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STATUS REPORT –FIBERBOARD PROPERTIES AND DEGRADATION RATES FOR STORAGE OF THE 9975 SHIPPING PACKAGE IN KAC

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Summary

Thermal, mechanical and physical properties have been measured on fiberboard samples following accelerated aging for up to approximately 12 years. The aging environments include elevated temperature up to 250 ºF (the maximum allowed service temperature for fiberboard in 9975 packages) and elevated humidity. The results from this testing have been analyzed, and aging models fit to the data. Correlations relating several properties (thermal conductivity, energy absorption, weight, dimensions and density) to their rate of change in potential storage environments have been developed. Combined with an estimate of the actual conditions the fiberboard experiences in KAC, these models allow development of service life predictions.

Development of the current models recognizes that the primary constituents of fiberboard (cellulose, hemicellulose and lignin) are polymers, and the data are analyzed in a manner consistent with polymer behavior. This includes the assumption of Arrhenius behavior and identifies a characteristic activation energy that describes the temperature dependence of fiberboard degradation. This activation energy is seen to vary as the moisture level changes. Data found in the literature show large variation in the activation energy depending on test method and environment (test atmosphere), but the general trends are consistent with those observed for fiberboard. Specifically, the activation energy decreases, and the temperature dependence of the reaction rate increases as the moisture level (absolute humidity) increases. This trend is reversed when compared to relative humidity since relative humidity has a built-in interrelationship with temperature.

KAC has recently completed calculations that supported extending the service life of 9975 packages in storage from 15 years to 20 years. These calculations addressed the potential for degraded fiberboard properties following 20 years in storage using degradation models developed previously. The updated degradation models developed in this report have been compared to the assumptions in these calculations, and the current results remain consistent with those calculations.

Some of the predicted degradation rates presented in this report are extreme. However, these relate to environments that do not exist within KAC, or would be postulated only as upset conditions that would not likely persist for an extended period. For a typical package stored in KAC with ~10 watts internal heat load or less, and ambient temperatures below 90 ºF, the fiberboard experiences storage conditions less severe than any of the aging environments. Fiberboard in conforming packages with lower internal heat loads should experience little or no degradation, and is expected to provide a service life beyond the currently approved 20 year storage period. Packages with higher internal heat loads may not continue to perform their required safety functions beyond 20 years. Ultimately, the service life will be determined by the cumulative effect of degradation from all the conditions these packages might encounter. Additional data continue to be collected to permit future refinements to the models and assumptions.

The results and model predictions presented in this report are applicable to 9975 packages with cane or softwood fiberboard overpack assemblies. These degradation models do not address the effects of non-conforming conditions such as the presence of excess moisture and mold, or beetle infestations.

Background

Celotex® fiberboard material is used in the 9975 shipping package between the outer 304L stainless steel drum and the lead shielding, and provides three safety functions: thermal insulation to limit internal temperature during a fire, criticality control, and resistance to package crushing [1]. The fiberboard material must retain its dimensions and density within certain ranges to provide the required impact resistance, criticality control, and fire resistance. Several properties of interest to demonstrate acceptable long-term performance of the material include dimensional stability, density, compressive strength, thermal conductivity, and specific heat capacity.

Fiberboard samples have been conditioning in elevated temperature or elevated temperature / humidity environments since 2005 to identify degradation trends. These samples have been taken from multiple fiberboard assemblies fabricated from both cane and softwood fiberboard, and provide data for tracking changes in thermal, mechanical, and physical properties [2]. Duplicate samples from multiple package sources have been tested to identify the range of variability in fiberboard properties and degradation rates.

Baseline and long-term testing of mechanical and thermal properties have been reported previously. References 3 and 4 summarize available data on cane and softwood fiberboard, respectively, through August 2015 and present degradation models for the measured properties. Additional data have since been collected, and the cumulative data set through November 2017 is analyzed in this report for the refinement of aging models.

Test Data

The aging times vary for different samples in each environment due to new samples and aging environments being added on several occasions. Table 1 summarizes the maximum conditioning times for each environment through November 2017. Environments which include humidity control typically have shorter durations since only a single environmental chamber was available through 2010, and samples were conditioned sequentially. Since 2010, three or more environmental chambers have been available for conditioning samples.

Data from samples taken from the following source packages have been used in estimating degradation rates and developing aging models:

- LD1 and LD2 – undamaged portions of 2 cane fiberboard lower assemblies from drop tested packages, which were in storage for ~10 years prior to this effort. The first samples began conditioning in 2005.
- MSC – undamaged portions of several cane fiberboard lower assemblies from drop tested packages, which were in storage for ~10 years prior to this effort. Traceability to specific assemblies was not maintained for these samples. The first samples began conditioning in 2005.

- 2234 – cane fiberboard lower assembly removed from package 9975-02234 following 2 years service in KAC, and subsequent surveillance activities. The first samples began conditioning in 2006.
- New – remnant portions of a new cane fiberboard assembly (upper and lower) purchased in 2005 for a separate effort. The first samples began conditioning in 2006.
- SW – a new softwood fiberboard lower assembly provided by KAC. The first samples began conditioning in 2008.
- T4SW and T5SW – softwood fiberboard lower assembly from training packages T4 and T5. Samples began conditioning in limited environments in 2014.
- 6100 – softwood fiberboard from package 9975-06100 following field surveillance and destructive examination. Samples began conditioning in 2014.

Samples from the following additional source packages have also been conditioned and tested, but the number and type of samples is limited. Therefore, these samples were generally not considered in developing aging models.

- KT2 – cane fiberboard lower assembly from an unused package following several (<5) years in storage. These samples began conditioning in 2006.
- 826 – cane fiberboard lower assembly removed from package 9975-00826 following 3 years service in KAC, and subsequent surveillance activities. The first samples began conditioning in 2006.
- 826U, 600U – cane fiberboard upper assemblies removed from packages 9975-00826 and 9975-00600 following 3 and 5 years service in KAC, respectively, and subsequent surveillance activities. Samples began conditioning in 2009.

Previously, separate aging models were developed for cane fiberboard and softwood fiberboard. As additional aging time has accumulated on the softwood fiberboard samples, the apparent differences between these two materials has decreased, with significant overlap in the range of properties for these materials in most aging environments. While limited bias remains in some aging environments, it is now judged that combining data from both materials provides the best overall representation of bulk fiberboard degradation.

Compression Tests

Unlike the thermal and physical tests, compression testing is destructive – each sample can be tested only once. Therefore, these samples become increasingly important after extended conditioning periods as fewer conditioned samples remain for future testing. Additional compression test samples have been added to the aging environments on several occasions following periodic review of the data and identification of need for additional data. Compression testing has been performed following aging for as long as 11 years in some environments.

Compression test samples are nominally 2 x 2 x 2 inches in size, and are tested at a crosshead speed of 1.9 inch/minute. The load is applied either parallel or perpendicular to the fiberboard layers. The test continues until a limit is reached, either a maximum strain (85%), or a maximum load (20,000 or 25,000 pounds, depending on the load cell used).

Typical compression stress-strain curves are shown in Figures 1-2 for samples conditioned in two of the aging environments – 185 ºF and 250 ºF ovens. The compression test provides data relevant to the safety function of resistance to package crushing. This function relates to the energy absorbed by the fiberboard during crushing. The integrated area under the stress-strain curve up to a strain of 40% provides a relative measure of the energy absorption capability of each sample. The 40% strain level is arbitrary, but provides a consistent point of comparison.

While fiberboard samples have been compression tested in both orientations (parallel and perpendicular to the fiberboard layers), samples tested in the perpendicular orientation are more relevant to the behavior of a package in storage. This is because the self-constraint against lateral spreading provided by the glue layers and general fiber orientation reasonably mimics the constraint provided to the fiberboard assembly by the 9975 drum. Samples tested in the parallel orientation without lateral constraint can significantly underestimate the energy absorption capacity of the fiberboard assembly due to sections of the sample splitting off under load. Accordingly, only data collected in the perpendicular orientation will be used in modelling the degradation of energy absorption capability. Values of the area under the stress-strain curve up to 40% strain for perpendicular samples are shown in Figure 3.

Thermal Tests

Thermal conductivity samples are typically ~7 x 7 inches by 1 – 2 inches thick. Some of the initial samples were larger in area (~12 x 12 inches). The samples are removed from the fiberboard assemblies in an orientation that characterizes heat flow through the assembly in either the axial (perpendicular to the fiberboard layers) or radial (parallel to the fiberboard layers) direction. Thermal conductivity samples have been removed from each of the source packages, although varying numbers of source packages are included among the samples aged in each environment.

Thermal conductivity is measured in a LaserComp Fox 300 or Fox 314 heat flow meter instrument. Tests are conducted at mean temperatures of 25 and 50 ºC (77 and 122 ºF) on all samples. Samples aging in the higher temperature environments have also been tested at a mean temperature of 85 ºC (185 ºF). The LaserComp instruments conduct the test in accordance with ASTM C518-91 (Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus).

Thermal conductivity data for each environment are summarized in Figure 4. For ease of comparison, the thermal conductivity data for each sample are normalized to the first measurement taken after conditioning began. These first conditioned values are summarized in Table 2 for both cane and softwood fiberboard to show the range of sample-to-sample variation, and the degree to which thermal conductivity varies with each environment.

Specific heat capacity is measured in accordance with ASTM C351-92b (Reapproved 1999) (Standard Test Method for Mean Specific Heat of Thermal Insulation) at mean test temperatures of 25 and 51 ºC (77 and 124 ºF). Samples are cylindrical in shape, with 1 inch diameter and ~1.5 inch height. Between 3 and 5 specific heat capacity samples (removed from the same source package) are aged in selected environments, and each may experience multiple trials at each test interval. The specific heat capacity data can show a significant degree of scatter from one trial to the next. Accordingly, the results are averaged over all samples and trials for a given conditioning interval and test temperature. A summary of these averaged data is shown in Figure 5. The nominal rate of decrease in specific heat capacity is shown under Figure 5 by the coefficient in each equation that was fit to the data for each environment.

Physical Tests

Fiberboard weight, dimensions, and density have been tracked with small samples (~2 inch cubes) in each environment. In addition to the elevated temperature environments mentioned above, these physical property samples have also been conditioned at temperatures of 50 and 15 ºF, at ambient humidity and in a desiccated environment. For these low temperature environments, the ambient humidity is approximately 10% at 50 ºF and 60% at 15 ºF. No significant change in physical properties was noted in these low-temperature environments.

In order to better compare samples and highlight changes among samples with different initial property values, the properties (weight, density, height and length / width) of each sample are normalized to their initial conditioned value. The normalized data from these samples are summarized in Figures 6-9. Samples from multiple material sources are conditioned in each of the elevated temperature environments. Initially, data were collected on a single sample source (MSC) only. Samples from additional package sources were added subsequently.

Ovens are used to provide nominally dry environments for aging fiberboard samples. These environments typically have a very low relative humidity consistent with the ambient laboratory environment (40 %RH at 75 ºF corresponds to ~10 %RH at 125 ºF, ~2 %RH at 185 ºF, etc). Since the laboratory experiences seasonal variations in relative humidity, samples in these dry environments can exhibit comparable seasonal variation in physical properties. This is most pronounced at the lowest temperature (125 ºF), and less obvious at the higher temperatures.

Termination and Re-purposing of Samples

Compression testing is destructive in nature, and the compression samples are tested once and retired from further testing. The remaining tests are non-destructive, and provide for repeated testing after periods of aging. Some of these samples have been retired from testing for various reasons. Typically, samples will be retired after their properties degrade to a point beyond the established acceptance criteria, or if they become too fragile to take measurements without significant risk of damage from handling. This has been the case for many samples aging in the 250 ºF oven, and all samples in the 185 ºF 70 %RH environment.

In the milder environments, some of the physical property samples have been removed from test as well. This was done after significant exposures were achieved (typically 3 – 8 years), and the samples from the various source packages were observed to have very similar degradation rates. Several of the retired physical property samples were re-purposed as compression test samples to provide a few data points at much longer exposures than were otherwise available. In addition, some of the retired thermal conductivity samples have been cut, re-glued and re-machined to provide additional compression test samples. This technique uses only axial orientation thermal conductivity samples. If the compression test is then conducted in the parallel orientation, care is exercised to ensure the re-glued sample does not have significantly more glue layers than normal (typical samples have 4 – 5 glue layers)

Analysis

To date, twelve packages have been removed from storage for destructive examination (DE), after being held in storage for periods ranging from ~5 months to 12.8 years. The internal heat load of these packages has ranged from 3 to 16.5 watts. Fiberboard degradation was evident in only one of these packages, which was in storage for 9.6 years with 16.5 watts internal heat load [5]. The remaining DE packages (with heat loads below 15 watts) indicate the storage environment is sufficiently mild to preclude significant degradation over this time period for the large majority of packages which contain lower heat loads.

Several physical property samples have been maintained at ambient laboratory conditions, and measured periodically. While these control samples show a small loss of material from handling, they also suggest there is no significant permanent change in properties occurring at ambient conditions. Further, prior reports [3, 4] observed that fiberboard samples aged at 125 ºF (both at 70%RH and in a dry oven) consistently show very low degradation rates, and occasional positive changes in property values. Given these small changes, on the order of sample variability and measurement uncertainty, it was concluded that a threshold for fiberboard degradation may exist at about 120 ºF. For many packages in KAC with internal heat loads below ~10 – 12 watts, most or all of the fiberboard assembly would remain below this apparent threshold temperature.

The fiberboard within a heated package will develop temperature and moisture gradients. Moisture will tend to migrate to the cooler regions of the fiberboard, while the total moisture content will change very slowly (if at all). Testing to characterize the moisture / humidity distribution within a fiberboard assembly suggests that moisture re-distributes in a manner that maintains a relatively constant level of absolute humidity [6, 7].

The 9975 SARP notes that the package does not provide an air- or water-tight seal. However, upper fiberboard subassembly testing [8] has demonstrated that a properly closed drum does provide a significant degree of isolation of the fiberboard from the ambient environment. Accordingly, any moisture originally in the fiberboard assembly will likely remain in the package for a long time. In addition, as the fiberboard degrades at elevated temperature, water is released as a byproduct. Therefore, a balance will eventually develop within each package where the rate of water generation due to fiberboard degradation equals the net transfer of moisture into / out of the package. It is likely that the equilibrium moisture content of the fiberboard will vary between packages (driven by variation in the degree to which each package can "breathe") and with seasonal changes in the ambient humidity level.

The range of moisture content measured in the upper fiberboard assemblies exposed to the ambient environment is ~6 – 14 %WME (wood moisture equivalent) or ~7 – 12 wt%. This moisture content will define the relative humidity within a package, which needs to be identified to correlate the laboratory test data to degradation under storage conditions. Humidity measurements have been taken in the upper air space of a number of packages in KAC [9]. However, significant variation is observed between packages. Some of this variation is driven by the package internal heat load and the storage position, but these variables do not explain all the scatter. The primary unknowns are the initial moisture content of the fiberboard assembly, and the equilibrium level to which moisture from fiberboard degradation will build.

Some efforts have been performed or are in progress to develop an improved understanding of the fiberboard moisture environment within the 9975 drum in storage. KAC personnel collect fiberboard moisture data during field surveillance activities. These data should help understand the actual range of moisture conditions among the many packages in storage. Humidity readings are also taken within the package during field surveillance. However, these data are less useful since the package is moved from its storage location prior to measuring relative humidity, and any change in the ambient temperature around the drum will alter the humidity equilibrium. The relative humidity within a limited number of packages still in the storage environment has been measured [9]. Finally, laboratory tests have characterized the humidity profile within packages with internal heat loads to show the range of environments that might exist within a package for a given moisture level and heat load [6, 7]. Note, however, that a wide range of potential moisture conditions are possible, with a corresponding range of fiberboard degradation behaviors. The actual moisture content of most packages is unknown.

In the laboratory testing of fiberboard samples, there are two contributions to property changes – immediate, reversible effects due to change in moisture content, and long-term irreversible changes due to degradation. When fiberboard is placed in an environment, there may be a change in moisture content as the sample comes to equilibrium with the environment (typically within ~1 day for smaller samples, or after several weeks for a full assembly). Reversible changes likely to occur due to moisture change include the following.

- Thermal properties (thermal conductivity and specific heat capacity) decrease as the moisture content decreases. This effect is reported in the literature [10] for wood products (and by extension is applicable to fiberboard) and is observed in the laboratory data.
- The general trend for compression tests in the perpendicular orientation is increasing energy absorption as moisture content decreases, and decreasing energy absorption as moisture content increases.
- Physical properties (weight, density, dimensions) all decrease as moisture content decreases.

Table 3 summarizes short-term (initial) physical property changes observed in the various environments. The weight changes are generally consistent with an initial moisture content of up to 10 wt%.

In addition to short-term moisture effects, longer term changes may occur as a result of degradation. The literature identifies that slow pyrolysis occurs at modest temperatures [11]. In addition to water vapor, compounds from pyrolysis are evolved at temperatures as low as 203 ºF. This is strongly evidenced by samples conditioned at 250 ºF, with an immediate weight loss of 8-10% (moisture loss), followed by an additional 15 – 20 %/year weight loss. At the higher temperature and humidity levels, the samples also change visually. The samples darken, and the coarse fibrous appearance changes to a finer particulate texture. The aging models that are discussed below deal with long-term degradation rates. They do not include the short-term effect of initial moisture change.

There may be sources of degradation to the fiberboard that are not captured in the above testing. For example, a limited number of 9975 packages have been removed from service and found to contain mold or were infested with drugstore beetles. The identified scope of beetle infestation to date is 3 packages. However, the possibility of additional / future infestations exists. On the other hand, mold spores are ubiquitous, and mold growth can be expected whenever the environmental conditions are favorable.

Laboratory testing has observed mold growth at high humidity (approximately 100% RH) with temperatures of approximately 50 and 77 ºF. Mold was not observed on samples at approximately 100% RH and 125 ºF, indicating a modest temperature increase beyond ambient may be sufficient to limit or prevent the growth of mold. However, given the tendency for moisture in the fiberboard to migrate toward the cooler regions of the package, packages with higher internal heat loads are more likely to develop conditions conducive to mold growth on the outer, cooler surfaces of the fiberboard.

Mold has been observed in at least 11 9975 packages in service in K Area. In one case (9975-01903), small patches of mold were observed near the bottom of the lower fiberboard assembly. The fiberboard moisture content was 11 – 18 %WME, with readings around 17 %WME near the mold [12]. An extreme example of mold associated with apparent water intrusion was observed in package 9975-01819. Moisture levels were elevated throughout the fiberboard (16 %WME on the ID, 20 – 26 %WME on the OD, and the bottom ~2 inches were saturated) [13]. The specific impact of mold on fiberboard properties or package service life has not been examined, and is not addressed in this report.

Data for Modeling

In order to combine data from multiple samples, the data were first normalized to the first conditioned value. All samples from a given environment were then averaged to better represent the overall bulk behavior of the fiberboard assembly. For physical properties and thermal conductivity, the degradation rate was calculated for each sample, and the rates for each sample within the environment were then averaged. For compression test results, a single degradation rate was fit to all the samples from a given environment, since each compression test sample provides only a single data point. A model has not been developed for specific heat capacity due to the relatively large scatter in that data and the small degradation rates observed to date.

For each property, there may be a different combination of source packages represented in each environment. With some degree of package-to-package variation, this may lead to a varying bias from the different environments. However, there are generally insufficient data from a single source package among all environments to develop a complete degradation model for each source package, so the available data from all sources are averaged.

The degradation models for thermal conductivity are based on the data taken at a mean temperature of 25 ºC only. It is observed that the thermal conductivity at the higher mean temperatures (50 and 85 ºC) degrades at comparable rates.

Data for some properties show a varying degradation rate. For example:

- The rate of weight loss at 250 ºF decreases somewhat after about 25% weight loss, and again after about 45% weight loss (Figure 6). Similar changes in the degradation rate are seen at 215 ºF, 185 ºF 70 %RH, 185 ºF30 %RH and 160 ºF 50 %RH after ~15 - 35% weight loss.
- The rate of decrease in length / width at 250 º F and 185 ºF 70%RH increases after ~3-5% decrease. Similar rate changes are not obvious yet in other environments.

- Thermal conductivity degradation rates decrease after ~30% loss at 250 ºF, but don't yet show significant variation in other environments.
- Energy absorption degradation rates decrease after ~70% loss at 250 ºF. Similar but less pronounced behavior is suggested at 215 ºF and 185 ºF 70 %RH.

These observations suggest the following possibilities:
- Fewer changes in the degradation rates in the milder environments likely reflect the fact that the samples in these environments generally haven't yet reached the degree of degradation experienced in the more aggressive environments.
- These changes occur at different degrees of degradation, depending on the property and environment. Arrhenius theory and time-temperature superposition (TTS) would suggest that changes should occur at a similar degree of degradation if temperature were the only difference between environments. But the moisture content is also changing between the more aggressive environments, which could change equilibrium reaction rates at different stages of degradation.
- While fiberboard is described as being composed primarily of cellulose, most woody tissues actually contain three polymeric compounds: cellulose, hemicellulose and lignin [11]. It is postulated that the degradation of cellulose, hemicellulose and lignin occurs at different rates with different chemical reactions. The initial degradation rate reflects primarily the behavior of the compound that is degrading fastest. As that compound starts to deplete, the overall degradation rate shifts based on the behavior of the remaining compounds.

To enable a fair comparison of degradation rates across the aging environments, it is important to look at data from within the same degradation regime. Therefore, only data prior to the initial change in slope should be considered in developing degradation models. For the milder environments that display only one regime, all the data from these environments can be considered. The degradation models are therefore applicable only below a threshold that maintains degradation behavior within this regime. The excluded data from the more severe environments can be considered to estimate behavior beyond this regime if such behavior becomes relevant to the 9975 packages in storage. Fortunately, storage conditions are typically much milder that these more severe aging environments.

<u>Modeling Approach</u>

Each of the polymers in woody tissue (cellulose, hemicellulose and lignin) is chemically different, but it is expected that thermo-oxidative degradation of fiberboard might progress in a manner similar to that of other polymers, such as EPDM or Viton O-ring materials. This includes an expectation of Arrhenius behavior over temperature regimes with a consistent degradation mechanism(s). With Arrhenius behavior, the degradation rate varies exponentially with inverse temperature, and this temperature dependence is described by an activation energy:

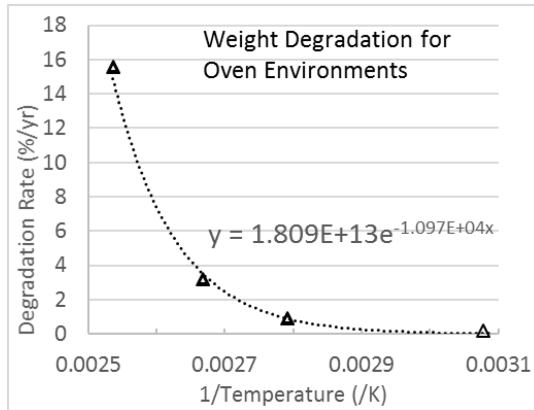
Rate Constant = $A * \exp(-E_a / RT)$

Where: A is a constant
E_a is the activation energy
R is the ideal gas constant, and
T is the absolute temperature

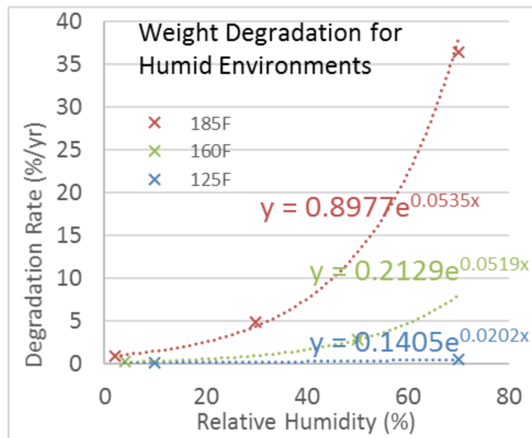
For the data of interest (restricted to the initial ~constant degradation rate), the following steps are taken to develop a degradation model. Specific steps are illustrated for the change in weight.

1. The data are normalized to their initial conditioned value, to show the relative decrease in each property over time (see Figure 6 for normalized weight change).

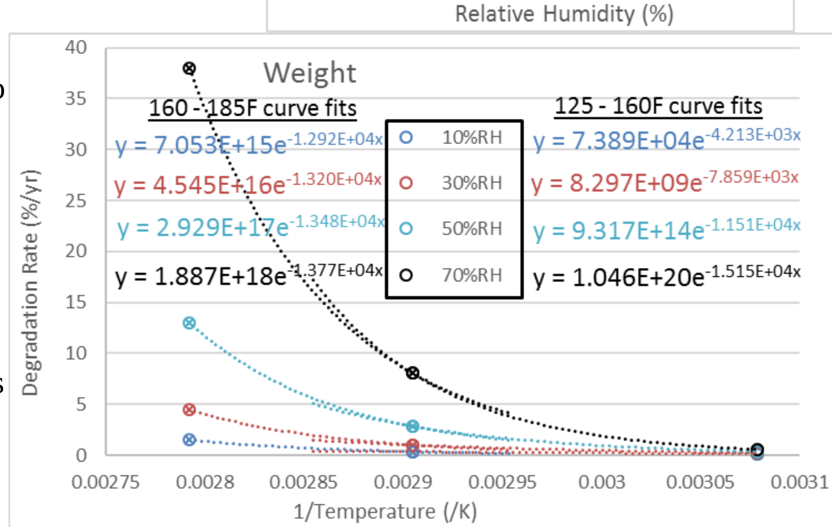
2. A curve is fit to the degradation rate vs reciprocal temperature for the 185, 215 and 250 °F oven environments. Consistent with Arrhenius behavior, an exponential relationship is used. A better curve fit is obtained by excluding the 125 °F degradation rate, possibly because this is close to the apparent threshold for degradation and the degradation mechanisms may be changing. Despite this exclusion, the curve fit generally comes close to matching the 125 °F value, indicating the validity of extrapolating this curve fit to lower temperatures.



3. A curve is fit to the degradation rate vs relative humidity for constant temperatures of 125, 160 and 185 °F. An exponential relationship provides a good fit to the three data points at 185 °F, and will therefore also be used for the other temperatures. Two data points are available at 125 °F, and a second data point at 160 °F is obtained by interpolation of the oven environments correlation.



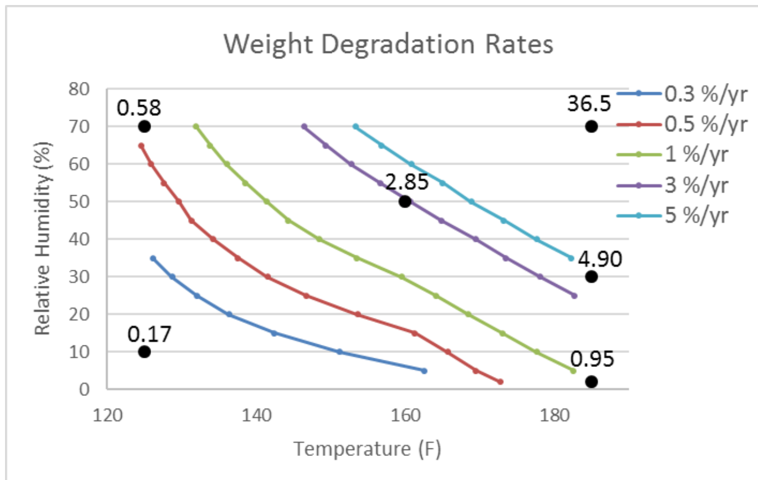
4. For a given value of relative humidity, exponential curves are fit to the interpolated values from the previous step. (With temperature the only variable in this step, Arrhenius theory should again apply, although the activation energy may differ from that for the oven environments.) To reduce additional deviation from the original degradation data rates, curves are fit separately for the 125 – 160 °F and 160 – 185 °F regions. This is performed in 5%RH increments from 10 to 70 %RH, although the figure illustrates only four humidity levels.



5. Using the curve fits for a given relative humidity (from 10 to 70%) in the previous step, the degradation rate can be calculated for any desired temperature between 125 and 185 °F. Since both correlations for a given relative humidity level are equally valid at 160 °F, the results of both correlations are averaged at this temperature.

6. Based on ambient laboratory conditions of ~40 – 50 %RH, the 125 °F oven is at ~10 %RH, and the 185 °F oven is at ~2 %RH. The correlation for the oven environments in step 2 is applicable to these environments below 10%RH. Specifically, this correlation is used to give the approximate degradation rate for ~5 %RH between 156 and 173 °F, and the approximate degradation rate for ~2 %RH between 174 and 185 °F. Between 174 and 185 °F, a weighted average of the degradation rates for 2 %RH and 10 %RH is used to estimate the degradation rate at 5 %RH.

7. For the developed array of degradation rates as a function of temperature and relative humidity, those environments that correspond to a given degradation rate are identified by contour lines. The degradation rate for environments not represented by a specific contour line can be estimated through interpolation.



Degradation Models

Using the approach described above, aging models have been constructed based on the observed changes in several fiberboard properties. These include weight, density, dimensions, thermal conductivity (axial and radial) and energy absorption (area under the stress-strain curve to 40% strain). These models are based on the average behavior of all samples, from both cane and softwood fiberboard, and do not reflect any variation among packages or samples.

In following this approach, there were several specific details and exceptions to note:

- Since the physical property samples are measured many times, there is opportunity for material loss (i.e. reduced weight and dimensions) due to handling. Several control samples (maintained at ambient conditions) are also tracked (Figure 10, for example), and any change in their properties can be used to adjust the observed changes in the samples from the aging environments. The adjustment reflects the average change from 6 control samples. A seventh control sample was not used due to its relatively short duration (3.2 years), and significant seasonal scatter produces a large uncertainty in average behavior over the short term. Note that samples aging in the more aggressive environments become the most fragile, and will lose more material from handling than the other samples. Therefore, this adjustment will conservatively under-predict any handling-related effects in these samples.

- For the physical property samples aging in the 125 ºF oven, the average change in length and width is positive (i.e. negative degradation rate). Since an exponential fit does not apply for values that change between positive and negative, a degradation rate of 0.0085 %/year was assumed for this environment. This value is obtained from extrapolating the curve fit for the other oven environments. This approach was also applied to the 125 ºF oven energy absorption data; the positive change was changed to 0.041 %/year degradation rate.
- The degradation rate for thermal conductivity (both radial and axial) at 125 ºF 70%RH is less than that for the 125 ºF oven. Although the rates for both environments are low, this trend is opposite from that observed for the other properties. Therefore, the higher degradation rate observed for the 125 ºF oven is applied to all humidity levels at this temperature.

The aging models are shown graphically in Figures 11 – 17 for weight, height, length/width, density, thermal conductivity (axial and radial orientations) and energy absorption (perpendicular orientation). Each of these models was developed through the same process described above for weight. Specific data correlations and curve fits used to develop each aging model are shown in the Appendix.

A further check on the model predictions for weight comes from the thermal conductivity samples. The weight of these samples was measured periodically, but was not used in developing the physical property models. These samples therefore present a set of independent data for comparison. Trends for the change in weight of thermal conductivity samples compared to the physical property samples and model predictions are shown in Table 4 for several environments most relevant to storage conditions. Thermal conductivity samples have similar or smaller weight changes compared to physical property samples under the same environment. Since they are measured less frequently, these samples experience less bias from handling.

The limiting need for fiberboard compressive strength is the postulated forklift impact event in KAC. In this scenario, an impact of the forklift tine near the elevation of the containment vessel closure can compromise the containment vessel leak-tight seal without sufficient energy absorption by the fiberboard. As a significant moisture gradient develops in the fiberboard, some of the moisture migrates toward the bottom of the package, with the result that the fiberboard near the seal elevation is relatively drier and stronger.

The property limits are developed as bulk average properties. It is judged that even if local surface regions were to degrade at a significant rate, the overall average rate of change in the bulk fiberboard property may still be low. This judgement is supported by observation of packages removed from service after up to 13 years storage in KAC. Examination of these packages has shown a range of fiberboard properties (density, thermal conductivity, specific heat capacity and compression strength). With one exception, these properties are consistent with that of un-aged fiberboard, with no evidence of significant degradation. The one exception was for 9975-02644, which was in storage for 9.6 years and had the highest heat load (16.5 watts) of any destructively examined package.

Additional data continue to be collected for each property, following successive conditioning intervals. In time, the models will be re-visited based on the additional data, and revised service life predictions can be developed. Note, however, that since the compression tests are destructive, most of the available samples being aged have been tested, and relatively little additional compression test data will become available in the future.

Fiberboard Degradation Activation Energy

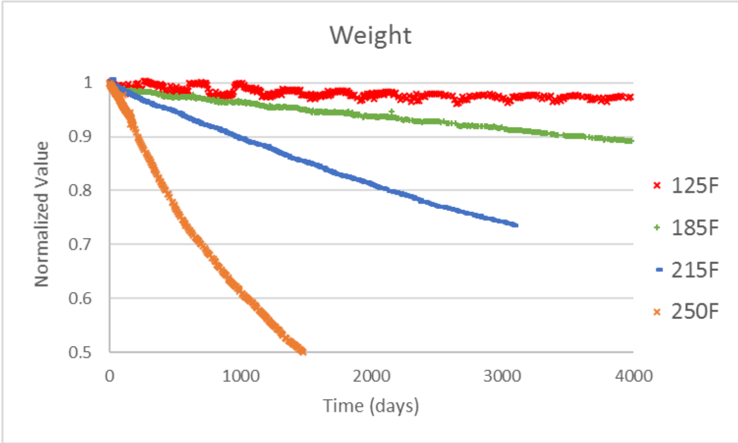
As discussed above, the temperature dependence of fiberboard degradation can be described by an activation energy. In the models developed above, the activation energy for fiberboard in nominally dry oven environments is estimated for each property using the average degradation rate from three oven environments (185, 215 and 250 ºF). Similarly, the temperature dependence of fiberboard degradation at a specific value of relative humidity can be examined. Using the degradation models, the estimated degradation rate at varying temperatures for a constant relative humidity of 30%, 50% and 70% have been compiled. At each humidity level, an Arrhenius relationship can be used to estimate the activation energy (Figure 18).

It is observed from Figure 18 that a single slope describes the temperature dependence for some properties at some humidity levels, and two slopes are needed in other cases. Where more than one slope is observed, the slope for the lower temperature regime (closer to KAC storage conditions) is used. The activation energy estimates for each property and each humidity condition are summarized in Table 5.

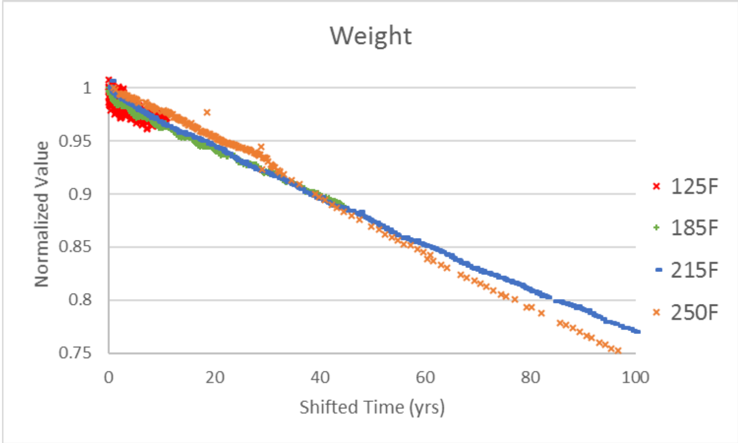
The activation energy is observed to change for varying levels of relative humidity. In general, this apparent activation energy increases as the relative humidity increases. It is appropriate to look at this activation energy for constant values of relative humidity when considering the current data since that is the parameter that is measured and for which the degradation models were developed. However, this does not represent a true activation energy since the concentration of water in the air (and therefore in the fiberboard) changes with temperature for a constant value of relative humidity. In other words, this apparent activation energy is describing how the reaction rate changes with temperature combined with a change in the concentration of moisture present. An estimate of the true activation energy (describing the effect of temperature change only) can be obtained by looking at degradation rates for constant values of absolute humidity. This is illustrated in Figure 19 for fiberboard weight change at an absolute humidity of 40 g/m^3. The average behavior over the full temperature range follows an activation energy of 38400 J/mol.

The activation energy estimates in Table 5 were developed using average degradation rates from each aging environment. The activation energy can also be estimated with an alternate approach that draws from all the individual data points. With time-temperature superposition (TTS), if the shape of the degradation curve is similar across a temperature range, then the degree to which the time scale needs to be shifted to align the different degradation curves is proportional to the activation energy. For simplicity, this approach is illustrated for the weight change of a typical sample from each temperature. This analysis also includes data from the 125 ºF oven.

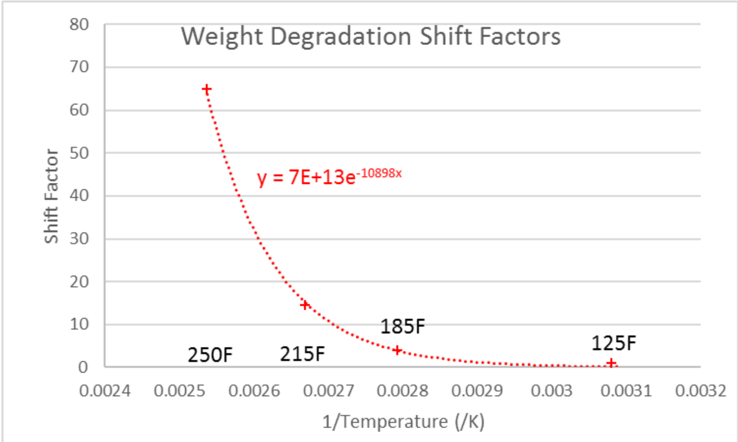
1. The data for each temperature are plotted on a single graph. As before, the focus is on the initial portion of the curve with ~constant degradation rate, although the remainder of the curve can be considered as well if multiple temperatures show the same shape curve.



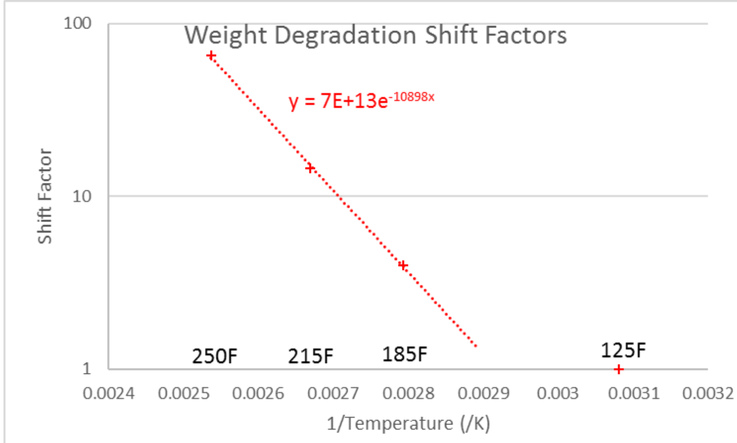
2. By trial and error, the time scale for each sample is multiplied by a shift factor until the curves all superimpose on each other (with the focus still on the portion of each curve displaying the initial degradation rate). Note that due to varying initial normalized values, the curves may not exactly superimpose, but they do share the same slope.



3. The shift factors used to adjust each curve are plotted as a function of reciprocal temperature, and the reciprocal temperature coefficient in this relationship is proportional to the activation energy.



4. The exponential curve fit to the shift factors is based on the three highest temperatures only. While the curve appears to also fit the 125 °F point when plotted on a linear scale, shifting to a log scale shows that this point does not fit with the other data in an Arrhenius relationship. This likely results from changing degradation mechanism(s) at the lower temperature near the degradation threshold.



5. Multiplying the reciprocal temperature coefficient by the ideal gas constant (8.3145 J/mol-K) gives the activation energy, which can be compared with values calculated in the models from the average degradation rates.

Coefficient from shift factors = 10898 K
Activation energy = 90611 J/mol

Coefficient from weight degradation model = 10970 K
Activation energy = 91210 J/mol

The same process was performed for several other properties (energy absorption was not considered due to the small number of individual data points and relatively high degree of scatter, and density was not considered since that property is derived from the weight and dimensions which are separately considered). For each of these cases, the activation energy can be compared to the value obtained from the corresponding degradation model. The shift factor curves for these properties, and corresponding activation energy estimates, are shown in Figure 20. The activation energy values obtained from this figure are generally in good agreement with those from the degradation models (Table 5). The one exception is with radial thermal conductivity, for which the TTS-based estimate is significantly higher than that from the degradation model.

Comparison and Relevance to Literature Data

The literature includes studies of the thermal degradation of biomass, such as wood, to determine kinetic parameters and decomposition products, with the goal of understanding and modeling their behavior in a fire. Researchers showed the kinetic effects of individual components (i.e. cellulose, hemicellulose, and lignin) are additive by comparing thermogravimetric curves of biomass and synthetic biomass (mixture of components) [14]. They obtained reasonable models of thermal degradation and product distribution by weighted correlations of each component's degradation. Their findings indicate cellulose will predominantly influence thermal degradation reactions of softwood and sugarcane, composed of 40-51 wt. % and 27-54 wt. %, respectively, of cellulose [15]. Hemicellulose will affect thermal degradation reactions due to its low thermal stability, while lignin will minimally affect reactions due to its slow pyrolysis rate [16, 17]. LeVan demonstrated that the thermogravimetric analysis (TGA) curves of holocellulose, composed of cellulose and hemicelluloses, overlapped well with a softwood sample [17].

Literature values of the activation energy of cellulose are reported around two different ranges, 109-151 kJ/mol [16, 18, 19] and 225-250 kJ/mol [R16, 20, 21]. The lack of consistency in values is because the determination of kinetic parameters is highly dependent upon experimental conditions (e.g. heating rates, atmosphere, sample source and size), which varied between studies. Literature values of activation energy for sugarcane and softwood in nitrogen are 168 and 161 kJ/mol, respectively [22], and 151 and 150 kJ/mol, respectively [23]. The sugarcane and softwood samples in the studies were composed of similar wt. % of cellulose, and hemicellulose, and within the range of fiberboard samples in 9975 packages [15]. These comparable values for activation energy in the literature corroborate the study's findings that cane and softwood fiberboards have similar thermal degradation characteristics.

SRNL determined the activation energy is ~91 kJ/mol for fiberboard samples aged in a dry oven by measuring weight loss, and the literature reported ~150-170 kJ/mol for wood samples in nitrogen using TGA. This agrees with the literature trend for activation energy to be lower in air than in inert atmosphere; the reported activation energy of cellulose is 71 kJ/mol in air and 155 kJ/mol in a nitrogen atmosphere [18]. Stamm observed thermal degradation of wood is greater in the presence of air and water with significantly lower activation energy under steam (66 kJ/mol) than under dry conditions (123 kJ/mol) [19]. The impact of water on fiberboard was also observed in SRNL's study as the calculated activation energy is ~38 kJ/mol at absolute humidity of 40 g/m^3. Overall, the activation energies calculated from SRNL aging studies are consistent with literature results and trends (humid air < air < inert atmosphere). As lower activation energy means faster rate when comparing the same reaction, the rate of thermal degradation for fiberboard samples is highest in humid air.

Impact on 20 year Storage Life

KAC has recently completed thermal and structural calculations [24 – 27] that supported extending the service life of 9975 packages in storage from 15 years to 20 years. These calculations addressed the potential for degraded fiberboard properties following up to 20 years in storage using degradation models developed previously. The maximum fiberboard temperature based on a bounding average ambient temperature is 146 °F at beginning of life, and decreases to 145 °F after 20 years. The following degraded conditions were assumed in these calculations for both cane and softwood fiberboard:

- Density, dimensions and thermal properties were reduced by 10%, representing an average degradation rate of 0.5 %/year. Additional fiberboard loss beyond this 10% was assumed in the calculations to address the possible effects of mold in the bottom fiberboard layers and fiberboard compaction leading to an increased axial gap.
- For structural calculations, the fiberboard strength (stress-strain curve) was reduced by zones. In the hottest zones (with fiberboard temperatures up to ~145 °F), the strength was reduced by either 33% or 40%, representing an average degradation rate of 1.6 %/yr or greater. In the zone with fiberboard temperatures from ~120 °F up to 140 °F, the strength was reduced by 26%, representing an average degradation rate of 1.3 %/yr. No reduction in strength was assumed in the coolest zone with fiberboard temperatures below 120 °F.

While the relative humidity will vary throughout the fiberboard depending on the local temperature, the absolute humidity tends to remain approximately constant [6, 7]. The relative humidity and temperature within the upper air space was measured within 26 packages while stored in KAC in 2015 [9]. From these measurements, the average observed absolute humidity was 12.6 g/m^3, with a maximum value of 16 g/m^3. While additional humidity measurements are available from field surveillance data, these are not considered here because they were taken after the packages were moved from the storage location and do not reflect the equilibrium storage condition.

Additional evidence of absolute humidity values is found in a series of laboratory tests on packages that were modified to allow humidity measurements throughout the fiberboard [6, 7]. These tests included both cane and softwood fiberboard, and started with either a typical fiberboard moisture content (~8 - 9 wt%) or an elevated fiberboard moisture content (~11 - 13 wt%). All observed absolute humidity values were below 37 g/m^3, except for a single reading of 41.2 g/m^3 near the top of a package with cane fiberboard and 19 watts internal heat load. As the package internal heat load decreased, the equilibrium absolute humidity also decreased. For example, with an internal heat load of 15 watts, the maximum observed absolute humidity was 35.2 g/m^3. Based on this, a bounding absolute humidity of 42 g/m^3 will be assumed for conforming packages in storage. At this moisture level, the relative humidity will vary from 53% at 120 °F to 26% at 150 °F. In Figures 11 – 17, the environments with an absolute humidity of 42 g/m^3 are shown. The fiberboard environment within conforming packages stored in KAC is expected to lie below this line.

By comparing the 42 g/m^3 line in each model (Figures 11 – 17) with the degradation rate contours, the following statements can be made about the behavior of fiberboard maintained below the 42 g/m^3 line:

- The degradation rate for fiberboard dimensions and density is below ~0.4 %/yr for all temperatures up to 160 °F.
- The degradation rate for radial thermal conductivity is less than 0.5 %/yr for all temperatures up to 160 °F.
- The degradation rate for axial thermal conductivity is less than 0.5 %/yr for all temperatures up to ~150 °F, and is approximately 0.5 %/yr between 150 and 160 °F.
- The degradation rate for energy absorption is approximately 1 %/yr or less for all temperatures up to 160 °F. While the data do not directly support extrapolations of the model below 125 °F, no significant degradation is expected below 120 °F, as discussed above.

Since the maximum degradation rates associated with a bounding absolute humidity of 42 g/m^3 and a bounding temperature of 160 °F are within that assumed for the thermal and structural calculations, these updated models remain consistent with the established KAC storage life of 20 years.

Conclusions and Recommendations

Thermal, mechanical and physical property data for cane and softwood fiberboard samples have been summarized following aging in several environments (elevated temperature and/or humidity) for periods up to ~12 years. Most of the aging environments are bounding to the conditions expected within the 9975 shipping package during storage in KAC. Models have been developed from these data to provide estimates of degradation rate under potential storage conditions for several fiberboard properties, including thermal conductivity, energy absorption, weight, dimensions, and density. Additional data continue to be collected to permit future refinements to the models and assumptions.

In contrast to previously developed degradation models, development of the current models recognizes that the primary constituents of fiberboard (cellulose, hemicellulose and lignin) are polymers, and analyzes the data in a manner more consistent with polymer behavior. This includes the assumption of Arrhenius behavior and identifies a characteristic activation energy that describes the temperature dependence of fiberboard degradation. This activation energy is seen to vary as the moisture level changes. Data found in the literature show large variation in the activation energy depending on test method and environment (test atmosphere), but the general trends are consistent with those observed for fiberboard. Specifically, the activation energy decreases, and the temperature dependence of the reaction rate increases as the moisture level (absolute humidity) increases. (This trend is reversed when compared to relative humidity since relative humidity has a built-in interrelationship with temperature.)

Predictions from the degradation models developed in this report have been compared to the degree of degradation assumed in recent calculations that were performed in support of establishing a KAC storage life of 20 years. These calculations relied on previous degradation models and the corresponding estimated degradation rates. For each property addressed in this report, the predicted degradation after 20 years under maximum KAC conditions is less than assumed in these calculations. Therefore, these updated degradation models do not compromise the basis for the 20 year service life in KAC.

Some of the degradation rates and model predictions presented in this report are extreme and do not represent the behavior of the typical package in KAC. The internal heat load and temperature profiles within many packages in storage are such as to produce milder conditions in storage than in any of the aging environments. Many conforming packages with lower internal heat loads are expected to experience no degradation, and should provide a service life beyond the currently approved 20 year storage period. Nevertheless, the possibility of accelerated degradation to a limited number of packages, whether from high heat load, elevated moisture levels, or other conditions, should be recognized. These packages will experience degradation in service and may not perform their required safety functions beyond 20 years.

The assumptions and inputs behind the predictions in this report should be well understood before attempting to estimate an actual service life in KAC. A limited number of 9975 packages have been found with non-conforming conditions (e.g. moldy fiberboard). The analysis and predictions of this report should not be applied to non-conforming packages. Additional efforts would be needed to address the integrity of the fiberboard in such packages.

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Table 1. Summary of maximum sample exposure times prior to testing, for data through November 2017.

Environment	Maximum exposure time (years) through November 2017				
	Thermal Conductivity		Specific Heat Capacity	Comp. Strength (perpendicular)	Physical Properties
	Axial	Radial			
	Cane fiberboard				
250 ºF oven	4.9 [1]	4.9 [1]	5.0	3.7	5.3 [1]
215 ºF oven	11.2	11.2	—	7.4	11.5
185 ºF oven	12.3	12.3	12.1	8.7	11.7
185 ºF 30% RH	8.3	8.3	8.7	7.2	8.7
185 ºF 70% RH	0.4	0.4	0.4	1.2	1.3
160 ºF 50% RH	7.0	7.0	5.2	3.4	7.2
125 ºF oven	none	11.8	11.3	11.4 [2]	11.0
125 ºF 70% RH	5.8	5.8	0.3	3.6	6.3
	Softwood fiberboard				
250 ºF oven	3.6	3.9	4.4	3.4	3.7
215 ºF oven	8.3	8.3	none	8.5	8.8
185 ºF oven	3.1	3.1	none	7.1	9.0
185 ºF 30% RH	7.2	7.2	6.9	5.3	7.4
185 ºF 70% RH	1.4	0.9	none	0.8	1.4
160 ºF 50% RH	6.5	6.5	none	4.8	6.6
125 ºF oven	3.6	3.6	none	2.8	0.3
125 ºF 70% RH	5.8	5.8	none	2.9	6.3

[1] Due to a thermal gradient in the 250 ºF oven, the temperature of the thermal conductivity samples ranged from ~242 – 279 ºF during the first year, and the temperature of the physical property samples was ~236 ºF during the first half-year.

[2] Some of these samples were tested after aging in a 125 ºF oven for the stated period, but also include additional aging time at 125 ºF 70 %RH. No significant degradation has been observed from the aging periods at 125 ºF 70 %RH.

Table 2. Thermal conductivity data at 25 ºC mean temperature for each sample following initial period (typically 2 – 8 weeks) in the aging environment. Variation results primarily from moisture level and sample source package.

Aging Environment	Thermal Conductivity (W/m-K) at 25 °C (77 °F)			
	Radial orientation		Axial orientation	
	Cane fiberboard	Softwood fiberboard	Cane fiberboard	Softwood fiberboard
250 °F oven	0.0838 – 0.1015	0.0882 – 0.0957	0.0503 – 0.0575	0.0492 – 0.0505
215 °F oven	0.0872 – 0.1063	0.0934	0.0518 – 0.0585	none
185 °F oven	0.0868 – 0.1092	0.0952	0.0522 – 0.0595	0.0516
125 °F oven	0.0909 – 0.0955	0.0940	0.0587 – 0.0591	0.0544
185 °F 70%RH	0.0862 – 0.0972	0.0954 – 0.0992	0.0582 – 0.0602	0.0548 – 0.0557
185 °F 30%RH	0.0888 – 0.1138	0.0975 – 0.1056	0.0545 – 0.0624	0.0526
160 °F 50%RH	0.0854 – 0.1144	0.0987 – 0.1032	0.0589 – 0.0633	0.0546 – 0.0554
125 °F 70%RH	0.1002	0.1054	0.0581 – 0.0629	0.0578

Table 3. Change in physical properties during initial transition to aging environment

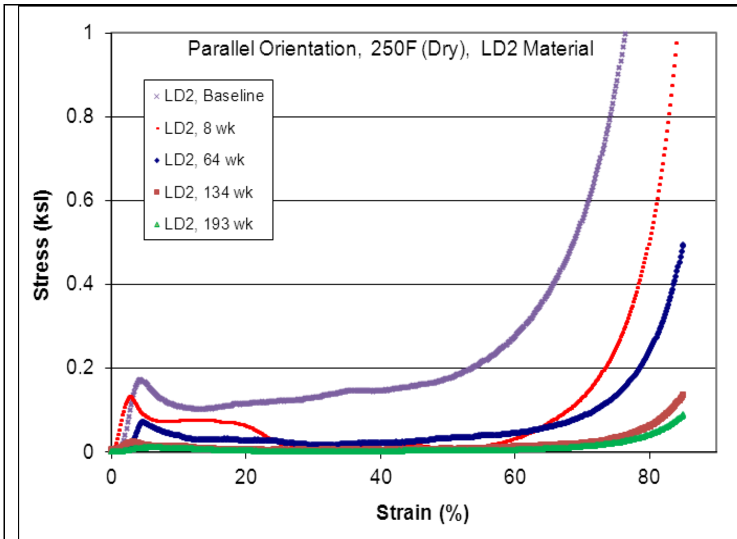
	Approximate initial change in			
Environment	Weight	Density	Height	Length, Width
250 ºF, dry oven	8 – 10% decr	3 – 6% decr	2 - 3% decr	0 - 2% decr
215 ºF, dry oven	7 – 9% decr	3 – 6% decr	0.5 - 3% decr	0 - 2% decr
185 ºF, dry oven	7 – 8% decr	4 – 5% decr	2 - 3% decr	< 1% (+ and -)
125 ºF, dry oven	5 – 6% decr	3 – 4% decr	1 - 2% decr	< 0.5% (+ and -)
185 ºF, 70%RH	< 1% (+ and -)	2 – 6% decr	1 – 3% incr	< 1% (+ and -)
185 ºF, 30%RH	4 – 5% decr	2 – 3% decr	1 - 2% decr	< 0.5% decr
160 ºF, 50%RH	< 1% (+ and -)	3% decr – 2% incr	< 1% (+ and -)	< 0.5% decr – < 1% incr
125 ºF, 70%RH	2% incr	0.5% decr – 2% incr	0 – 3% incr	1% decr – 2% incr

Table 4. Comparison of weight changes for physical property and thermal conductivity samples (averaged over all samples in each environment) to model predictions

Environment	Model Prediction (%/yr)	Average Slope from Actual Data (%/yr)	
		Physical Prop. Samples data through 9-12	Thermal Conductivity Samples
125 °F dry (~10%)	-0.17	-0.17	-0.17
125 °F 70%	-0.58	-0.58	-0.45
160 °F 50%	-2.87	-2.85	-2.72
185 °F dry (2%)	-0.90	-0.95	-0.91
185 °F 30%	-4.48	-4.90	-4.00

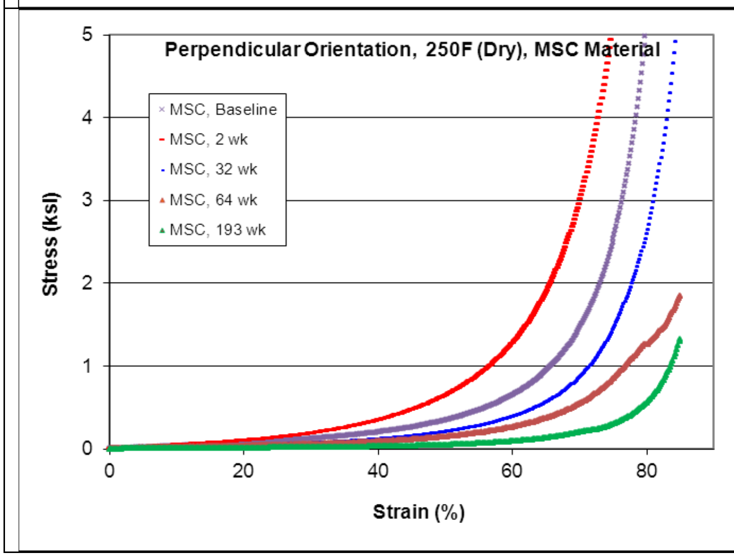
Table 5. Activation energy estimates from the degradation models for dry oven environments and for elevated values of relative humidity.

	Activation Energy (J/mol)			
	Dry ovens (185 – 250 °F)	30 %RH (125 – 185 °F)	50% RH (125 – 185 °F)	70% RH (125 – 185 °F)
Energy absorption	102518	125965	128293	130953
Weight	91210	79570	102518	121059
Height	90794	99774	115904	133697
Length, width	77408	105594	120643	145836
Radial thermal conductivity	85972	89963	124551	161800
Axial thermal conductivity	71937	53687	91709	124385



(a) LD2 material, parallel orientation

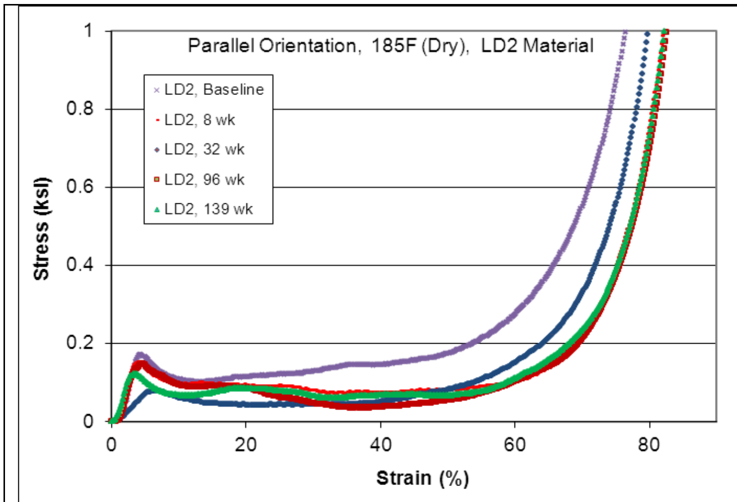
Conditioning Period (wks)	Area under Curve to 40% Strain (ksi)
0	0.0476
8	0.0199
64	0.0110
134	0.0034
193	0.0018



(b) MSC material, perpendicular orientation

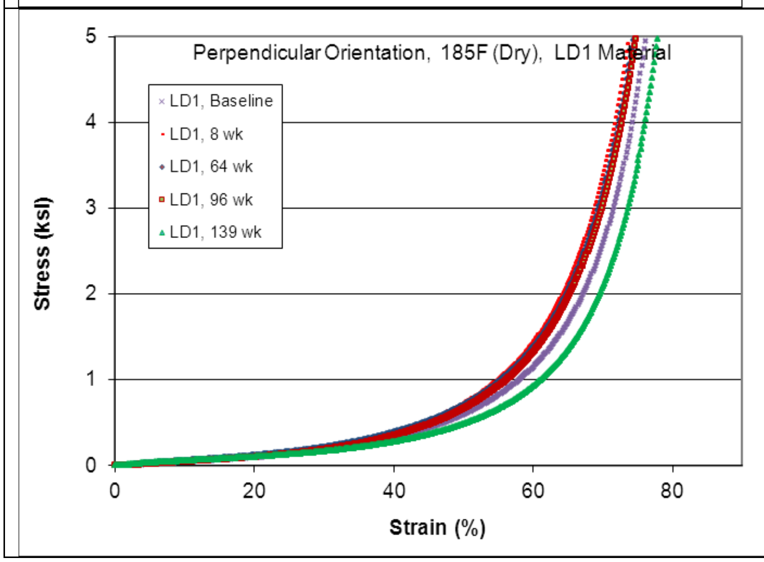
Conditioning Period (wks)	Area under Curve to 40% Strain (ksi)
0	0.0338
2	0.0522
32	0.0205
64	0.0160
193	0.0050

Figure 1. Engineering stress-strain compression curves for select fiberboard samples conditioned and tested at 250 ºF



(a) LD2 material, parallel orientation

Conditioning Period (wks)	Area under Curve to 40% Strain (ksi)
0	0.0476
8	0.0366
32	0.0190
96	0.0291
139	0.0298



(b) LD1 material, perpendicular orientation

Conditioning Period (wks)	Area under Curve to 40% Strain (ksi)
0	0.0461
8	0.0555
64	0.0573
96	0.0494
139	0.0457

Figure 2. Engineering stress-strain compression curves for select fiberboard samples conditioned and tested at 185 ºF

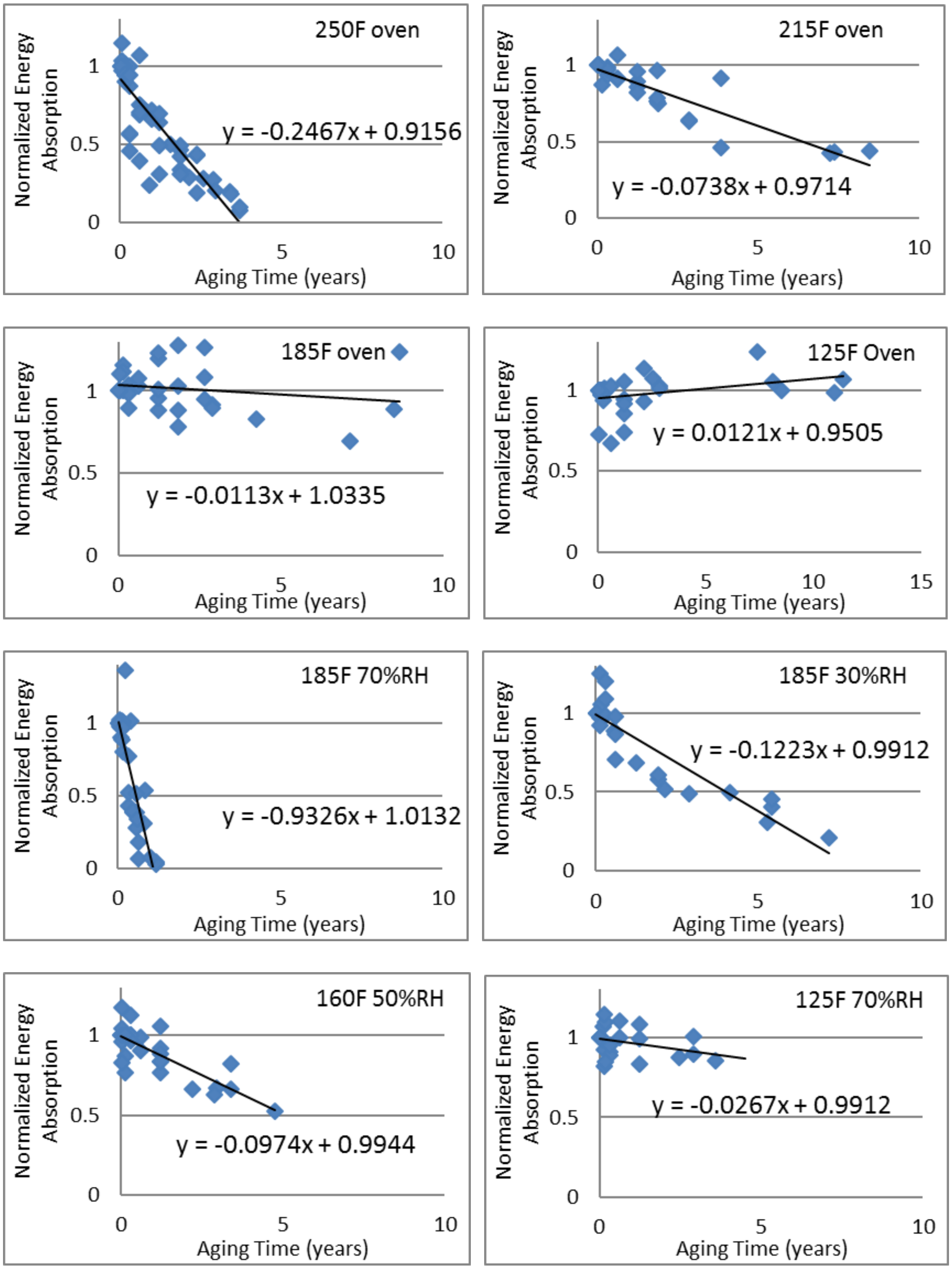


Figure 3. Energy absorption data from perpendicular orientation compression tests. Data includes both cane and softwood fiberboard. Data are normalized to the first conditioned value from their respective source package.

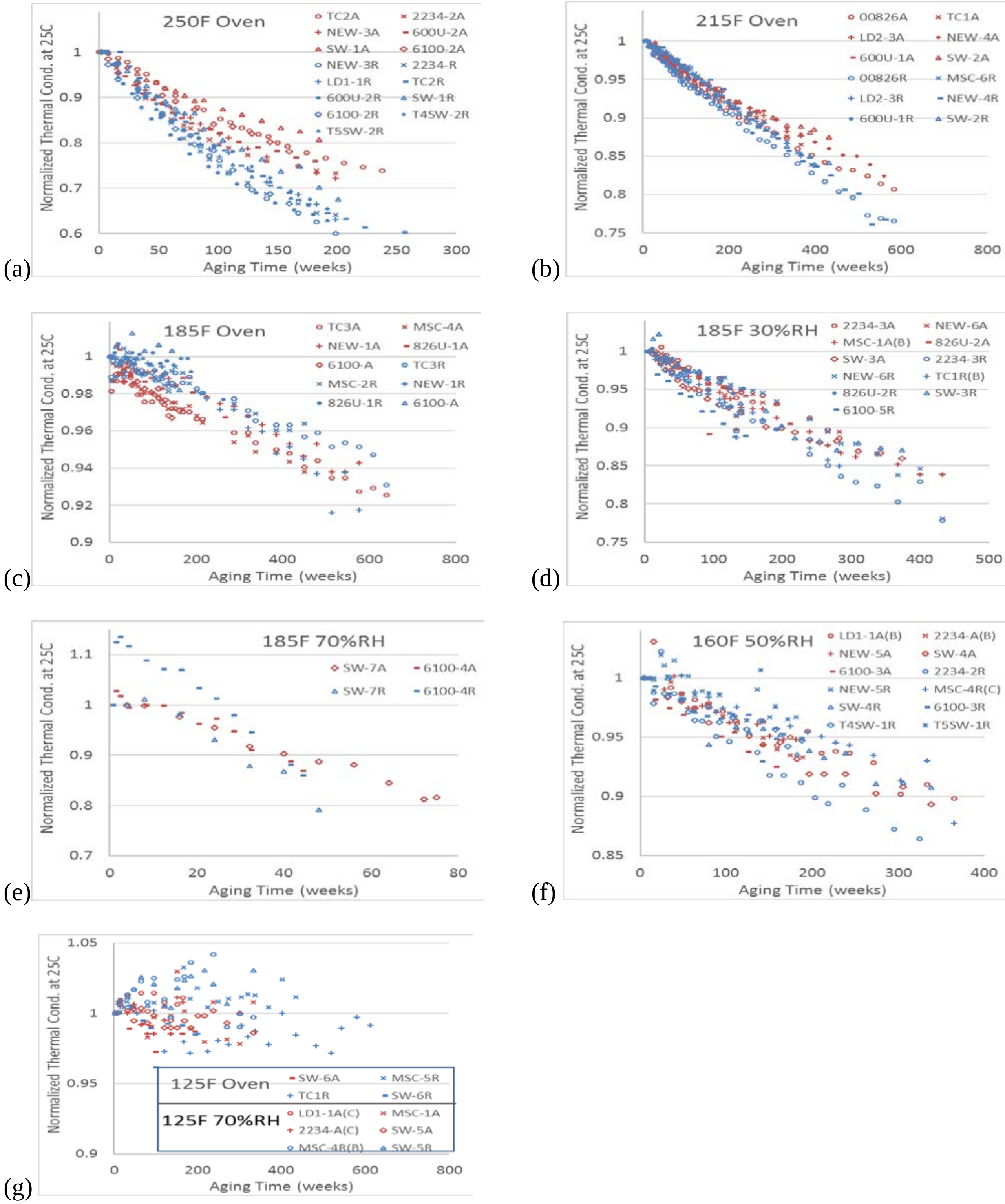


Figure 4. Thermal conductivity data measured at 25 ºC (77 ºF) mean temperature for each conditioning environment as noted. Data for each sample are normalized to the first conditioned value. Axial orientation samples are shown in red, and radial orientation samples are shown in blue.

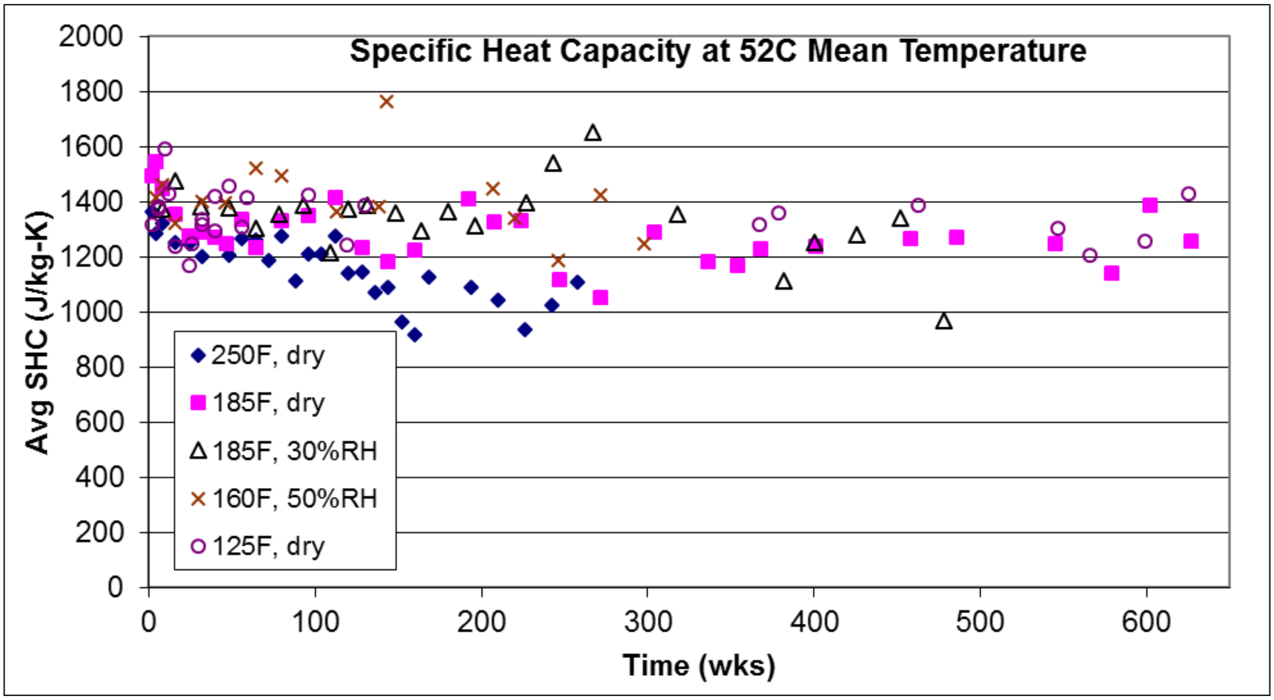
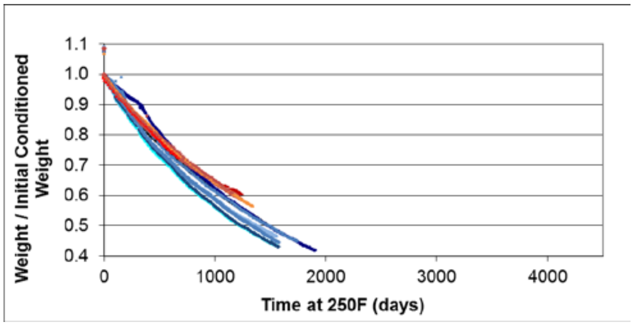
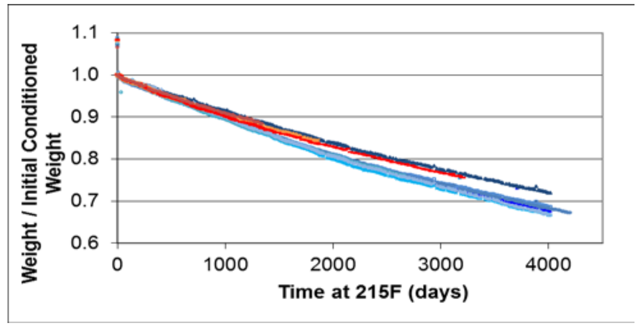


Figure 5. Specific heat capacity data at a mean temperature of 52 ºC (125 ºF) for each conditioning environment. The 250 °F and 185 °F 30% data include both cane and softwood fiberboard, the remaining data are from cane fiberboard only. A linear fit to the data for each environment produces the following trends:

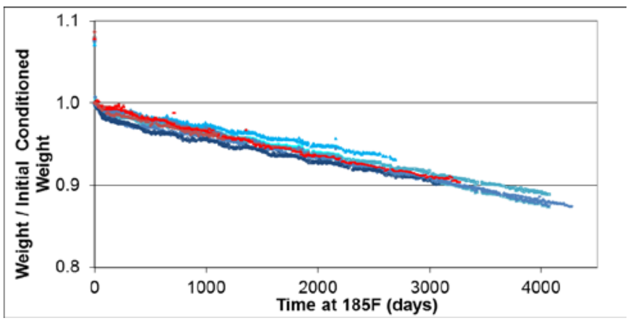
250 °F, dry	SHC (J/kg-K) = 1327.1 – 1.406 * time (weeks)
185 °F, dry	SHC (J/kg-K) = 1335.6 – 0.219 * time (weeks)
125 °F, dry	SHC (J/kg-K) = 1356.9 – 0.0695 * time (weeks)
185 °F, 30%RH	SHC (J/kg-K) = 1418.3 – 0.302 * time (weeks)
160 °F, 50%RH	SHC (J/kg-K) = 1460.3 – 0.389 * time (weeks)



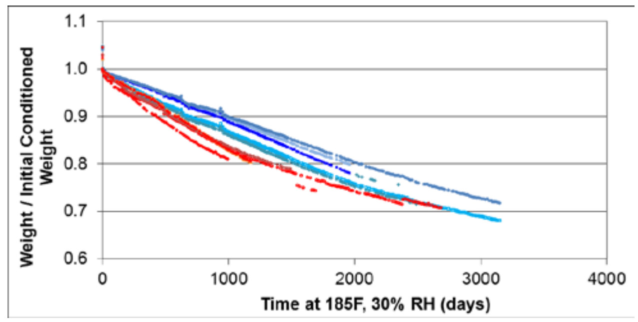
(a) 250 ºF, dry



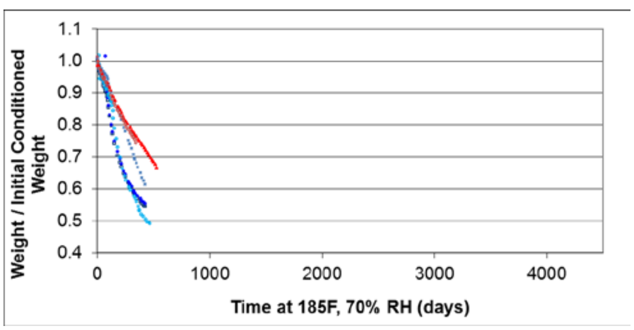
(b) 215 ºF, dry



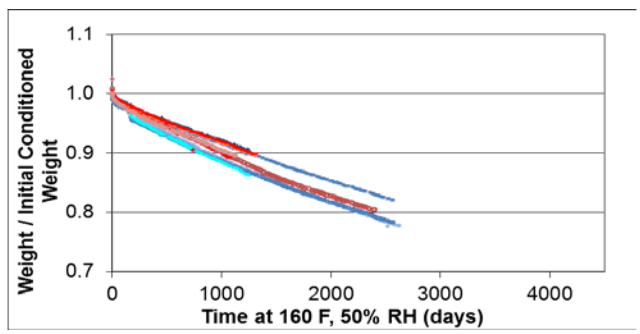
(c) 185 ºF, dry



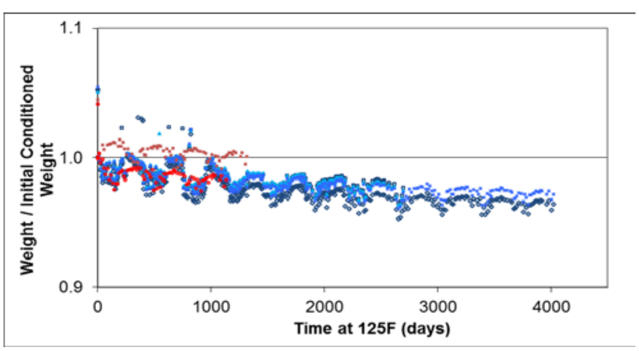
(e) 185 ºF, 30% RH



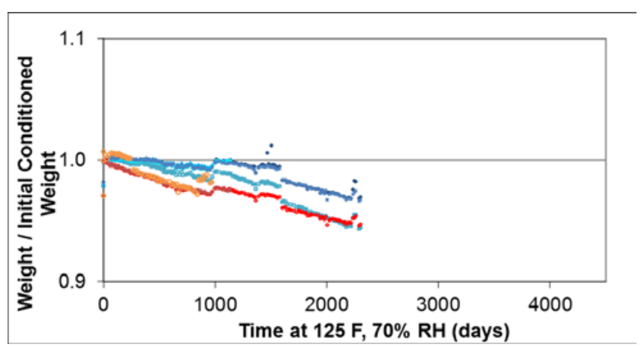
(e) 185 ºF, 70% RH



(f) 160 ºF, 50% RH

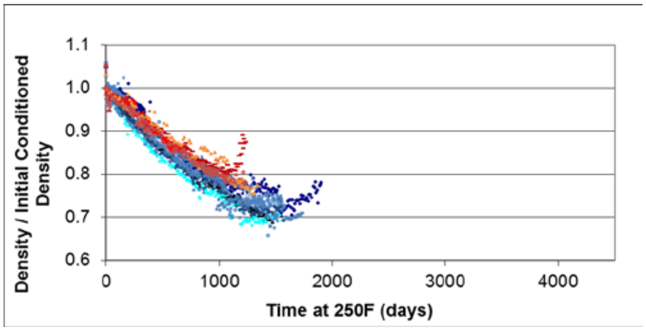


(g) 125 ºF, dry

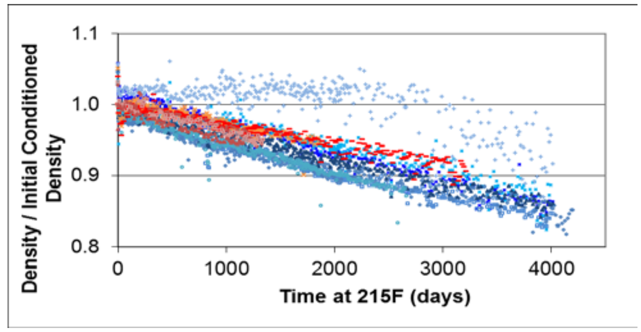


(h) 125 ºF, 70% RH

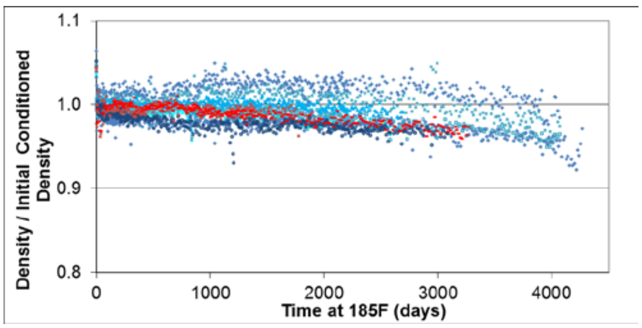
Figure 6. Weight data for physical property samples in the identified environments. Cane fiberboard samples are blue shades, and softwood fiberboard samples are red shades.



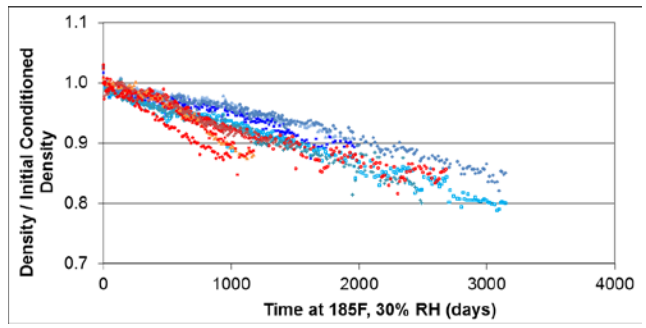
(a) 250 ºF, dry



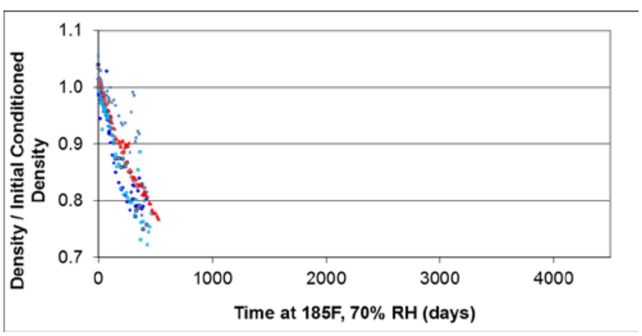
(b) 215 ºF, dry



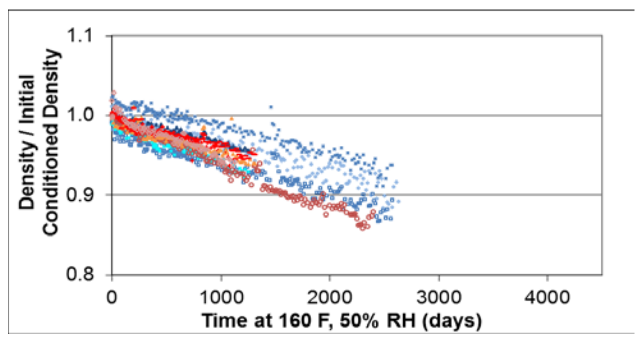
(c) 185 ºF, dry



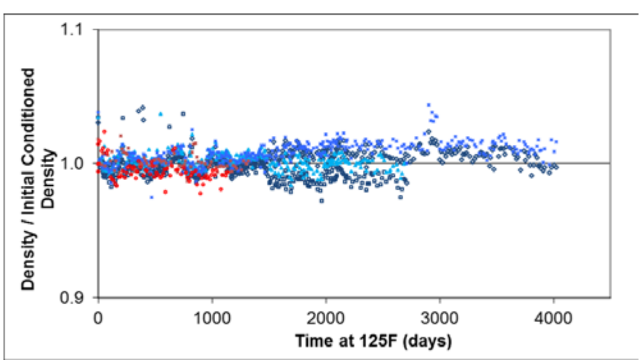
(e) 185 ºF, 30% RH



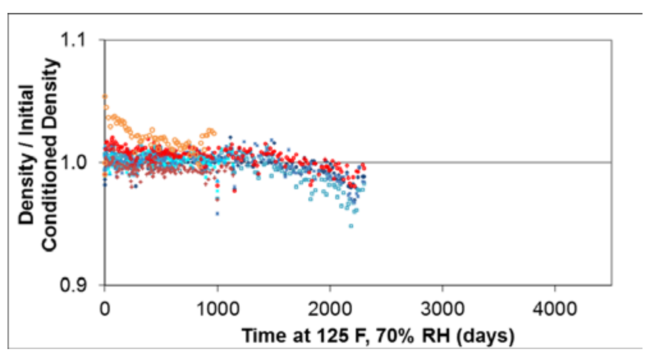
(e) 185 ºF, 70% RH



(f) 160 ºF, 50% RH

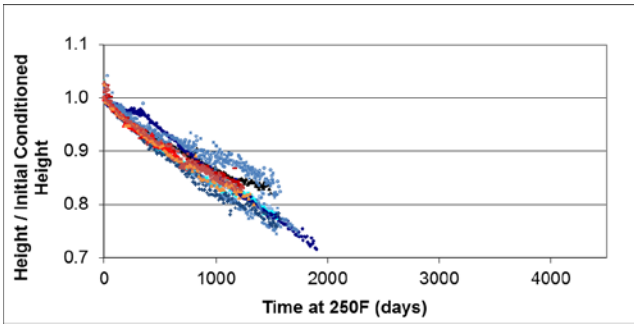


(g) 125 ºF, dry

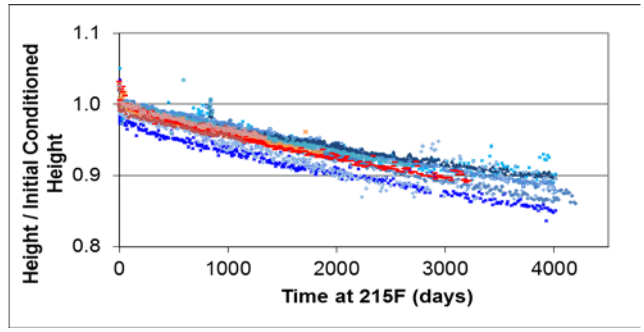


(h) 125 ºF, 70% RH

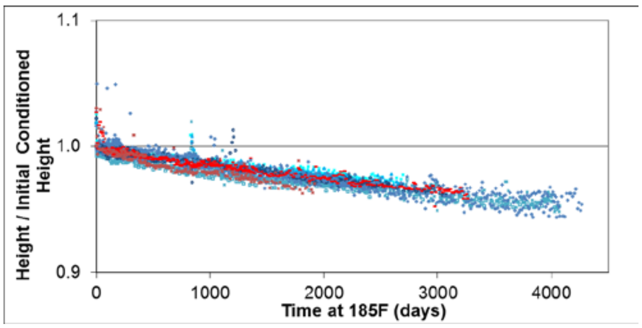
Figure 7. Density data for physical property samples in the identified environments. Cane fiberboard samples are blue shades, and softwood fiberboard samples are red shades.



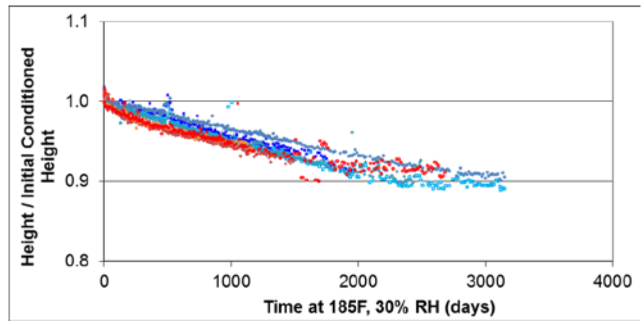
(a) 250 ºF, dry



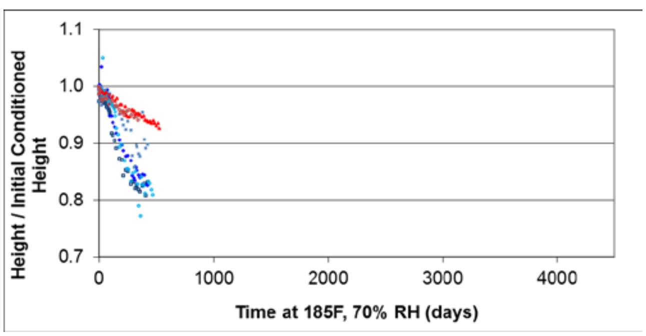
(b) 215 ºF, dry



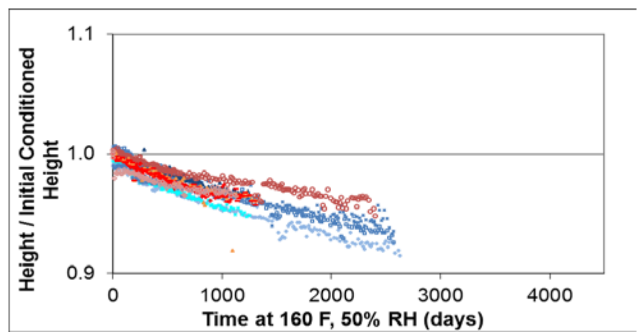
(c) 185 ºF, dry



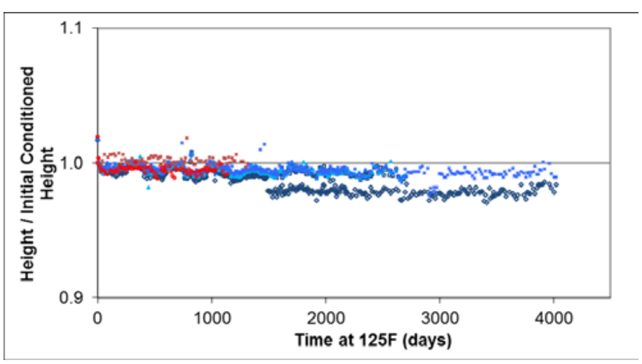
(e) 185 ºF, 30% RH



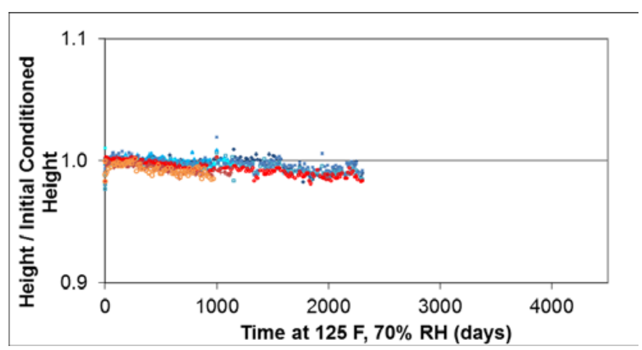
(e) 185 ºF, 70% RH



(f) 160 ºF, 50% RH

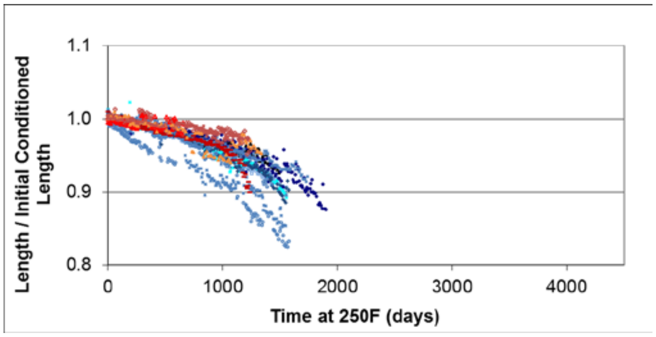


(g) 125 ºF, dry

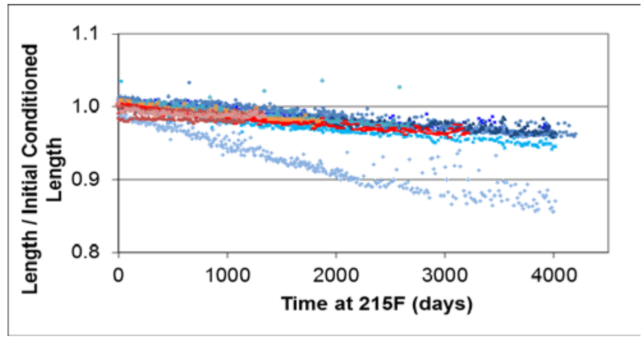


(h) 125 ºF, 70% RH

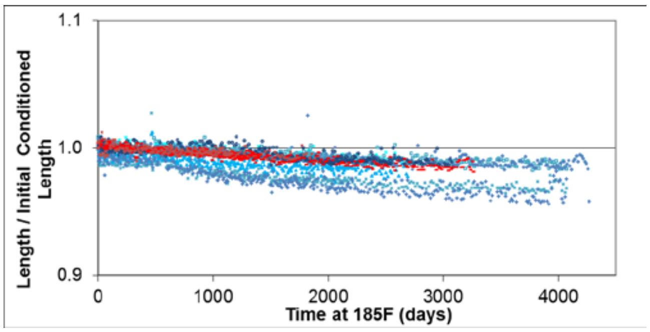
Figure 8. Height data for physical property samples in the identified environments. Cane fiberboard samples are blue shades, and softwood fiberboard samples are red shades.



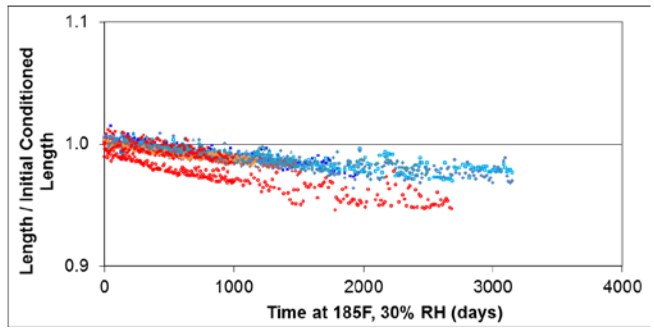
(a) 250 ºF, dry



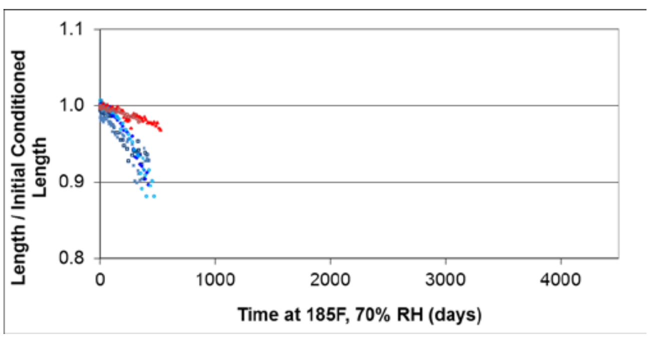
(b) 215 ºF, dry



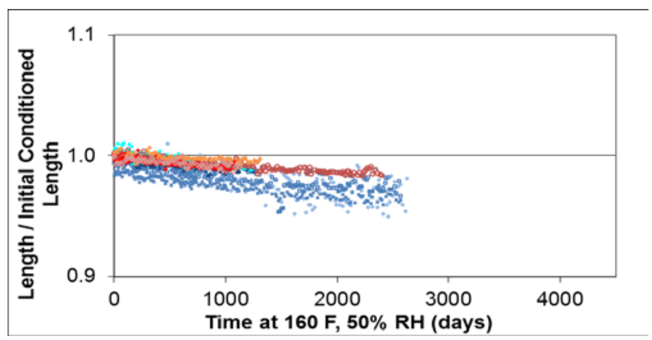
(c) 185 ºF, dry



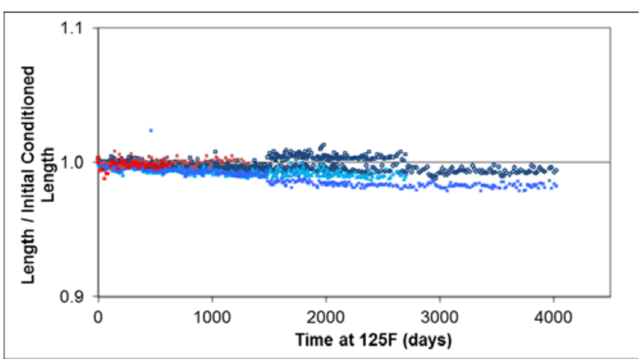
(e) 185 ºF, 30% RH



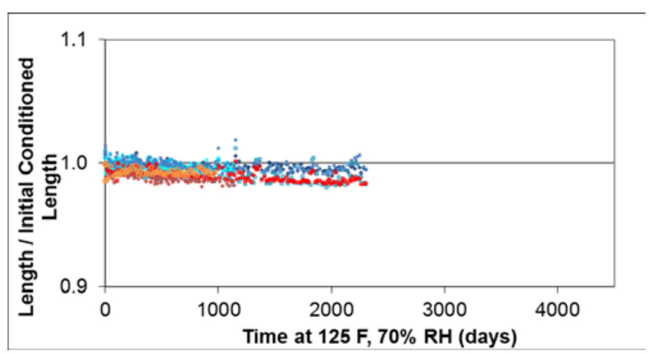
(e) 185 ºF, 70% RH



(f) 160 ºF, 50% RH



(g) 125 ºF, dry



(h) 125 ºF, 70% RH

Figure 9. Length data for physical property samples in the identified environments. (Width data are essentially the same as length data, and are not shown for clarity.) Cane fiberboard samples are blue shades, and softwood fiberboard samples are red shades.

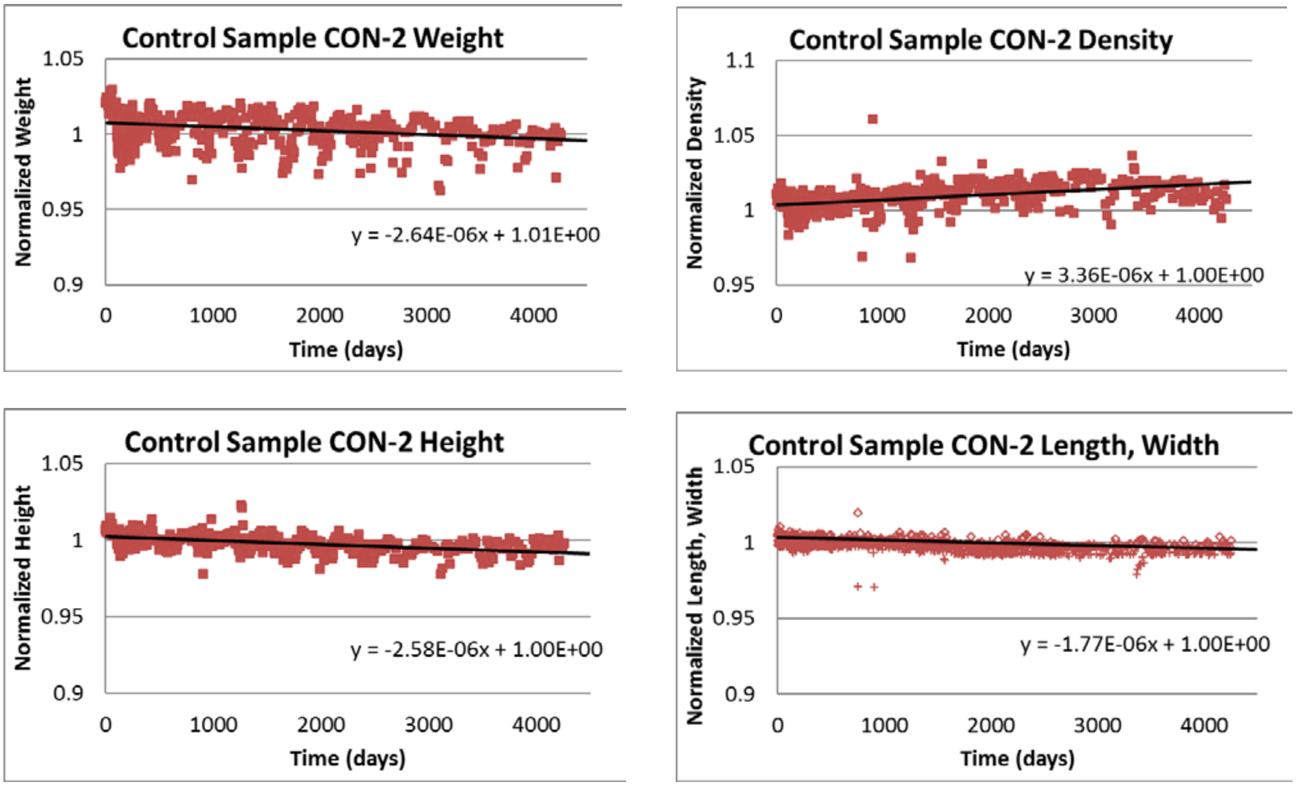


Figure 10. Physical property data for control sample CON-2, showing seasonal variation in properties, and a slight loss of material due to handling.

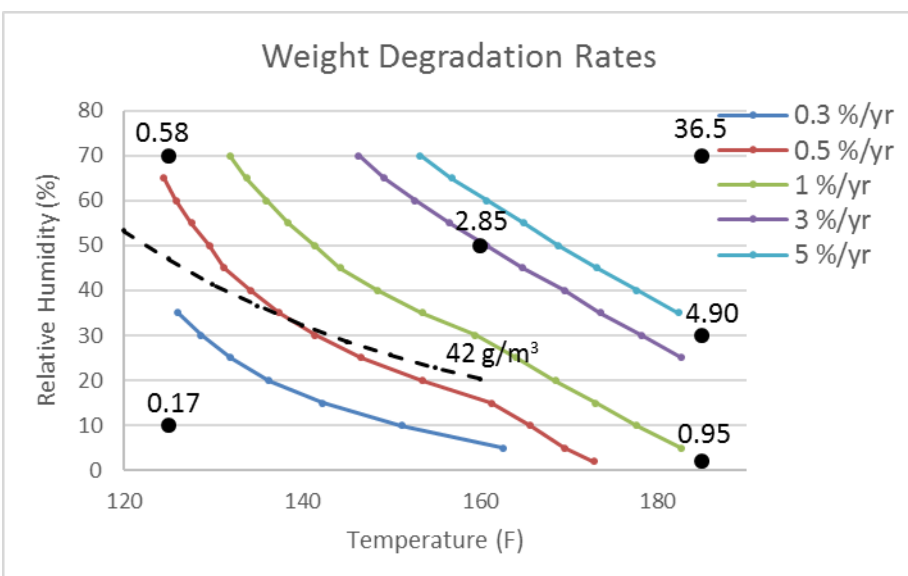


Figure 11. Fiberboard weight loss model. Lines represent contours of equal rate of weight loss. Numerical values are the average degradation rates of aged samples. The dashed line indicates those environments for which the absolute humidity is 42 g/m^3 – the fiberboard is expected to remain below this condition for conforming packages in storage.

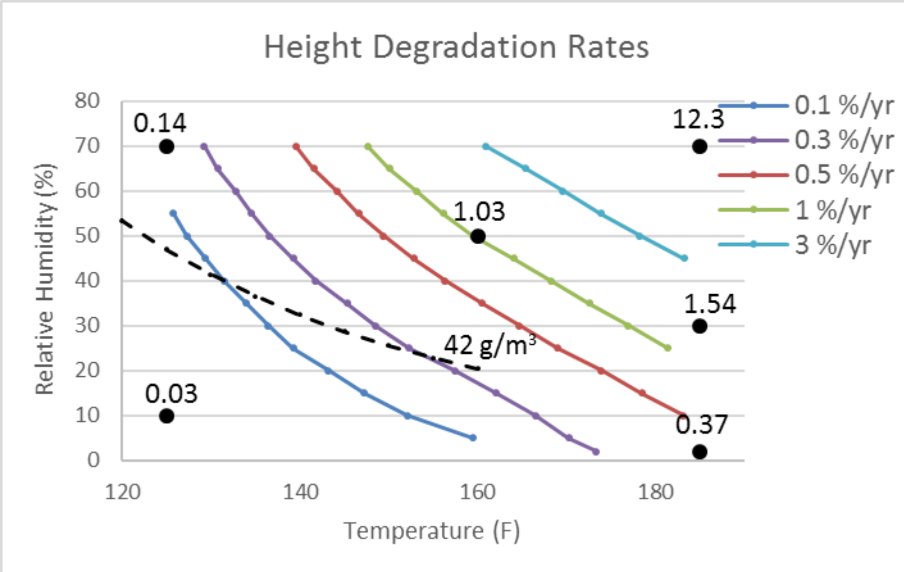


Figure 12. Fiberboard height loss model. Lines represent contours of equal rate of height loss. Numerical values are the average degradation rates of aged samples. The dashed line indicates those environments for which the absolute humidity is 42 g/m^3 – the fiberboard is expected to remain below this condition for conforming packages in storage.

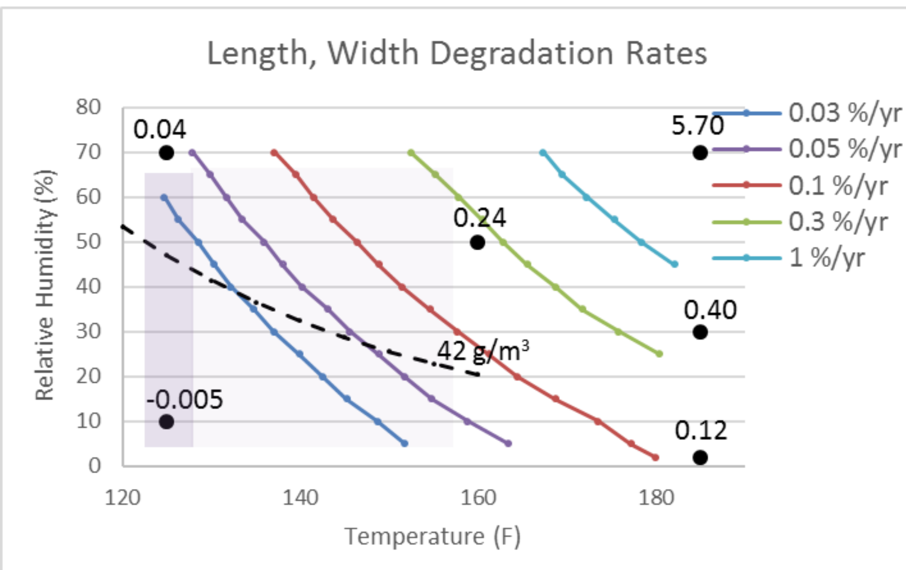


Figure 13. Fiberboard length / width loss model. Lines represent contours of equal rate of length / width loss. Numerical values are the average degradation rates of aged samples. The dashed line indicates those environments for which the absolute humidity is 42 g/m^3 – the fiberboard is expected to remain below this condition for conforming packages in storage. Predicted degradation rates within the shaded area are conservatively affected by the treatment of the negative degradation rate for 125 °F oven samples.

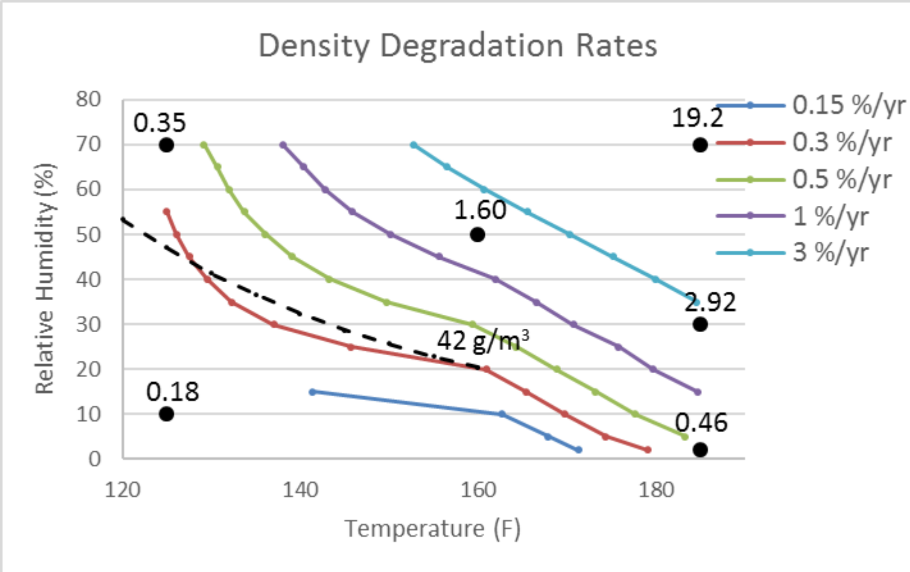


Figure 14. Fiberboard density loss model. Lines represent contours of equal rate of density decrease. Numerical values are the average degradation rates of aged samples. The dashed line indicates those environments for which the absolute humidity is 42 g/m^3 – the fiberboard is expected to remain below this condition for conforming packages in storage.

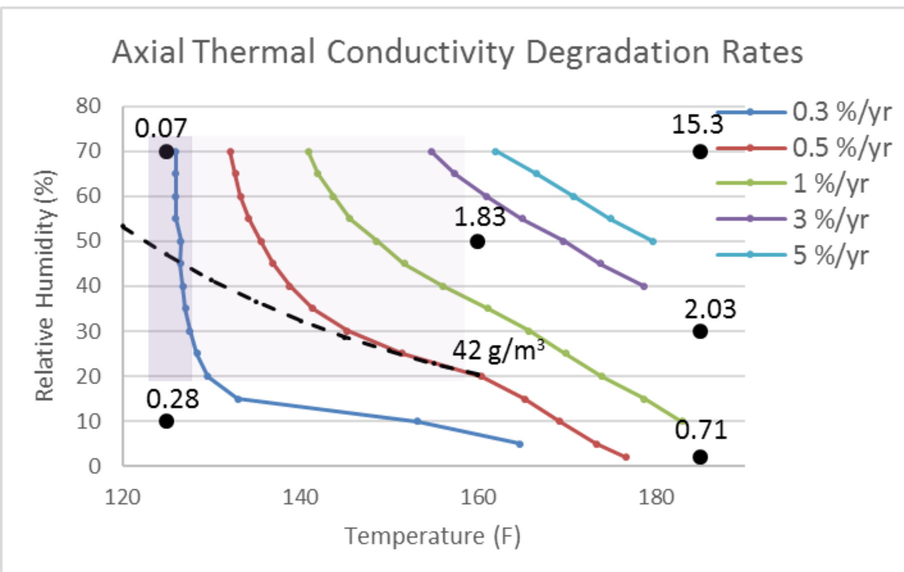


Figure 15. Fiberboard thermal conductivity, axial orientation model. Lines represent contours of equal rate of thermal conductivity decrease in the axial orientation. Numerical values are the average degradation rates of aged samples. The dashed line indicates those environments for which the absolute humidity is 42 g/m^3 – the fiberboard is expected to remain below this condition for conforming packages in storage. Predicted degradation rates within the shaded area are conservatively affected by the treatment of the 125 °F oven and 125 °F 70%RH data.

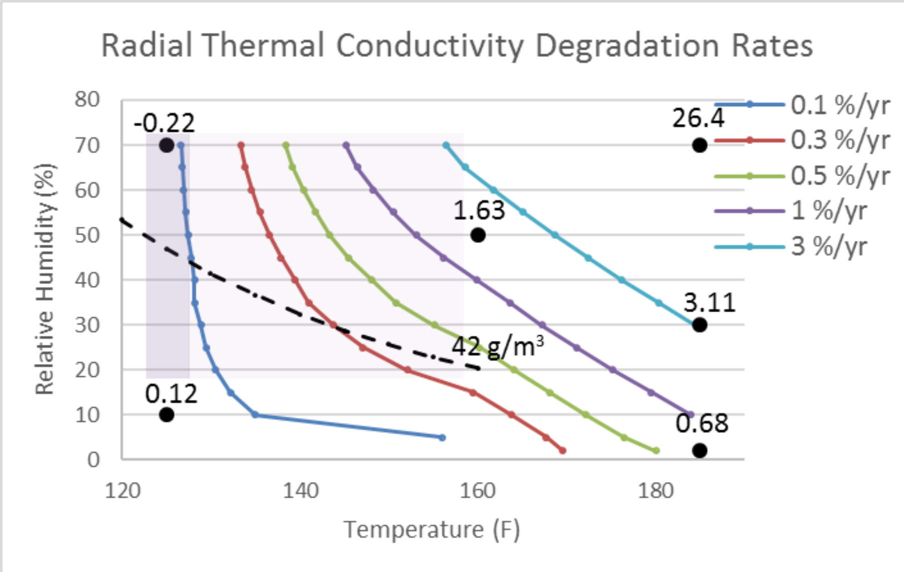


Figure 16. Fiberboard thermal conductivity, radial orientation model. Lines represent contours of equal rate of thermal conductivity decrease in the radial orientation. Numerical values are the average degradation rates of aged samples. The dashed line indicates those environments for which the absolute humidity is 42 g/m^3 – the fiberboard is expected to remain below this condition for conforming packages in storage. Predicted degradation rates within the shaded area are conservatively affected by the treatment of the 125 °F oven and 125 °F 70%RH data.

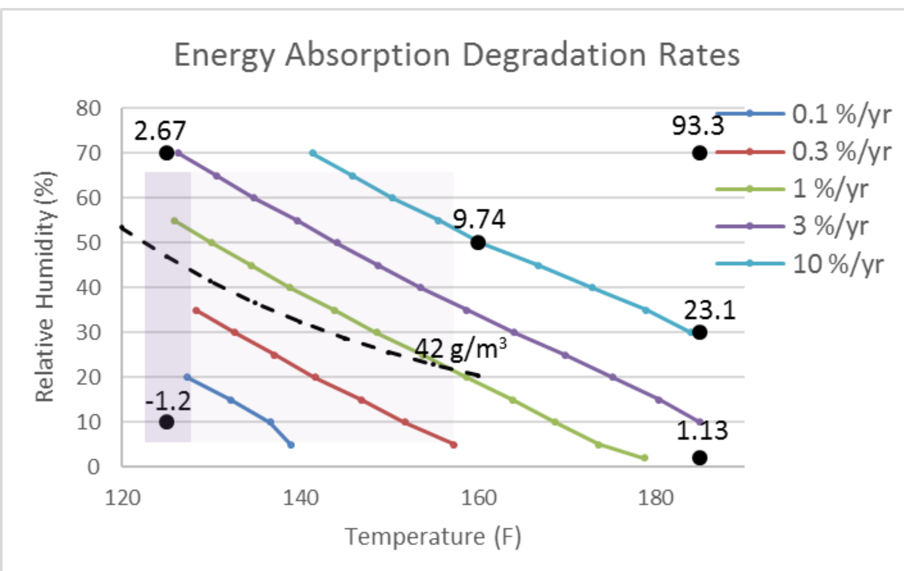
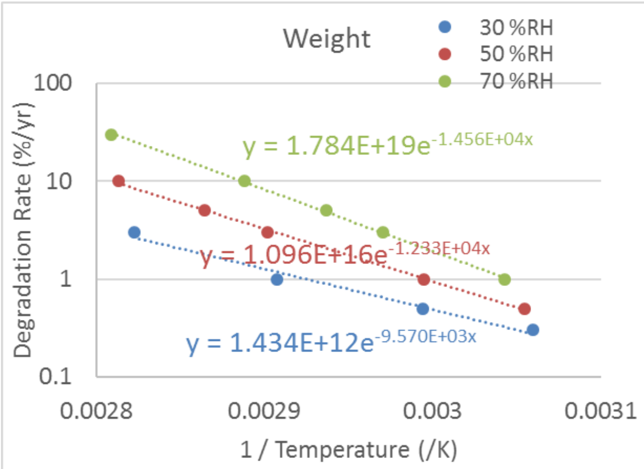
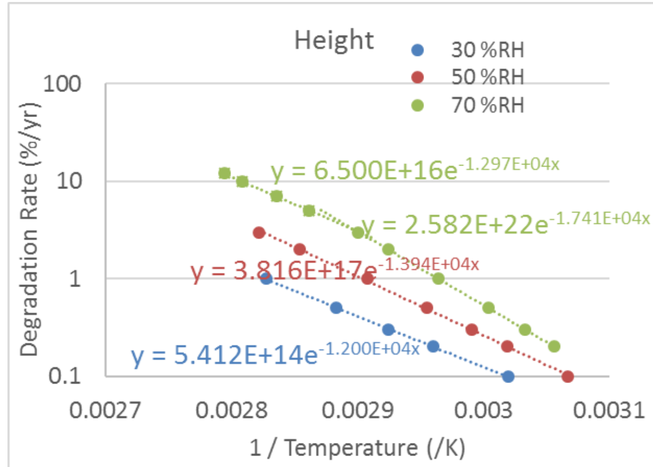


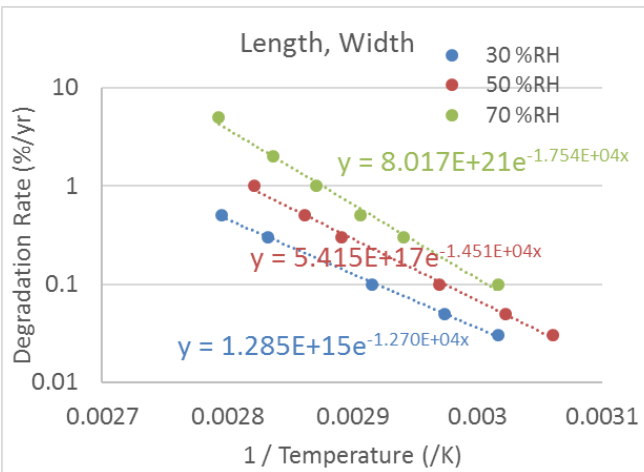
Figure 17. Fiberboard energy absorption model. Lines represent contours of equal rate of energy absorption loss for compression samples tested in the perpendicular orientation. Numerical values are the average degradation rates of aged samples. The dashed line indicates those environments for which the absolute humidity is 42 g/m^3 – the fiberboard is expected to remain below this condition for conforming packages in storage. Predicted degradation rates within the shaded area are conservatively affected by the treatment of the negative degradation rate for 125°F oven samples.



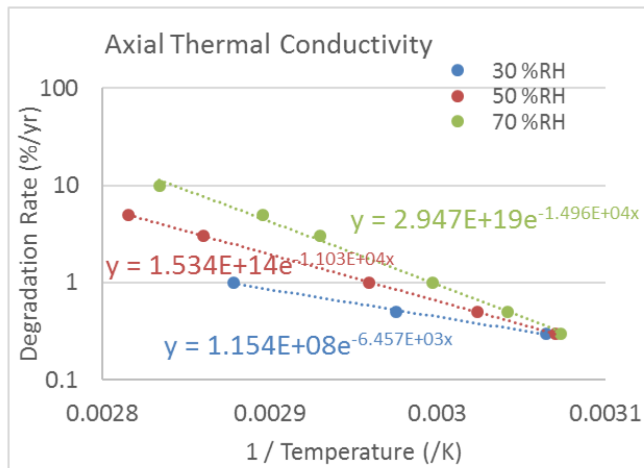
(a) Weight



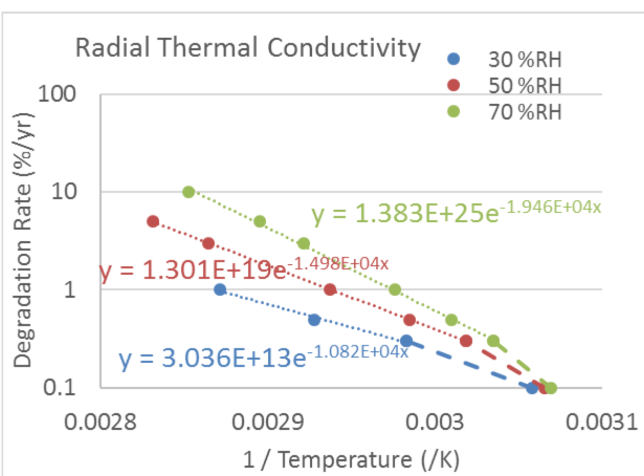
(b) Height



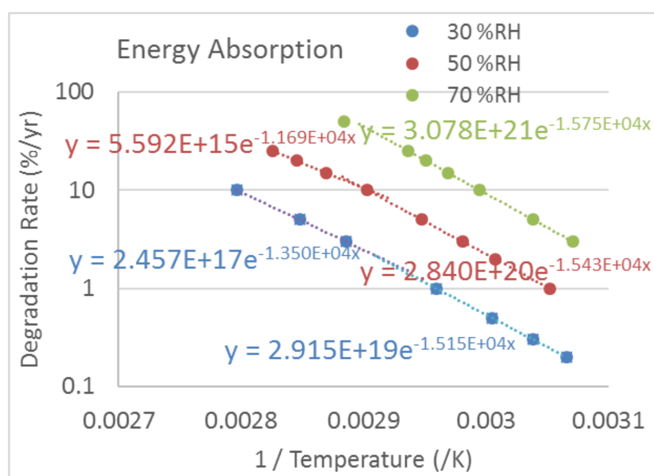
(c) Length, width



(d) Axial thermal conductivity



(e) Radial thermal conductivity



(f) Energy absorption

Figure 18. Arrhenius plots of degradation rates for the identified properties at values of elevated relative humidity.

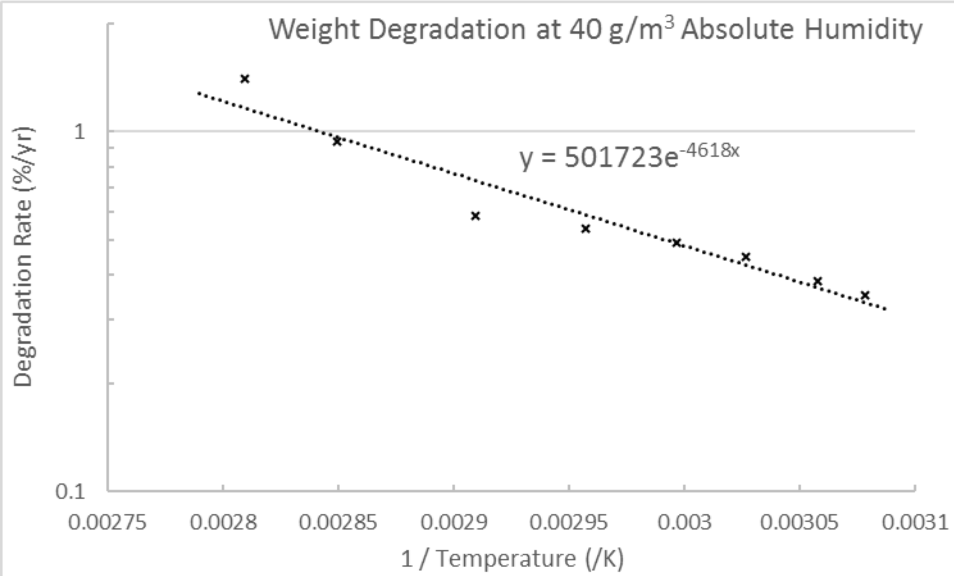
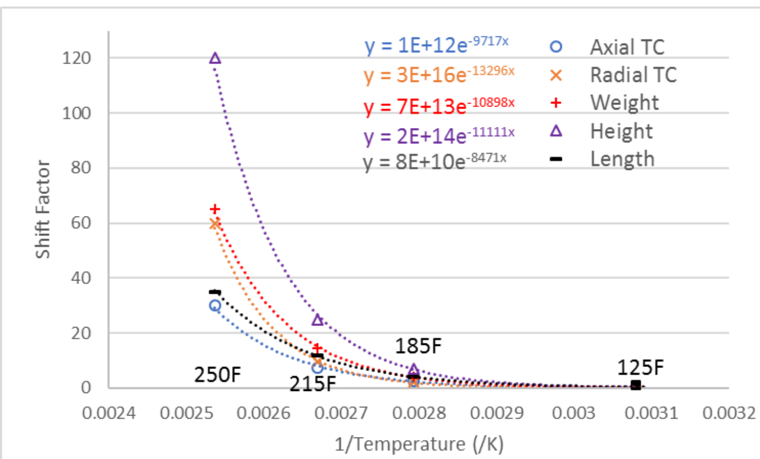


Figure 19. Arrhenius plot of weight degradation at 40 g/m^3 absolute humidity. Based on the exponential coefficient, the activation energy is 38400 J/mol.



	E_a (J/mol) from shift factors	E_a (J/mol) from degradation models
Weight	90600	91200
Height	92400	90800
Length	70400	77400
Axial TC	80800	71900
Radial TC	110600	86000

Figure 20. Shift factor correlations used to estimate the activation energy (E_a). This activation energy estimate is compared to values obtained from the degradation models.

Appendix
Data Correlations and Curve Fits Used To Develop the Aging Model for Each Fiberboard Property

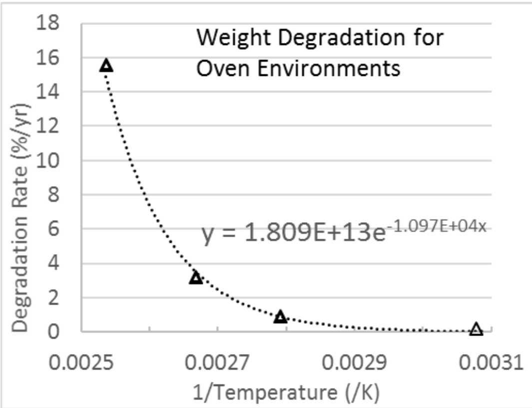
Average degradation rates (%/year) for physical properties. The rates for each aging environment have been adjusted by the control sample rates. Where a sample displayed a change in degradation rate over time, only the initial rate is reflected in this table.

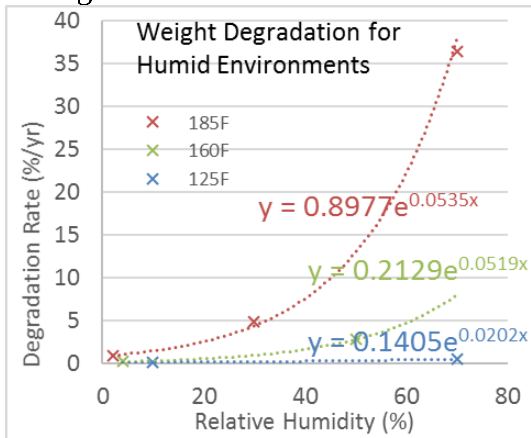
	Weight	Density	Height	Length, Width
Control samples	-0.083	0.097	-0.055	-0.061
125 °F oven	-0.17	-0.18	-0.031	0.005
125 °F 70%RH	-0.58	-0.35	-0.13	-0.040
160 °F 50%RH	-2.85	-1.60	-1.03	-0.24
185 °F oven	-0.95	-0.46	-0.37	-0.12
185 °F 30%RH	-4.90	-2.92	-1.54	-0.40
185 °F 70%RH	-36.50	-19.20	-12.27	-5.70
215 °F oven	-3.19	-1.36	-1.25	-0.40
250 °F oven	-15.57	-8.77	-5.99	-1.32

Average degradation rates (%/year) for thermal conductivity and energy absorption. Where a sample displayed a change in degradation rate over time, only the initial rate is reflected in this table.

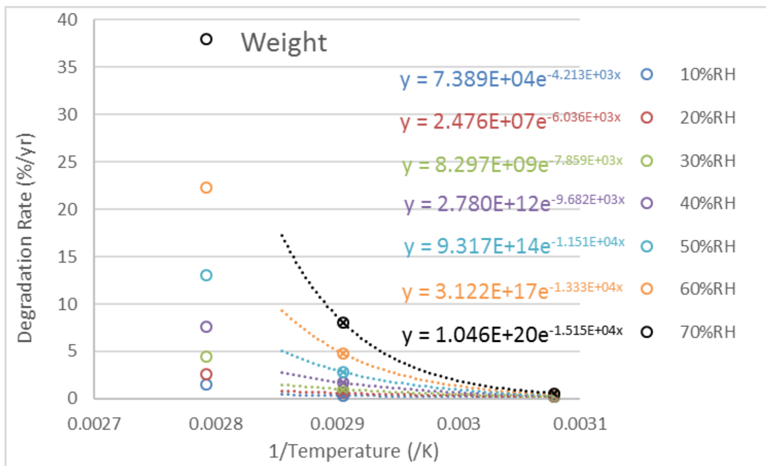
	Thermal Conductivity		Energy Absorption
	Radial orientation	Axial orientation	Perpendicular orientation
125 °F oven	-0.12	-0.28	1.20
125 °F 70%RH	0.22	-0.073	-2.67
160 °F 50%RH	-1.63	-1.83	-9.74
185 °F oven	-0.68	-0.72	-1.13
185 °F 30%RH	-3.11	-2.96	-23.1
185 °F 70%RH	-26.42	-15.25	-93.3
215 °F oven	-2.17	-2.16	-9.34
250 °F oven	-11.24	-7.97	-26.8

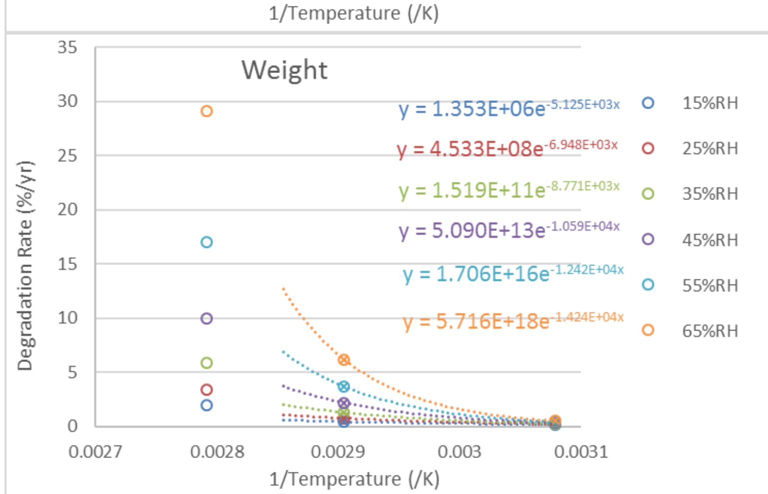
Data correlations and curve fits for Weight Change:



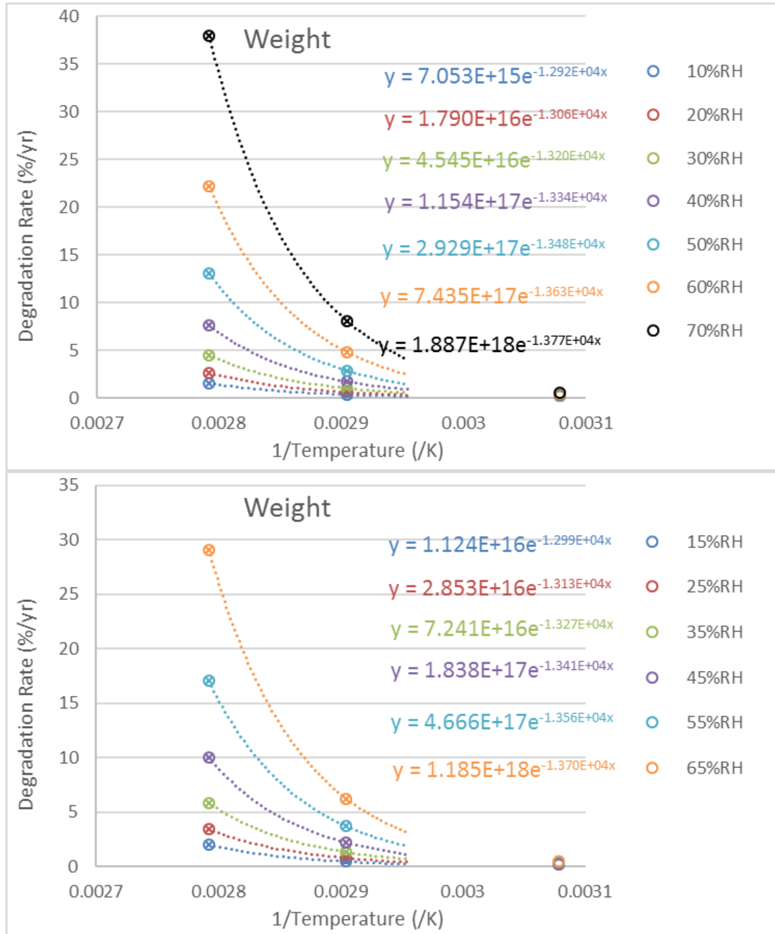


Degradation rate vs reciprocal temperature correlations for specified relative humidity levels: Correlations for 125 – 160 °F.

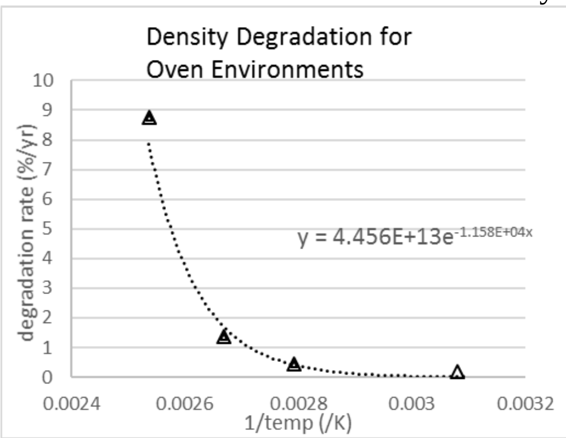


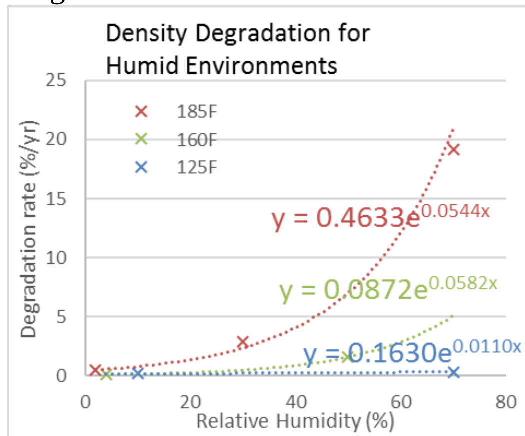


Degradation rate vs reciprocal temperature correlations for specified relative humidity levels: Correlations for 160 – 185 °F.

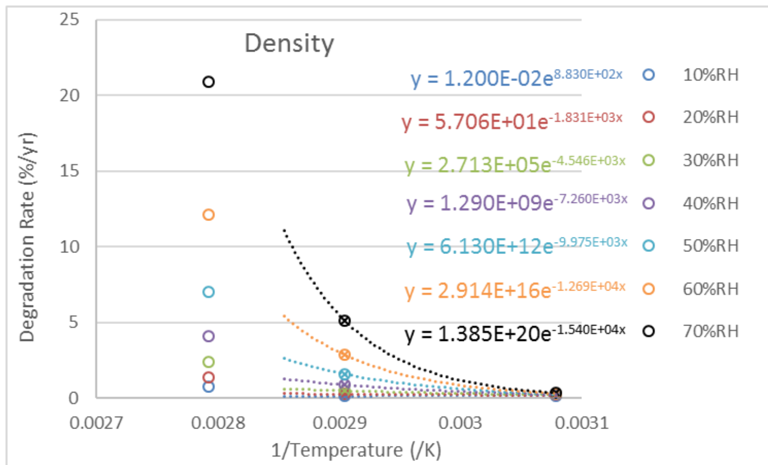


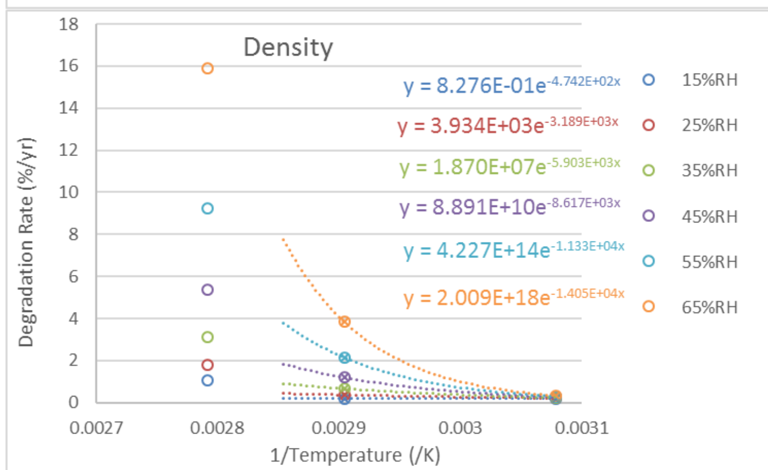
Data correlations and curve fits for Density Change:



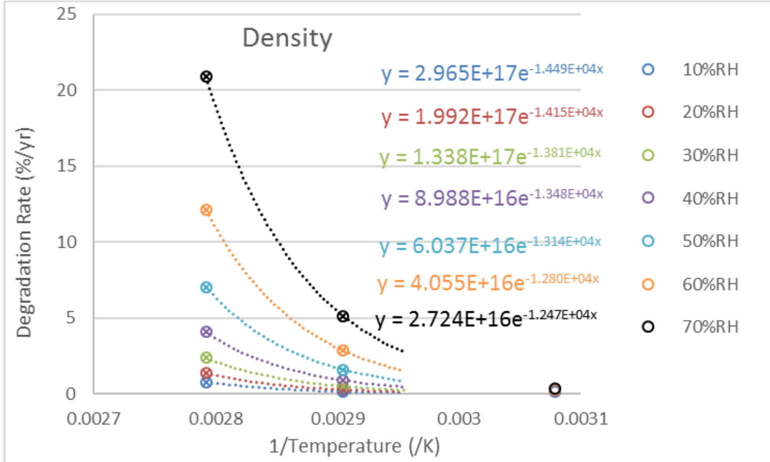


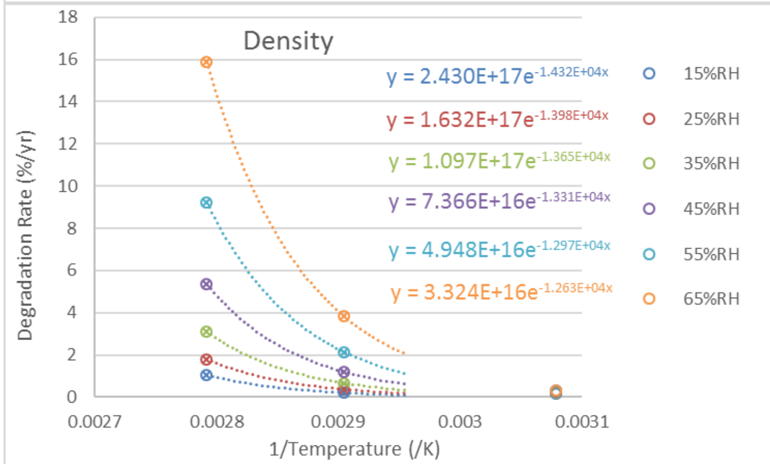
Degradation rate vs reciprocal temperature correlations for specified relative humidity levels: Correlations for 125 – 160 °F.



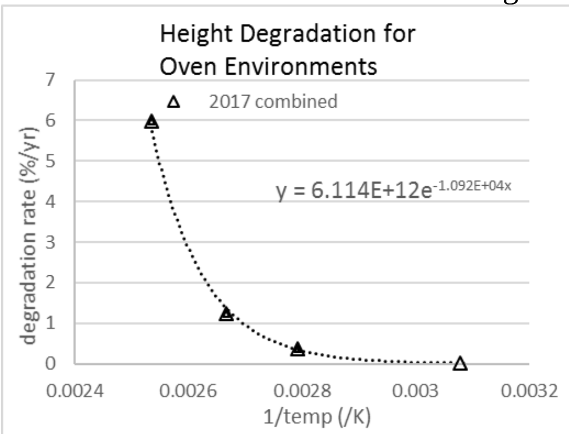


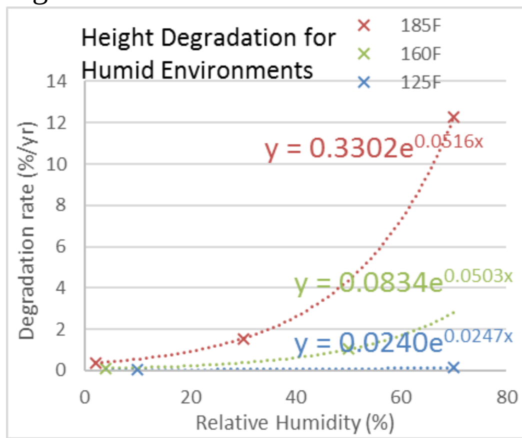
Degradation rate vs reciprocal temperature correlations for specified relative humidity levels: Correlations for 160 – 185 °F.



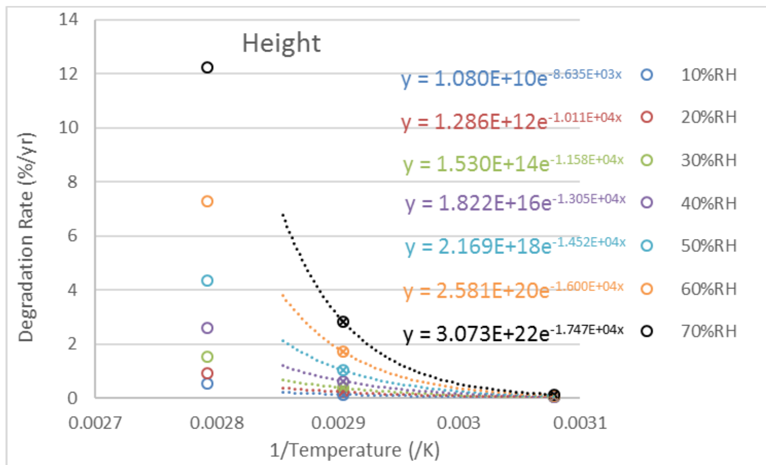


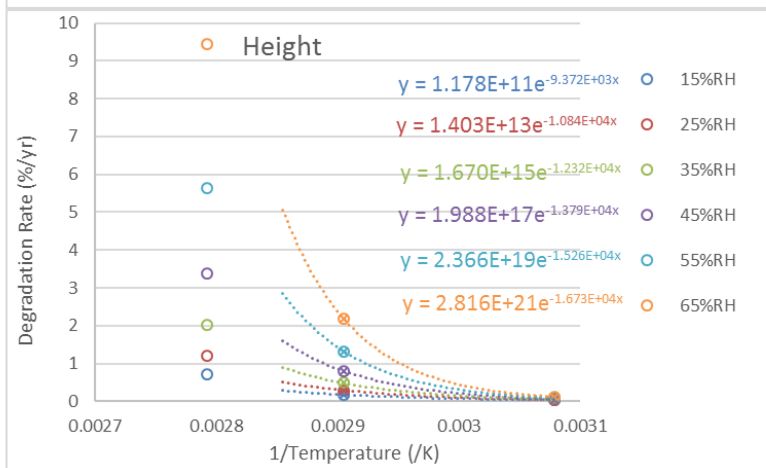
Data correlations and curve fits for Height Change:



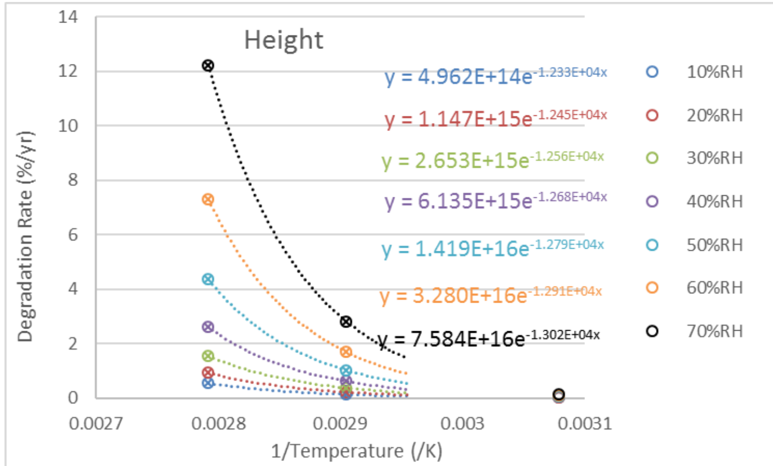


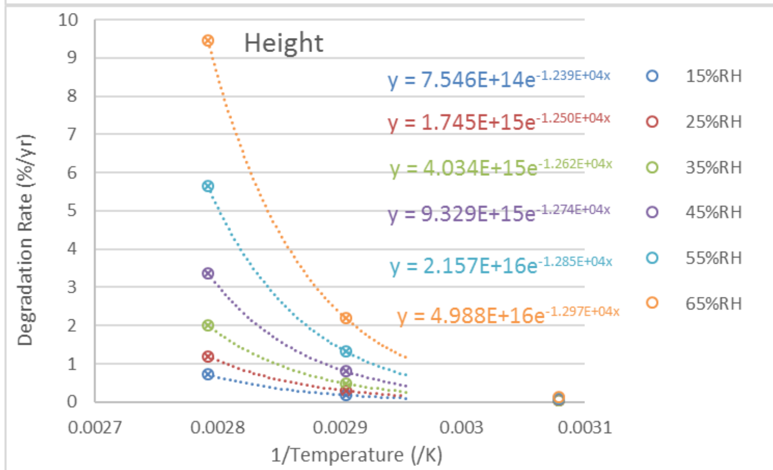
Degradation rate vs reciprocal temperature correlations for specified relative humidity levels: Correlations for 125 – 160 °F.



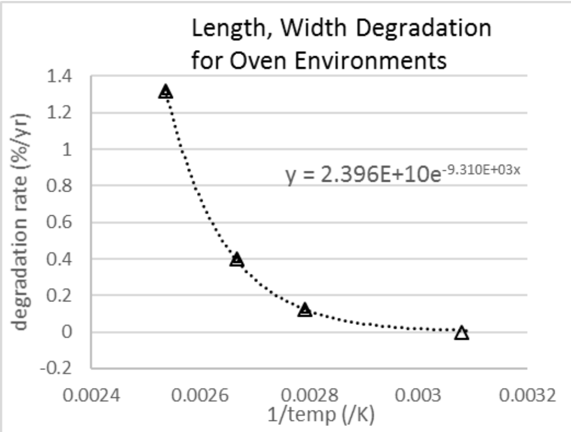


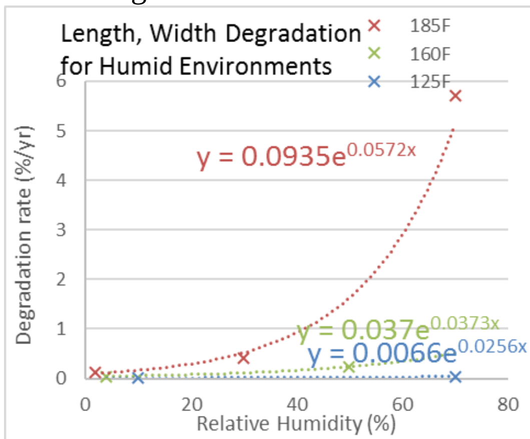
Degradation rate vs reciprocal temperature correlations for specified relative humidity levels: Correlations for 160 – 185 °F.



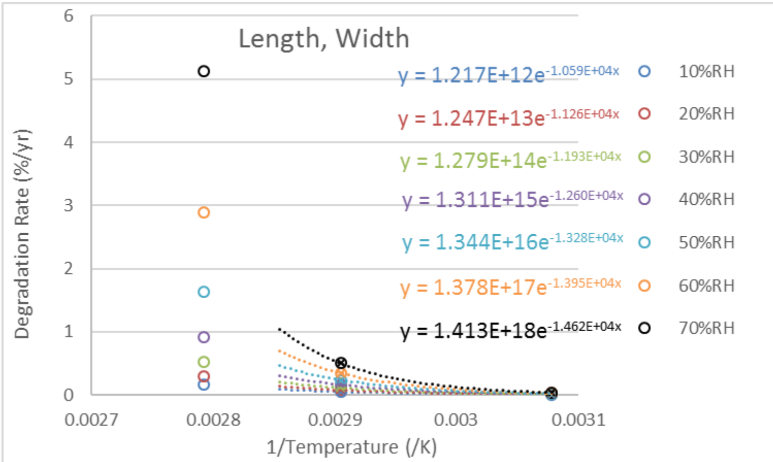


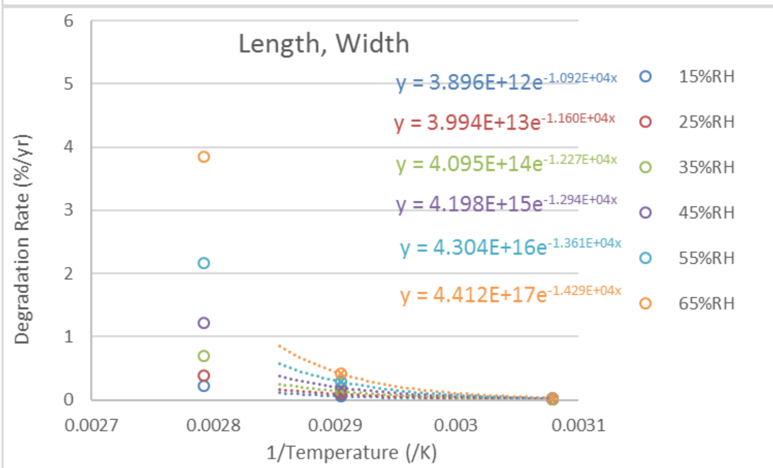
Data correlations and curve fits for Length, Width Change:



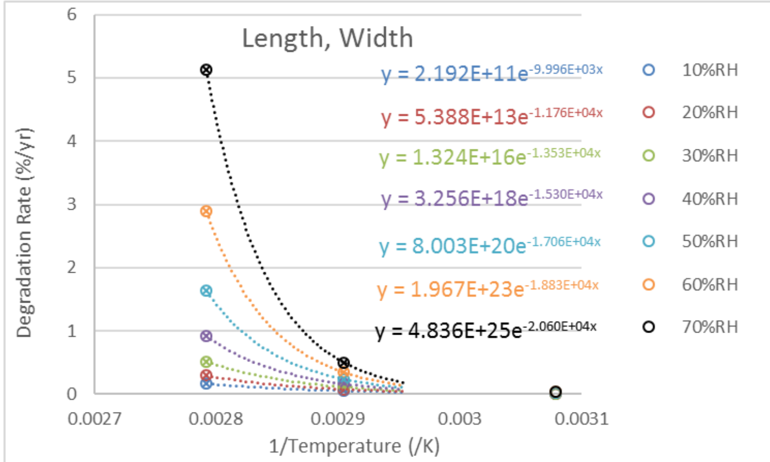


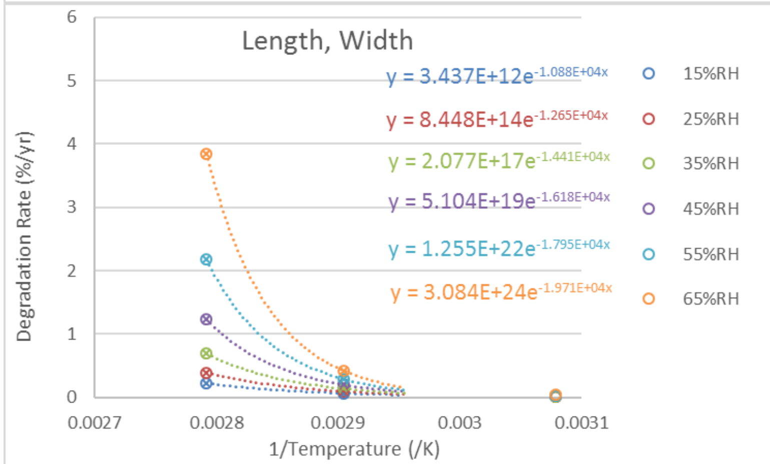
Degradation rate vs reciprocal temperature correlations for specified relative humidity levels: Correlations for 125 – 160 °F.



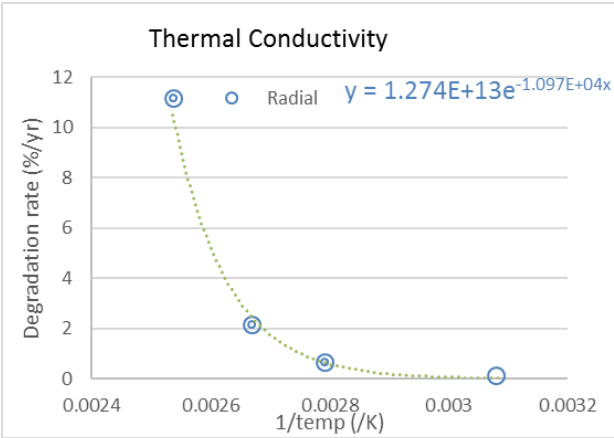


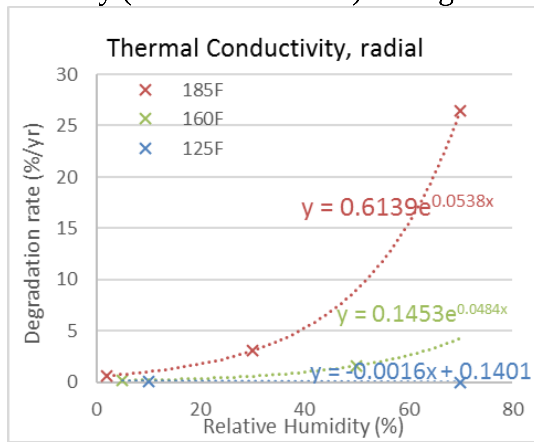
Degradation rate vs reciprocal temperature correlations for specified relative humidity levels: Correlations for 160 – 185 °F.



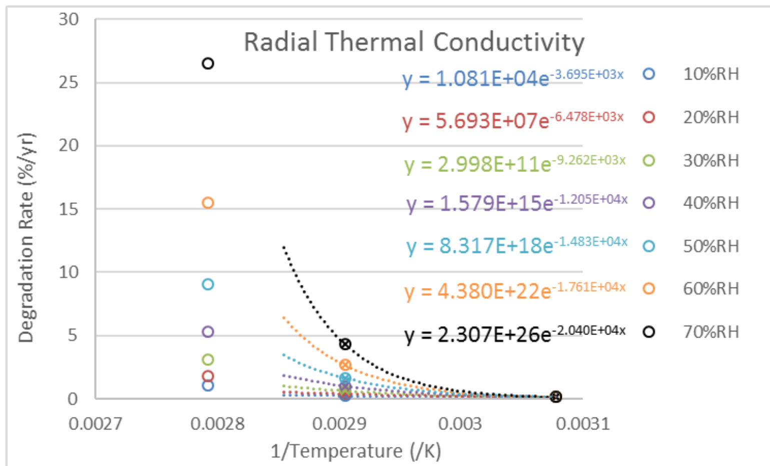


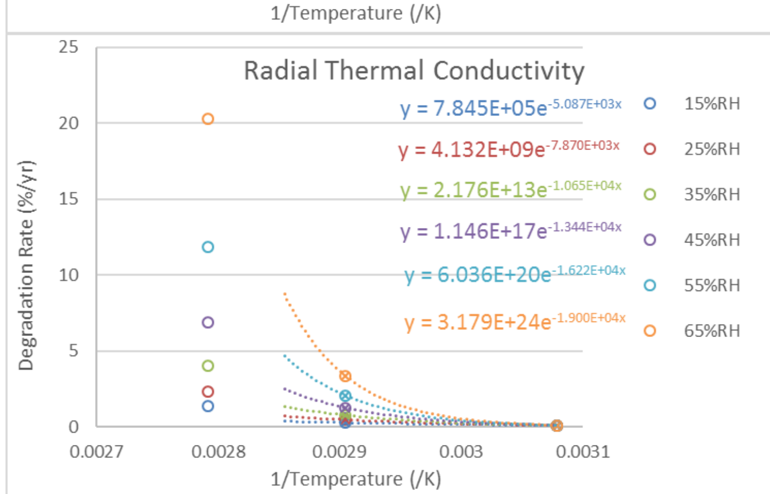
Data correlations and curve fits for Thermal Conductivity (radial orientation) Change:



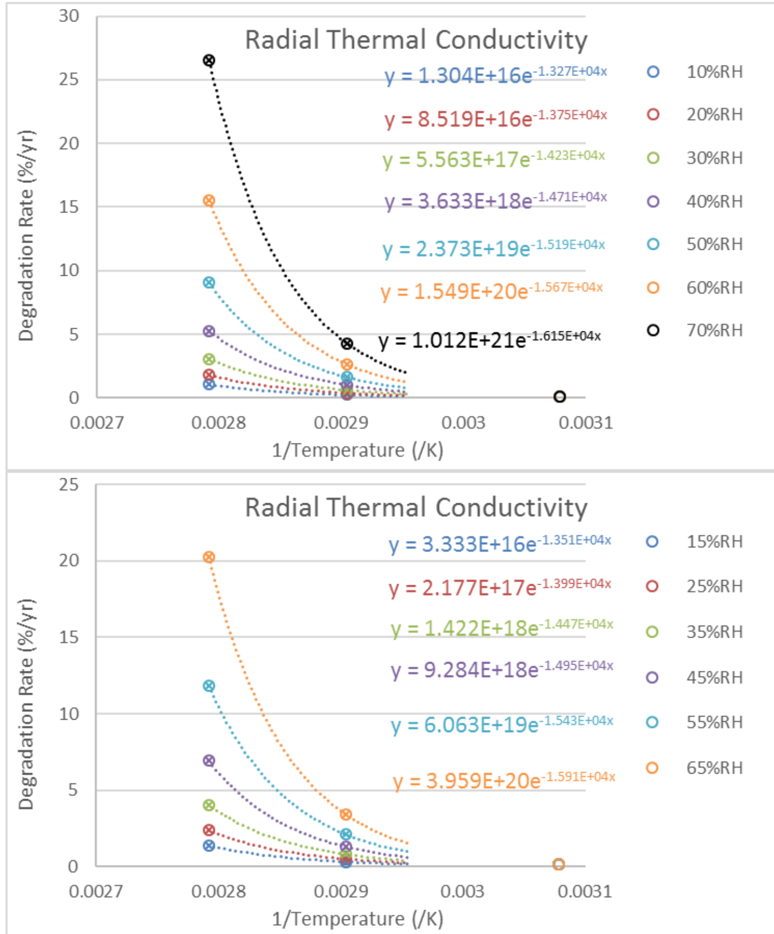


Degradation rate vs reciprocal temperature correlations for specified relative humidity levels: Correlations for 125 – 160 °F.

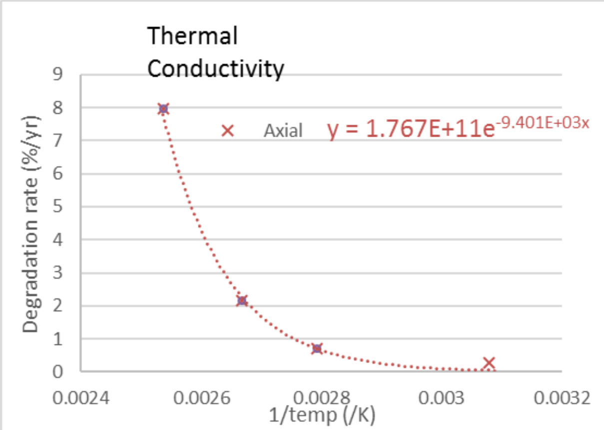


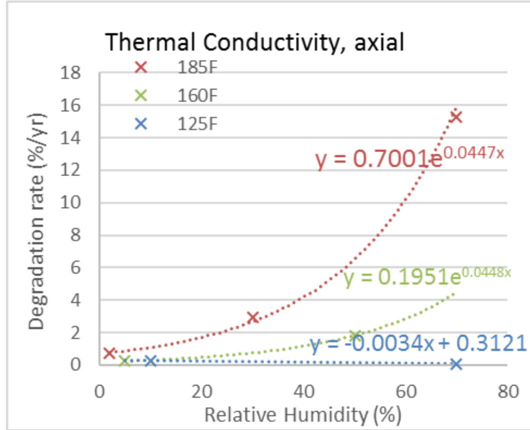


Degradation rate vs reciprocal temperature correlations for specified relative humidity levels: Correlations for 160 – 185 °F.

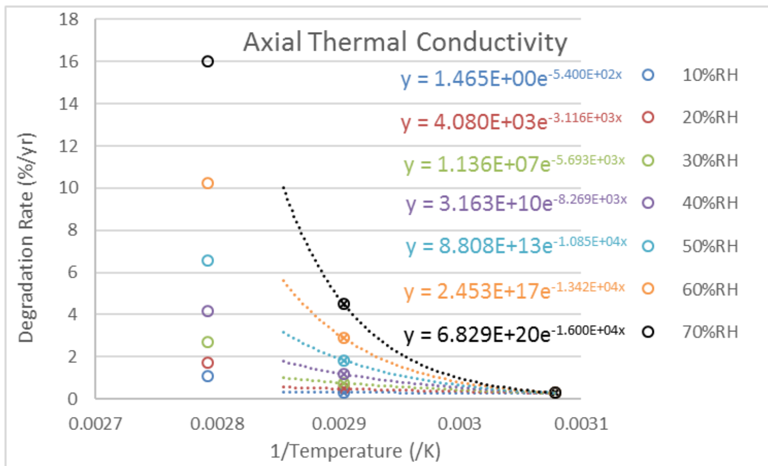


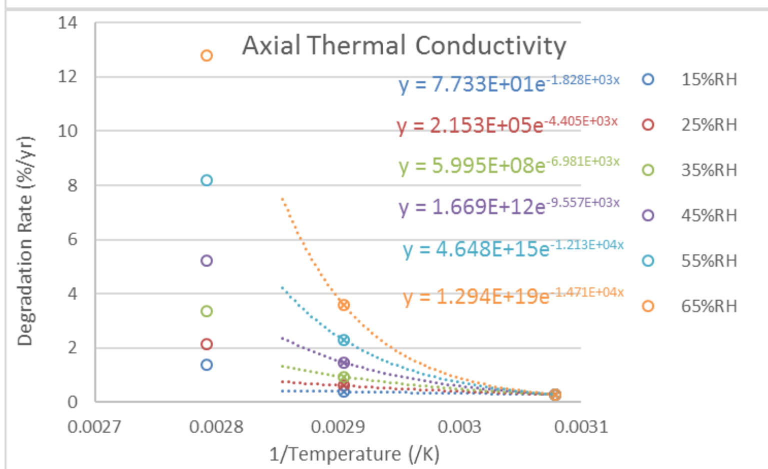
Data correlations and curve fits for Thermal Conductivity (axial orientation) Change:



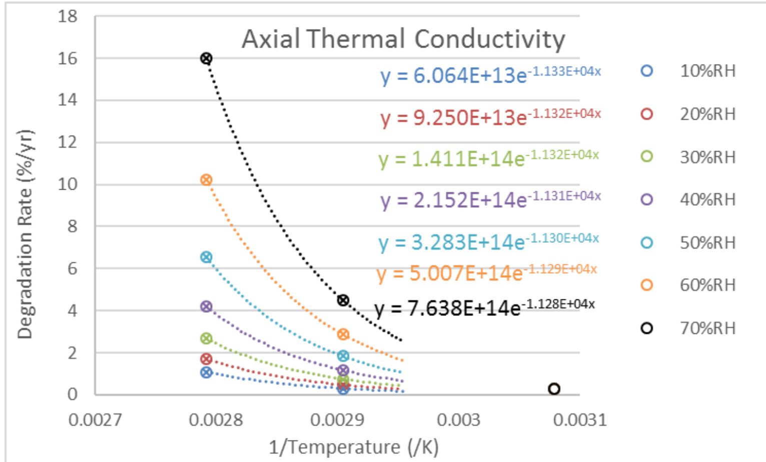


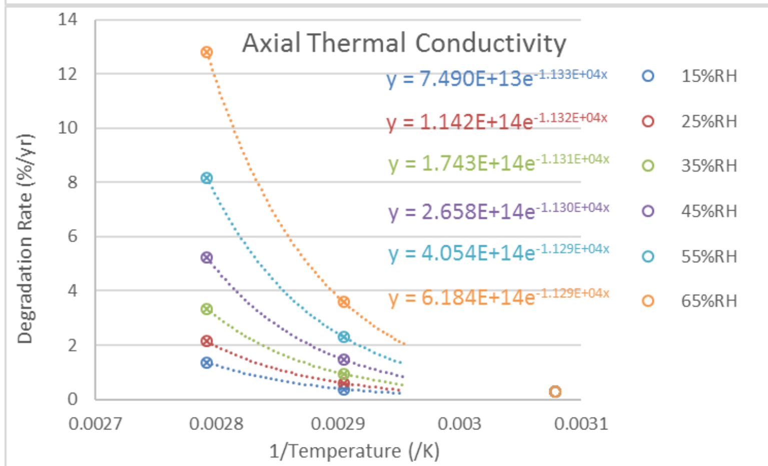
Degradation rate vs reciprocal temperature correlations for specified relative humidity levels: Correlations for 125 – 160 °F.



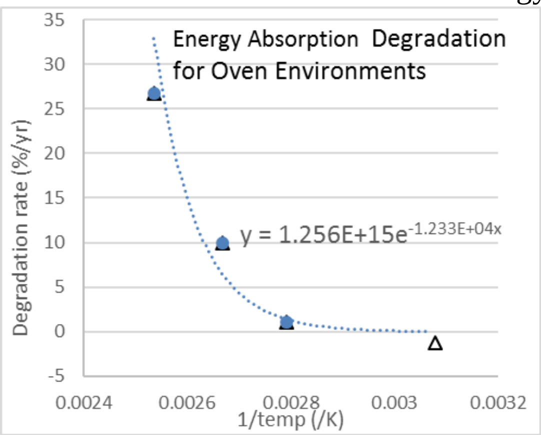


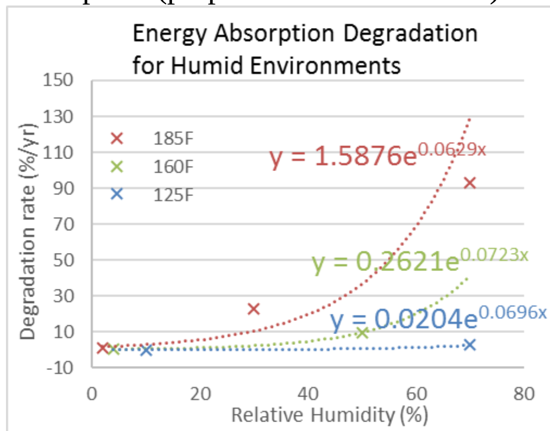
Degradation rate vs reciprocal temperature correlations for specified relative humidity levels: Correlations for 160 – 185 °F.



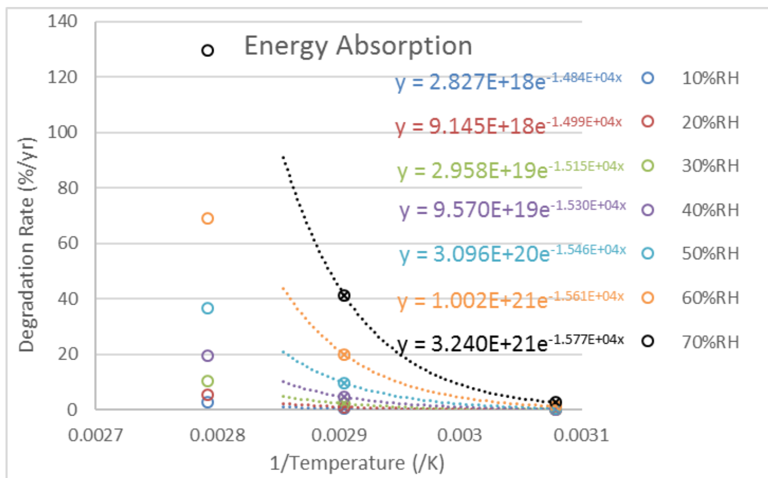


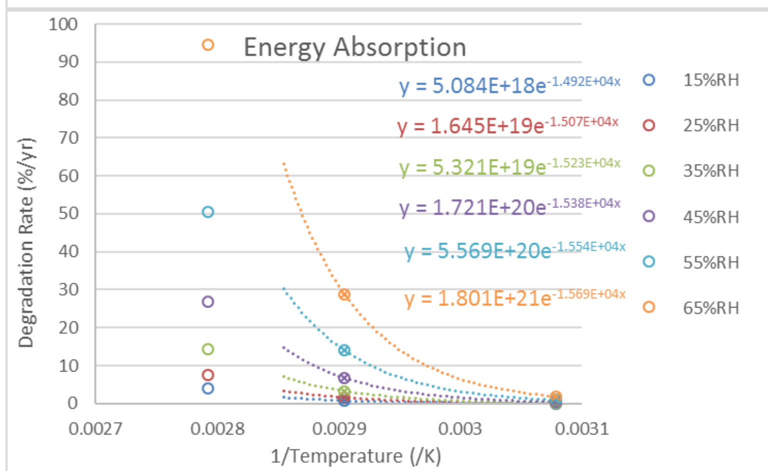
Data correlations and curve fits for Energy Absorption (perpendicular orientation) Change:



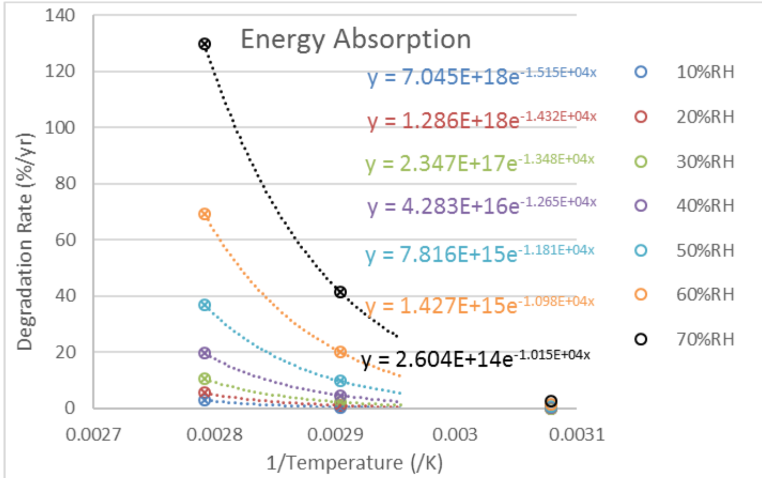


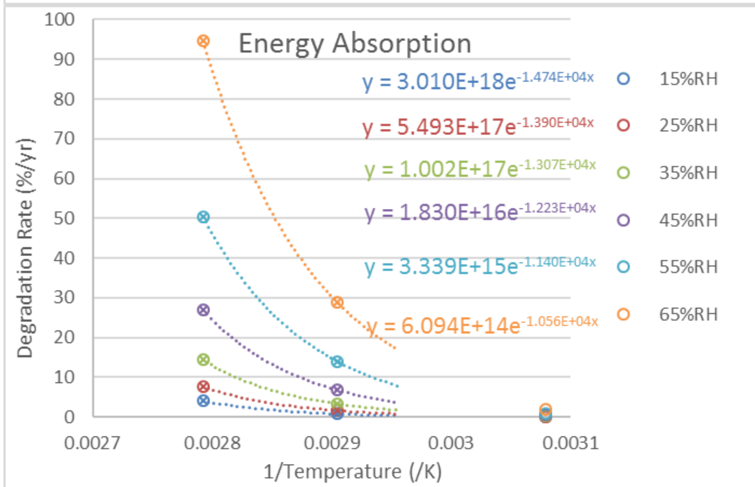
Degradation rate vs reciprocal temperature correlations for specified relative humidity levels: Correlations for 125 – 160 °F.





Degradation rate vs reciprocal temperature correlations for specified relative humidity levels: Correlations for 160 – 185 °F.





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