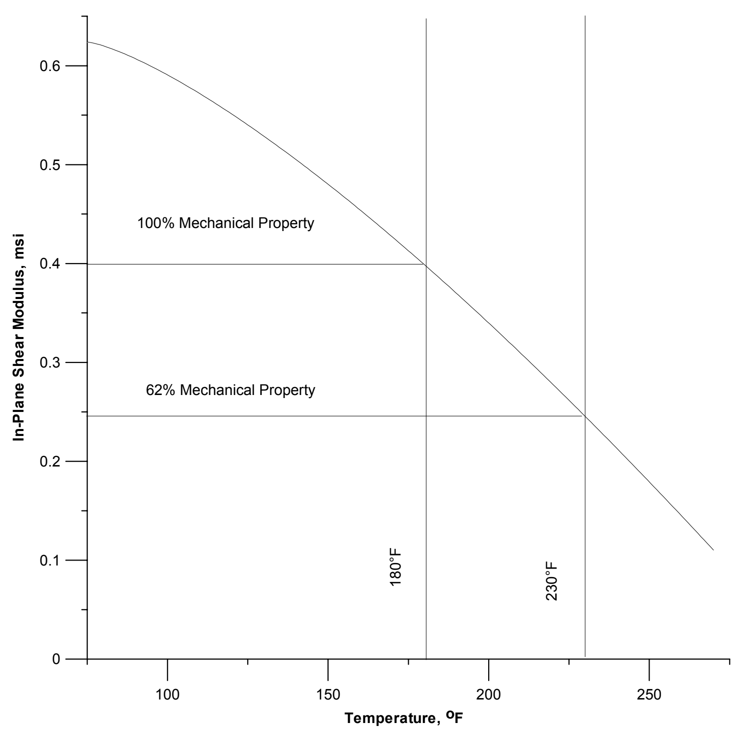

FIGURE 75. TWO-THIRD RETENTION METHOD SHOWN FOR NEWPORT 7781/NB321 GLASS FABRIC IN-PLANE SHEAR MODULUS (85% RH)

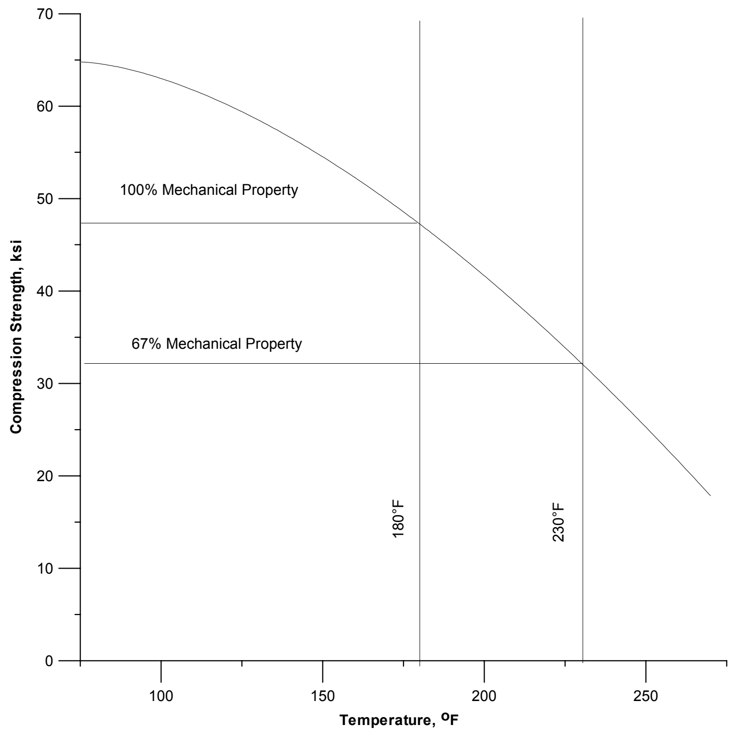

FIGURE 76. TWO-THIRD RETENTION METHOD SHOWN FOR NEWPORT 7781/NB321 GLASS FABRIC COMPRESSION STRENGTH (85% RH)

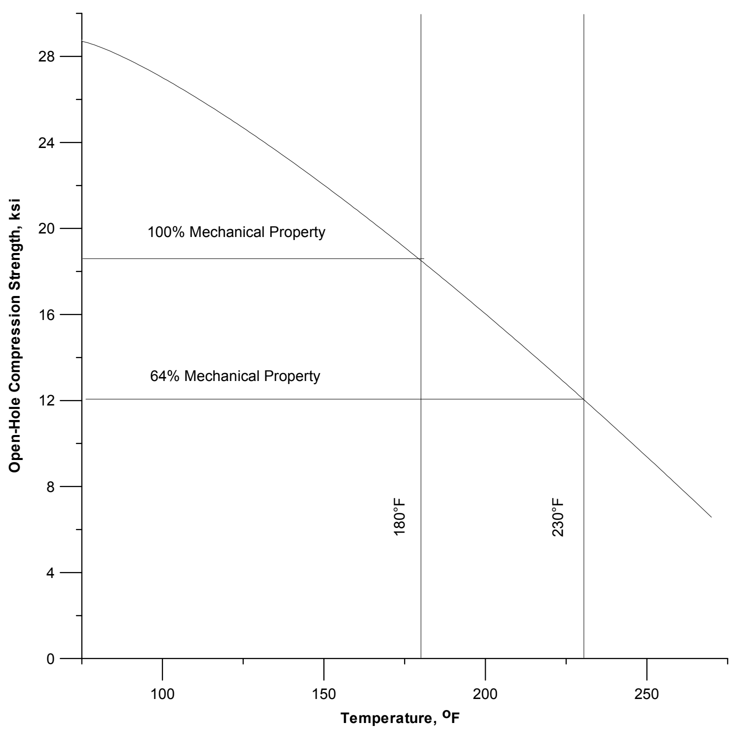

FIGURE 77. TWO-THIRD RETENTION METHOD SHOWN FOR NEWPORT 7781/NB321 GLASS FABRIC OPEN-HOLE COMPRESSION STRENGTH (85% RH)

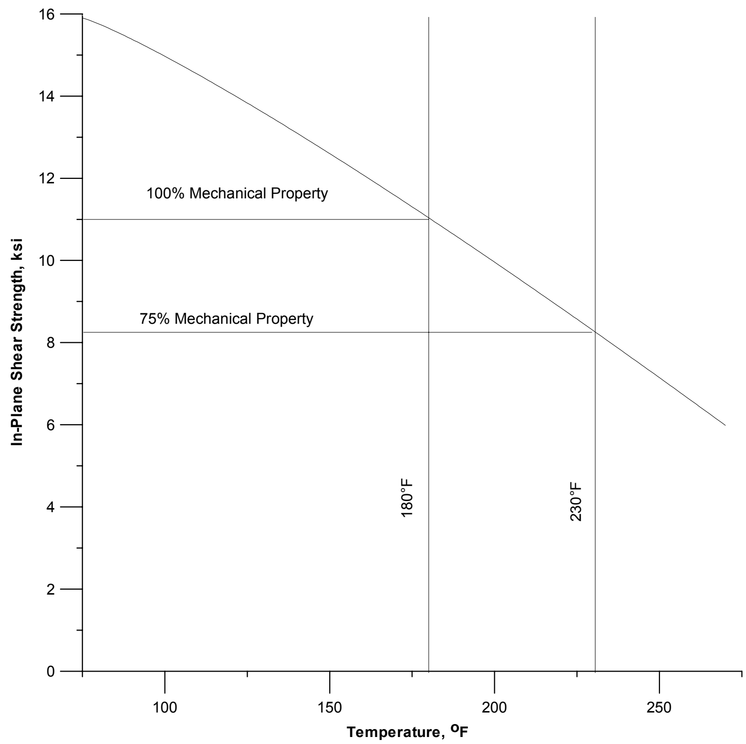

FIGURE 78. TWO-THIRD RETENTION METHOD SHOWN FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC IN-PLANE SHEAR STRENGTH (85% RH)

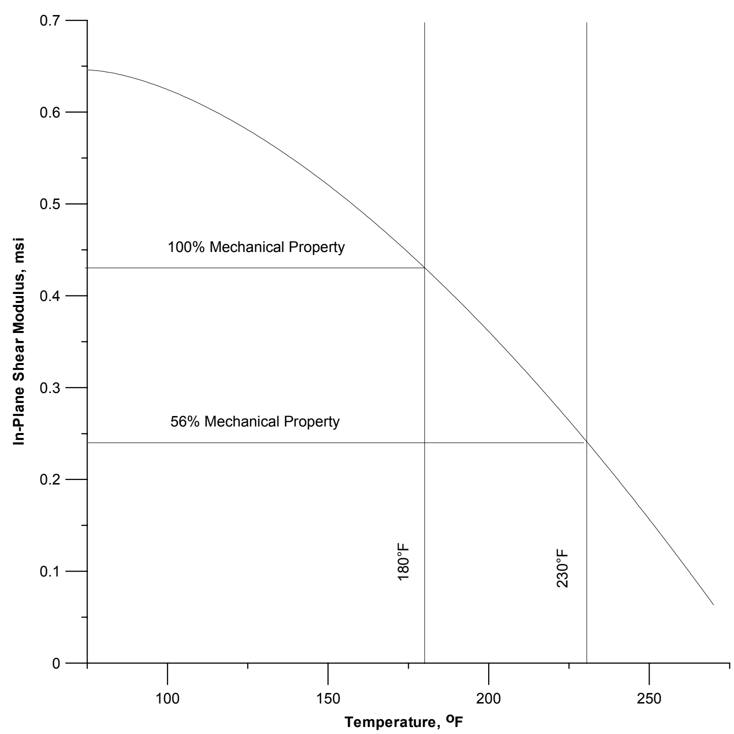

FIGURE 79. TWO-THIRD RETENTION METHOD SHOWN FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC IN-PLANE SHEAR MODULUS (85% RH)

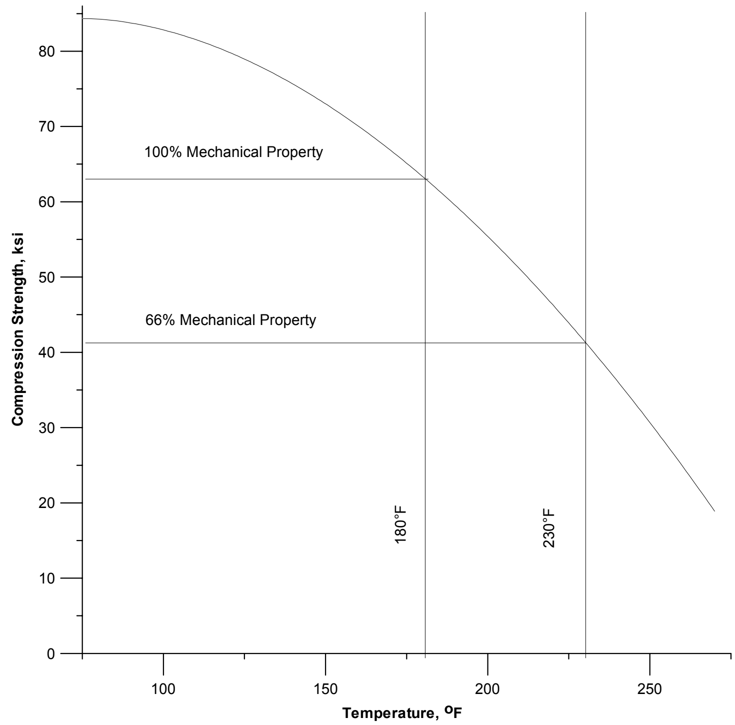

FIGURE 80. TWO-THIRD RETENTION METHOD SHOWN FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC COMPRESSION STRENGTH (85% RH)

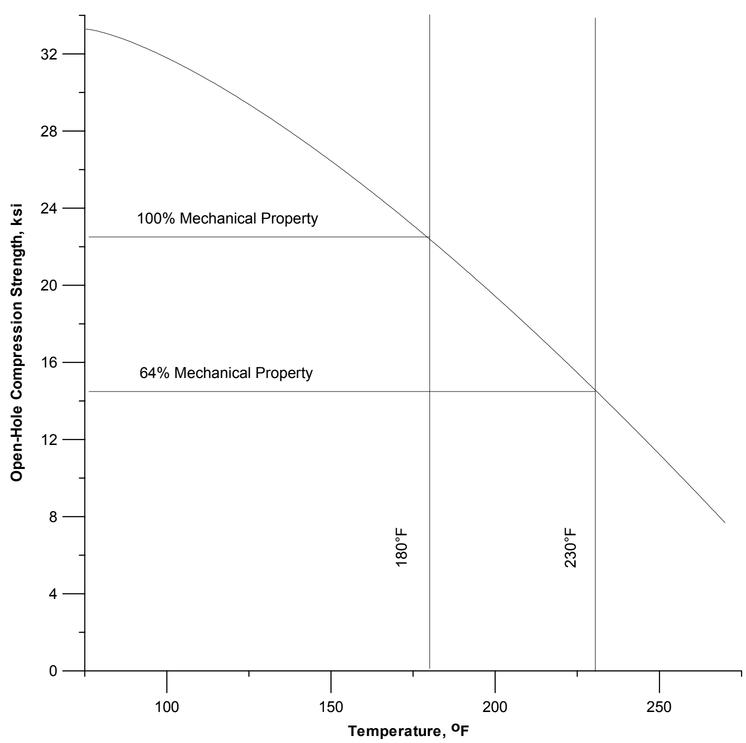

FIGURE 81. TWO-THIRD RETENTION METHOD SHOWN FOR FIBERCOTE E 765/T300 3KPW CARBON FABRIC OPEN-HOLE COMPRESSION STRENGTH (85% RH)

8. CONCLUSIONS AND RECOMMENDATIONS.

The purpose of this project was to first investigate the effect of temperature and humidity on the mechanical properties and the material operational limit (MOL); and second, to re-evaluate the validity of the 50°F margin method used to determine the MOL from glass transition temperature (T_g) values. Both efforts were for the 270°F cure material systems prevalent in the general aviation industry.

A series of specimens was manufactured to perform dynamic mechanical analysis (DMA) and mechanical tests. These specimens were conditioned to moisture requirements at three different relative humidity levels: 0%, 68%, and 85%. The mechanical tests performed included compression strength and modulus, in-plane shear strength and modulus, and open-hole compression strength. These tests were performed at six different temperatures: room temperature, 150°, 180°, 210°, 240°, and 270°F for the three relative humidity levels investigated. Two different temperature ramp-up rates (1°C/min and 5°C/min) combined with two different specimen thickness values were used to perform the DMA tests.

Glass transition temperatures were obtained for the two materials tested at two different thicknesses and ramp-up rates, as specified by ASTM E 1640 and SACMA SRM 18. No specific trends that would favor one standard over the other were found. Hence, the four results at each moisture level were averaged and used as the T_g measurement for that material. For both materials the T_g decreased with increase in the moisture level at the RH of 0%, 68%, and 85%.

For the mechanical tests performed, as the temperature was increased, significant property reductions occurred with the exception of the compression modulus values, which remained almost unchanged. Moisture increased the property degradation due to temperature. The in-plane shear modulus was found to be the most sensitive property to moisture and temperature. However, it should be noted that the knee observed in the property reduction curves, for materials cured at 350°F, was not evident in the curves obtained for the two material systems considered, which were cured at 270°F. The property degradation with temperature was a smooth decline.

The operational limit of both material systems was determined using the 50°F margin method at 85% RH. For the Newport 7781/NB321 glass fabric material system, the MOL was found to be 169°F. For the FiberCote E 765/T300 3KPW carbon fabric material system, the MOL was found to be 179°F, which is very close to 180°F, a typical maximum operating temperature.

This indicates if the 50°F rule was relaxed by 10°F to 40°F, both 270°F cure materials would pass the modified rule. Such a change in the rule is reasonable, as the mechanical properties are not reduced drastically at higher temperatures but decrease in a controlled manner.

The 50°F margin method was validated using the 2/3 retention method. The 50°F margin method is based on T_g values whereas the 2/3 rule is based on mechanical property data. At 0% RH, all the mechanical properties satisfied the 2/3 rule for both material systems. At 68% RH, in-plane shear modulus was the only property that failed the 2/3 rule specifications for both material systems. At 85% RH, in-plane shear modulus and open-hole compression strength failed the 2/3 rule for the Newport glass fabric material system. For the FiberCote carbon fabric material system, all the properties failed the specifications of the 2/3 rule with the exception of in-plane shear strength. However, it should be noted that the lowest retention value at 85% RH was 56%.

It is recommended that the 50°F margin method based on T_g values should be retained for the 270°F cured materials. If there is difficulty meeting this rule, 2/3 retention rule of mechanical properties should be used, but it will require additional testing at temperatures above MOL. An advantage of this additional testing was illustrated in the current study where the material having the lowest MOL by the 50°F margin method was shown to retain the highest mechanical properties at 50°F above 180°F.

The results obtained were based on static tests. More extensive research needs to be performed to determine a realistic method of finding the MOL. The method should consider results of time dependent properties such as creep, a probabilistic study, that would determine the likelihood of simultaneous occurrence of high temperature and moisture content, and a thermomodeling analysis that would determine realistic usage temperatures.

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APPENDIX A—BASIC PRINCIPLES OF DYNAMIC MECHANICAL ANALYSIS

A.1 AMORPHOUS VERSUS CRYSTALLINE.

Crystalline materials such as aluminum have constituent atoms stacked together in a regular order (i.e., repeating pattern). Materials that form crystalline structure in solid state typically have a distinct melting point. Water, for example, is a crystalline material (forms crystals on car windshields during cold seasons) and melts at 32°F at sea level. Amorphous materials such as amorphous polymer may have some molecular order but usually are substantially less ordered than those in crystalline materials. The molecules are generally long and randomly oriented, much like spaghetti. Amorphous materials typically do not have a distinct melting point.

A.2 DEFINITION OF GLASS TRANSITION TEMPERATURE (T_g).

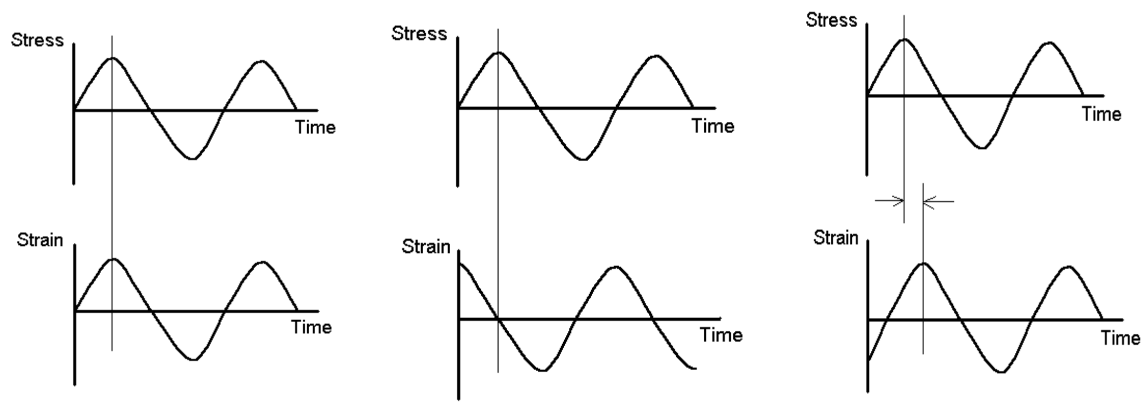
Amorphous polymer softens rather than melt when heated. The softening occurs over a wide range of temperature. The temperature range at which amorphous materials soften is known as the glass transition temperature. (Note that glass is an amorphous material and the term "glass transition temperature" is originally used to describe the softening behavior of glass at high temperature. Its ability to stay soft over a wide range of temperature allows glass to be formed/blown into complicated/artistic shapes). Since glass transition temperature is actually a range of temperature rather than a single value of temperature, the method of which a single temperature value is interpreted from this range can be a controversial issue.

In addition to becoming soft, amorphous materials undergo many other physical changes when heated above their T_g. The coefficient of thermal expansion (CTE) of the material usually increases when heated above its T_g. One of the methods, thermomechanical analysis (TMA), detects the T_g of a material by monitoring the CTE. The specific heat of the materials also increases when heated above their T_g. Differential scanning calorimetry (DSC) detects the T_g by monitoring the specific heat. Dynamic mechanical analysis (DMA) is used in this project for determining T_g. It detects the T_g by monitoring the modulus of the material.

A.3 DMA FOR DETERMINING T_g.

DMA is a test method for determining material viscoelasticity behavior. The term "viscoelasticity" is the derived from "viscosity" and "elasticity." DMA is capable of separating the viscous and elastic properties and allow each property to be analyzed separately. This report uses two methods to interpret a single T_g value from the range of temperature; the onset of storage modulus and the peak of tangent delta.

In order to discuss the method of which DMA separates the two responses, it is necessary to discuss viscosity and elasticity. Figure A-1(a) shows the response of a purely elastic material (Hookean solid) subjected to a sinusoidal load. The strain is in phase with the applied stress. In other words, there is no phase lag (δ=0°). The material behaves like a spring. Figure A-1(b) shows the response of a purely viscous material (Newtonian liquid) due to sinusoidal load. The material behaves like a dashpot. The phase angle, δ, is 90°. Figure A-1(c) shows the response of a viscoelastic material. The phase angle of a viscoelastic material is between 0° and 90° ($0° < \delta < 90°$).



(a) (b) (c)

FIGURE A.1. PHASE ANGLE OF (a) PURELY ELASTIC MATERIAL, (b) PURELY VISCOUS MATERIAL, AND (c) VISCOELASTIC MATERIAL

Knowing that the elastic and viscous responses are 90° apart, the two responses can be separated and represented in complex number form.

$$E^* = E' + iE''$$

where E^* = as-measured modulus (also known as complex modulus)
E' = storage modulus (related to the elastic portion of the material)
E'' = loss modulus (related to the viscous portion of the material)

The equation can also be represented by the triangle in figure A-2.

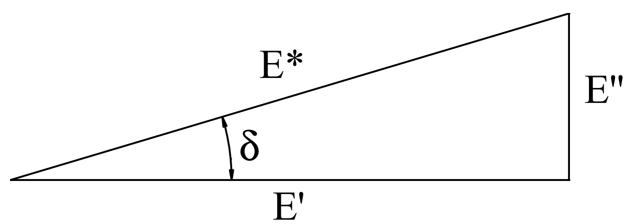


FIGURE A-2. COMPLEX MODULUS, STORAGE MODULUS AND LOSS MODULUS REPRESENTED BY A TRIANGLE

Figure A-3 shows a typical DMA result with three methods to interpret T_g values. Typically, the most conservative T_g value is obtained from the onset of storage modulus curve. The "onset" point is obtained from the intersection of two tangent lines. The second most conservative T_g value is usually obtained from the peak of loss modulus curve. This method is not used in this report. The third and generally least conservative T_g value is obtained from the peak of tangent δ curve. The onset of storage modulus curve (minus some temperature margin such as 50°F) is generally considered the most relevant method for establishing MOL because the interest is to avoid the temperature at which large material property degradation (i.e. modulus) starts to occur. It is also the method recommended by SACMA SRM 18 and ASTM E1640-94.

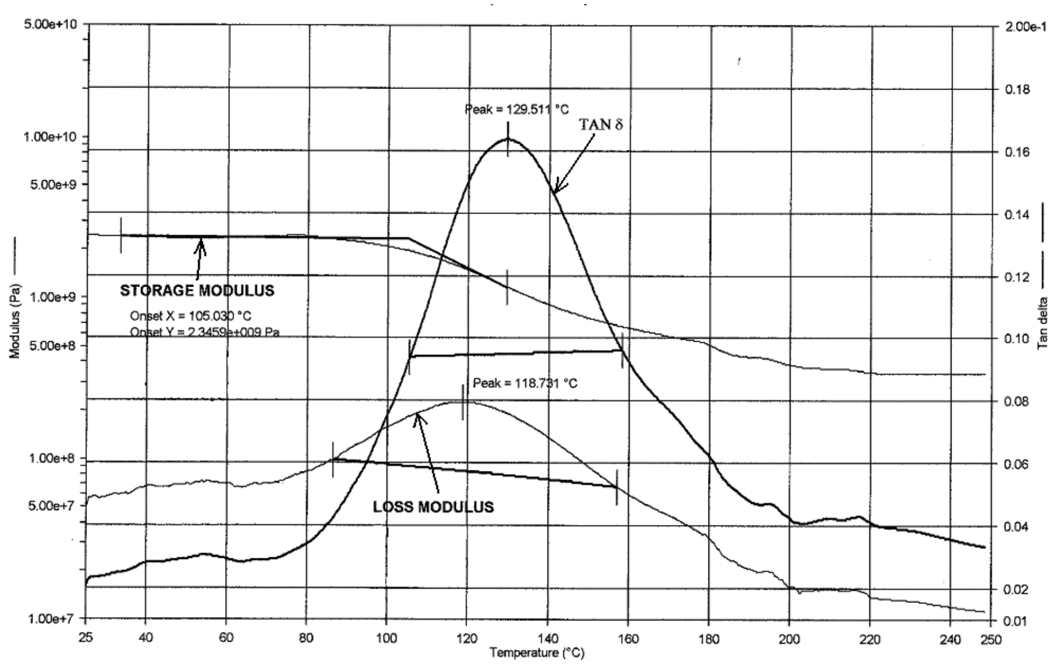


FIGURE A-3. A TYPICAL DMA RESULT

The T_g results from DMA are influenced by many factors. Increasing the test frequency generally increases the interpreted T_g values and decreases the slope of the storage modulus curve in the transition region. The T_g values obtained from dissimilar test frequencies are, therefore, not comparable. For this reason, all the DMA tests in this report were obtained using a fixed 1 Hz frequency in accordance with SACMA SRM 18 and ASTM E1640-94. Although these two specifications agree on the test frequency, they disagree on specimen thickness and temperature rate increase. ASTM E 1640 specifies 0.04 in (1 mm) thickness and 1°C/min ramp-up rate. SACMA SRM 18 specifies 0.12 in. (3 mm) thickness and 5°C/min ramp-up rate. The cross-linking density can influence the T_g values also. Higher cross-linking density generally result in higher T_g values. Humidity is another factor that affects the T_g values. Epoxy that has absorbed moisture tends to have a lower T_g. Although not well documented, the volatile in uncured epoxy resin is believed to have a negative impact on T_g.