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MODEL 9975 LIFE EXTENSION PACKAGE 3 – INTERIM REPORT

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Model 9975 Life Extension Package 3 – Interim Report

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Summary

Life extension package LE3 (9975-03203) has been instrumented and subjected to an elevated temperature environment for 259 weeks. During this time, the maximum fiberboard temperature was ~160 - 165 °F, and was established by a combination of internal (19 watts) and external heat sources.

Several tests and parameters were used to characterize the package components. Results from these tests generally indicate agreement between this full-scale shipping package and small-scale laboratory tests on fiberboard samples. These areas of agreement include the rate of change of fiberboard weight and dimensions, and change in fiberboard thermal conductivity. Dimensional measurements of the shield indicate no consistent trend to suggest creep deformation has occurred. This is consistent with literature data that predicts a very small creep deformation for the time at temperature experienced by this package.

This package provides an example of the extent to which moisture within a typical fiberboard assembly can redistribute in the presence of a temperature gradient such as might be created by a 19 watt internal heat load. The majority of water within the fiberboard migrated to the bottom layers of fiberboard, some of which eventually escaped from the package.

Two conditions have developed that are not consistent with package certification requirements. The axial gap at the top of the package has increased to 1.48 inches, exceeding the 1 inch criterion. In addition, staining and/or corrosion have formed in a few spots on the drum. However, the package remains capable of performing its function.

Background

This report summarizes information on a 9975 package tested per Task Technical Plan WSRC-TR-2005-00014 [1], which is part of the comprehensive 9975 package surveillance program [2]. This task provides an integrated assessment of the package response to environmental extremes, and demonstrates the extent to which data from small laboratory samples scale up to a full package. The primary goal of this task is to validate aging models based on lab scale testing of the fiberboard overpack and containment vessel O-rings. A secondary goal is to examine the behavior of the lead shielding under bounding conditions.

Three 9975 packages were modified to provide instrumentation for monitoring package performance and response to environmental aging. Each package has a different environmental exposure history. The first two packages have been previously removed from test [3, 4]. The third package (LE3, or 9975-03203) has been in test for 259 weeks (as of October 16, 2013) and remains in test. This report summarizes and analyzes the test data available to date for test package LE3.

The primary focus of LE3 was to age the fiberboard at a temperature bounding to KAMS storage conditions, but not excessively so. Ambient temperatures up to 104°F have been recorded in KAMS, although that level was not sustained for an extended period. Average ambient temperatures are much lower [5].

Experimental Method

This life extension package is a 9975 shipping package that was modified to add instrumentation and an internal heat source. An access port for fiberboard sample removal was added to the drum side wall. Thermocouples provide the temperature at a number of locations throughout the package, including the 3013 payload, PCV, SCV, multiple locations within the fiberboard, and drum surface. A sketch showing thermocouple locations is provided in Figure 1.

Package LE3 was placed within an environment with external temperature control, but no humidity control. A cartridge heater inside a modified 3013 container provides 19 watts internal heat. The external temperature was established by enclosing the package in a modified 55 gallon drum, and placing a drum heater around this larger drum (Figure 2). The external blanket heater was adjusted to provide a maximum fiberboard temperature of ~160°F. This was typically accomplished at a setting of ~100 – 105°F, which produced a drum surface temperature of ~120 – 130°F. This condition approximates the maximum ambient temperature observed in KAMS, and all packages in KAMS contain less than 19 watts. Therefore, this test provides internal temperatures that are conservative to actual conditions in storage.

The PCV and SCV were modified to allow placement of a cartridge heater through the bottom of the containment vessels and into a well in the 3013. The 3013 was welded shut with a surrogate load of steel shot. The cartridge heater conductors and thermocouples attached to the 3013, PCV and SCV exit the package opposite the side where the fiberboard is instrumented, to minimize disruption of the measured fiberboard thermal profile.

The modifications to the PCV and SCV provided open penetrations in the bottom of each vessel. Because of this, both O-rings in each vessel can receive a sensitive helium leak test. Normally, only the outer O-ring is leak-tested with a helium detector, which provides assurance of a leak-tight seal of 1×10^{-7} std cc air/sec (or 2×10^{-7} std cc He/sec). After loading the package, the leak-tightness of both O-rings is confirmed at a level of $\sim 1 \times 10^{-3}$ std cc air/sec with a less sensitive rate of pressure rise technique. With the modified vessels of LE3, the more sensitive helium leak test can be performed at any time.

Thermocouple data from the package is automatically recorded at preset intervals. Additional data is collected on an occasional basis during periodic examinations. This includes:

- Weight of the entire package
- Weight and moisture content of the removable fiberboard sections
- Visual observations of the package exterior
- Weight and dimensions of the fiberboard assemblies and shield
- Visual observations of other package components

The last 2 of the above data sources require opening the drum lid and removing internal components. These steps are not performed with every examination, so these data are not collected as often as the other items listed.

Results – Periodic Data

There are several metrics which provided evidence of change in the package over time. These include package weight, fiberboard moisture levels, and the temperature profile across the fiberboard. Additional indications of change might be seen in the weight and appearance of the fiberboard, dimensional variation of the lead shield, and from visual observations of the components.

The following observations were made of specific changes within the package:

- After 2 weeks in test, liquid condensation was observed on the underside of the drum lid and on the air shield (Figure 3). The moisture level of the bottom fiberboard layers is above saturation ($>38\%$ WME).
- After 5 weeks in test, mold was observed on the lower layers of the bottom removable fiberboard section. The mold changed color and texture through 45 weeks in test, and gradually became dormant by week 151. Little change has been observed after that. (Figure 4)
- After 7 weeks in test, increased corrosion of the shield was observed, along with some nodular corrosion deposits. Shield corrosion approached its heaviest visual appearance by 26 weeks, and the upper portion of the shield darkened slightly after that (Figure 5).
- After 109 weeks in test, corrosion was observed on the drum interior at the bottom crevice (Figure 6).
- After 119 weeks in test, possible corrosion was observed on one stitch weld along the bottom edge of the drum exterior (Figure 7). After 151 weeks, additional corrosion was observed on the same stitch weld and on additional stitch welds.
- After 190 weeks in test, staining and/or corrosion was observed on the drum interior at the top flange stitch welds (Figure 8).

A plot of package weight over time is shown in Figure 9. The weight of the upper and lower fiberboard assemblies was also tracked over time, although it was recorded less frequently. These data are shown in Figure 10, along with fiberboard density values calculated from the weight and dimensions. Two small sections of fiberboard were cut from the bottom of the lower assembly, and are accessible to remove through a hatch on the drum side. These sections are characterized more often than the drum is opened to inspect the upper or lower fiberboard assembly. Weight data for these removable sections are shown in Figure 11.

Fiberboard dimensions change over time. Table 1 summarizes some of the lower fiberboard assembly dimensions and their relative change. In general, the following changes are observed:

- Fiberboard height decreases over time
- Fiberboard thickness (in the dryer regions) decreases over time (except for 1 anomalous point)
- Fiberboard diameter at the bottom increases as that region gains moisture, then decreases as the package overall loses moisture.

Moisture was originally distributed uniformly throughout the fiberboard assembly. Upon heating, it quickly re-distributed towards the OD surface and bottom. Average moisture levels of several fiberboard regions are summarized in Table 2, along with moisture content values for the small removable fiberboard sections.

Given the relatively low melting temperature of lead (621°F), the shield may experience creep at service temperatures. Measurements of the LE3 shield were collected to investigate this possibility, and are summarized in Table 3.

The corrosion of the lead shield is also of interest. Significant corrosion product was observed after 7 weeks in test, and was white and nodular (Figure 5). The corrosion product had become somewhat heavier after 26 weeks in test, but did not appear to increase significantly after that. Instead, the appearance began to change slightly in that the upper portion of the shield (adjacent to the upper fiberboard assembly) yellowed slightly (observed after 109 weeks in test) and retained that appearance during subsequent testing. The corrosion typically seen on a 9975 shield is smooth and white. Once the corrosion product on a typical shield is sufficiently thick (a few thousands of an inch), it will begin to loosen and flake off.

Discussion

Significant moisture redistribution was occurring as the package was initially brought to temperature. The data in Figure 2 indicate that moisture in the bottom fiberboard layer had increased past saturation (~38 %WME) before the first inspection after 2 weeks (1.4 weeks at temperature). The inner (warmest) fiberboard regions would have begun losing moisture immediately, while moisture levels in the outer regions did not change significantly until ~35 weeks at temperature. Moisture redistribution likely occurred through evaporation of water in the hotter regions, and condensation on the relatively cool drum and lid surfaces. This is seen in Figure 3, where condensation appears to have formed on the lid and rained down onto the air shield. As significant water accumulates on the air shield, it is likely that some of it might spill over the side. At that point, it could either transfer to the drum wall or re-absorb into the fiberboard, depending on the gap between the fiberboard and drum wall. Additional condensation would form directly on the drum sides and run down to the bottom. These mechanisms would likely remain active until the majority of water in the fiberboard re-distributes toward the bottom of the package.

The annular fiberboard region between the two bearing plates weighs approximately 23 kg. For an initial moisture content of ~11 %WME to decrease to ~6 %WME (from ~10 wt% to ~6.7 wt%), this annular region alone would lose ~0.7 kg water. With movement of this amount of water, the amount of condensation on the lid and air shield shown in Figure 3 is not surprising. It is noted that significant staining from condensation on the lid and air shield was also observed on the second life extension package [3], however, that package was not opened for inspection until after 19 weeks, at which time the moisture was no longer present.

Migration of water to the cooler regions of the package would leave the bulk of the fiberboard much drier than it was initially. This in turn would decrease the thermal conductivity for the bulk of the fiberboard, and increase the temperature gradient across the fiberboard.

Twelve thermocouples monitor the temperature gradient within the fiberboard. The hottest measured temperature within the fiberboard is at the highest thermocouple elevation on the ID surface, and the coolest measured temperature within the fiberboard is at the lowest thermocouple

elevation on the OD surface. The temperature vs time data at the highest fiberboard ID and lowest fiberboard OD locations are summarized in Figure 12. It is possible that the fiberboard reached slightly higher temperatures at elevations above the uppermost ID thermocouple. However, the variation among the thermocouples along the ID surface in the central region adjacent to the shield shows a very small gradient (typically < 1°F). Therefore, the recorded temperatures are assumed to provide a reasonable approximation of the maximum fiberboard conditions.

While limited corrosion was observed on the LE3 drum, it is noted that corrosion also occurred on the drums of the first two life extension packages. In comparison, the LE3 drum began corroding later, and has corroded less than the other two packages. Some of this corrosion may be related to fabrication practices used to modify these drums, although some is also associated with non-modified areas. Corrosion of the stainless steel drum might be expected if moisture migration within the fiberboard leads to the concentration of chlorides on the drum surface, or from biological influences. However, none of the corrosion observed in any of these packages represents a challenge to the drum integrity.

Several specific attributes of the LE3 package can be compared directly to laboratory or literature data. Each of these is discussed separately below.

Thermal conductivity changes

Package LE3 data were examined for evidence of change in fiberboard thermal conductivity during the testing. The methodology follows that described in Reference 3. The sidewall region of the lower fiberboard assembly provides a region of relatively constant thermal response. Since the two lower ID thermocouples failed during the course of testing, the upper half of this sidewall region will be used to estimate change in thermal conductivity. This provides the data from 2 ID thermocouples and 2 OD thermocouples, which will be averaged. The temperature along either surface varies little within this region during steady state operation. Since some heat is lost through the top and bottom of the package, the heat conducting through this side region will be less than 19 watts, but it will be assumed constant over time. Heat conduction in this region is in the radial orientation.

Thermal gradient information was examined at several discrete times during periods of steady state operation indicated by the vertical arrows in Figure 13. The thermal conductivity will vary with changes in the temperature gradient and fiberboard thickness, as described by:

$$q/A = k * \Delta T / t$$

where, q/A = heat flux (assumed constant)

k = thermal conductivity

ΔT = radial temperature gradient

t = lower assembly radial thickness (inspection data shows an initial average radial thickness of 4.758 inches, and an average change in radial thickness of $-5.56 \text{ E-4 } \%/ \text{week}$)

At each of the arrows in Figure 13, the radial temperature gradient and lower assembly thickness are combined per this relationship to get a value proportional to the thermal conductivity (see Table 4). Any change in this value over time is proportional to the change in actual thermal

conductivity. (Since the actual heat flux through this region of fiberboard is unknown, the actual thermal conductivity cannot be calculated directly.) While thermal conductivity will vary with fiberboard temperature, variation in the mean temperature is sufficiently low among the selected data to have a negligible impact on thermal conductivity.

These results are listed in Table 4 and plotted in Figure 14, along with normalized laboratory data for sample MSC-5R conditioned at 125°F and tested at 122°F. In this presentation, the relative thermal conductivity values for LE3 vary, but the overall trend is a very slight increase in thermal conductivity over time (0.21 %/yr). This rate compares favorably with results reported for laboratory samples in Reference 6, including the following:

- In 125°F, dry environment, the average thermal conductivity increased 0.25 %/yr
- In 185°F dry environment, the average thermal conductivity decreased 0.39 %/yr
- In 125°F 70%RH environment, the average thermal conductivity increased 0.89 %/yr
- In 160°F 50%RH environment, the average thermal conductivity decreased 1.37 %/yr

The fiberboard within the upper sidewall region of LE3 is at an average temperature of 142°F. The average moisture level in this region had dropped to ~8 %WME after 26 weeks in test, and continued to drop to ~6 %WME or less. This condition falls within the bounds of the four environments cited above, and the calculated change in thermal conductivity for LE3 is consistent with that seen in the laboratory samples, especially those in the dryer environments. It is noted that the calculated change in thermal conductivity for LE3 includes some variation over time, including periods of both increased conductivity and of decreased conductivity. Additional aging of this package may clarify whether there is really a net change or not. Minor variations in the fiberboard moisture level could account for some of the observed variation in thermal conductivity.

Fiberboard mass changes

Variation in the total weight of the package is summarized in Figure 9. Variation in the weight of the upper fiberboard assembly and the removable fiberboard sections is shown in Figures 10 and 11, respectively. All fiberboard within the package was removed and weighed on relatively few occasions (to minimize the opportunity for inadvertent moisture changes). Additional information regarding fiberboard weight can be obtained from the total package weight, assuming the package weight has changed only as a result of fiberboard weight change and shield corrosion.

A heavy corrosion layer was noted on the shield after 26 weeks (21.3 weeks at temperature), and was not observed to change significantly during subsequent inspections. If the weight of all fiberboard is subtracted from the total package weight, the remaining components had a combined baseline weight of 138.9 kg. After 43.7 weeks at temperature (the next time all fiberboard was weighed), the remaining components had a combined weight of 139.1 kg. This residual weight has remained approximately constant since that time. This weight gain of ~200g is attributed primarily to shield corrosion. It will be assumed that the shield weight increased uniformly by 200g over the first 26 weeks, and has remained constant since then. Therefore, any additional change in the package weight (after adjusting for shield corrosion) represents fiberboard weight change. The fiberboard weight based on this method is plotted in Figure 15 with the “x” symbols. The red squares in Figure 15 show the combined total weight of all fiberboard sections. There is very good

agreement between the two methods in Figure 15, indicating that the greater amount of data from package weight can be used to represent the change in fiberboard weight as well.

Initially, the fiberboard had a uniform moisture content of ~11 %WME, which corresponds to ~10 wt%. This moisture content is typical of many fiberboard assemblies. It is also the maximum allowed moisture content for new fiberboard, although it is recognized that the moisture content of new material will change (increase or decrease) depending on ambient conditions. With a total fiberboard weight (assuming 6.622 kg nominal weight for the air shield and bearing plates) of 34.978 kg, the fiberboard initially contained ~3.18 kg of water. After 227.1 weeks at temperature, the average fiberboard moisture content was ~11 %WME below the lower bearing plate, and ~6.8 %WME above the lower bearing plate, giving an overall (weighted) average of 7.4 %WME (or 7.6 wt%). Assuming the original dry fiberboard weight remained the same, the fiberboard contained ~2.42 kg water after 227.1 weeks at temperature. During this time, approximately 0.76 kg (or 0.76 liters) of water was lost from the fiberboard.

The total fiberboard weight decreased by 2.419 kg over 227.1 weeks at temperature, with water loss accounting for ~0.76 kg of this amount. Therefore, the fiberboard itself lost ~1.66 kg, or 5.2% of the original dry weight. This rate of weight loss would be ~1.2 %/year. The majority of fiberboard in package LE3 has been maintained at ~140°F and ~6 %WME for most of its history. Per Reference 7, a moisture content of 6 %WME corresponds to an equilibrium relative humidity of ~30 – 40% at room temperature, and would increase to ~45 – 55 %RH at 140°F. The weight loss model developed in Reference [6] predicts a rate of weight loss of ~0.9 %/year for 140°F 50 %RH, in good agreement with the observed fiberboard weight loss. A similar rate would also be predicted for the cooler, moister environments typical of the bottom fiberboard layers.

Fiberboard dimension changes

Changes in radial thickness of the lower fiberboard assembly are shown in Table 1. If these data are combined with changes in radial thickness of the upper fiberboard assembly, the overall trend of these data is a decrease in thickness at a rate of 0.03 %/year, although there is significant scatter. (The baseline values are excluded from this trend because they represent a different moisture condition than the dryer subsequent data.) Laboratory data for length and width changes have not been modeled, but the environment of LE3 is bracketed by the following 3 laboratory sample test environments:

- 160F 50%RH, average rate of change in length & width of 0.55 %/year
- 125F dry, average rate of change in length & width of 0.11 %/year
- 125F 70%RH, average rate of change in length & width of 0.13 %/year

The laboratory data are conservatively bounding to the changes in radial thickness of package LE3 fiberboard.

As the fiberboard moisture migrates toward the bottom of the assembly, the bottom layers will tend to compress since the added moisture weakens the fiberboard and these bottom layers are under compressive load from the package internal components. In addition, the higher fiberboard regions that have lost moisture will shrink [8]. Both of these effects contribute to the 2.6% decrease in fiberboard height seen at the first inspection after the package began aging. Subsequent changes in fiberboard height result from aging of the fiberboard, and show a rate of

decrease in height of 0.40 %/year (Figure 16). In comparison, the model for fiberboard height [6] predicts a change of 0.3 %/yr at 140F 50%RH, in good agreement with this observation.

Related to the change in fiberboard height is the change in axial gap at the top of the package. The axial gap is shown in Figure 17. The baseline axial gap is smaller than nominal (0.545 inch vs 0.8 inch). During initial assembly, the axial gap was smaller on one side (0.459 inch) and interfered with drum closure such that the closure bolts were used to pull the lid tight. From this extreme axial gap condition, interpolation of the data indicate the axial gap exceeded 1 inch after ~20 weeks at temperature. This 1 inch threshold is taken as an action point for packages in service to investigate whether significant package degradation has occurred. The axial gap in LE3 continued to increase, with a maximum value of 1.483 inches after 249 weeks at temperature.

Potential for shield creep

The data in Table 2 summarize measurements of the shield wall thickness for LE3. Changes in the measured wall thickness might occur from the formation and/or shedding of corrosion product, from creep of material at the top toward the bottom of the shield, or from measurement uncertainties. Initially, corrosion product was observed to form with apparent uniformity on the shield, although at later times it may have flaked off locally. A consistent trend in which the thickness at the bottom increases by more than that at the top could be indicative of creep deformation. However, this was not observed in LE3. Rather, the measured thickness varied both up and down at both the top and bottom of the shield.

A similar analysis for the shield in life extension package 2 suggested a creep rate of 0.009 to 0.02 inch/year was active [3] in that package. That shield experienced a temperature of ~250°F (or 66% of the melting temperature). This estimate was bracketed by literature data which describes the creep rate for two extremes of grain size. Reference 9 predicts a creep rate of $\sim 7 \text{ E-9 /sec}$ (0.11 inch/year change in shield thickness) for the small grain size (10 microns), and $\sim 1 \text{ E-11 /sec}$ (0.00016 inch/year change in shield thickness) for the large grain size (1 mm). This same literature data predicts lower creep rates for the lower shield temperature of LE3. Specifically, at 170°F (58% of the melting temperature), the predicted creep rate for the small grain size is $\sim 3 \text{ E-10 /sec}$ (0.005 inch/year change in shield thickness), and for the large grain size is $< 1 \text{ E-12 /sec}$ ($< \sim 1 \text{ E-5}$ inch/year change in shield thickness). Since these predictions are at least an order of magnitude less than those for the higher temperature, it is reasonable to expect any creep of the LE3 shield will occur at least an order of magnitude slower, or on the order of 0.001 inch/year. Even with 5 years aging, the potential shield deformation is not likely to exceed 0.005 inch, which is small compared to measurement variability and uncertainties introduced by the corrosion layer. Therefore, while a very small amount of lead creep may have occurred, it is not enough to be readily detectable or to cause concern. Typical shields in a storage application will experience an average temperature even less than the LE3 shield, and creep rates for them should be negligible.

Conclusions

Life extension package LE3 has been instrumented and exposed to a bounding storage condition to help identify the extent to which laboratory test results for fiberboard, O-rings and other components apply to a full-scale package. Results to date indicate the following:

- As seen with the previous life extension packages, the presence of a thermal gradient across the fiberboard assembly leads to a corresponding moisture gradient, with moisture moving preferentially to the cooler regions of the package. Following the initial re-distribution of moisture, the package eventually lost ~0.76 kg water.
- With elevated temperature, the fiberboard loses mass and shrinks. Shrinkage is greatest in the axial direction. The rates of weight loss and dimensional changes are generally consistent with laboratory samples.
- Estimates of the change in fiberboard thermal conductivity (in the radial direction) vary, but show a slight increase over time, on average. Laboratory thermal conductivity samples show an increase over time at 125°F, and a decrease over time at 160°F and higher. At an average fiberboard temperature of ~140°F, the observed LE3 behavior is consistent with the laboratory samples.
- The lead shield experienced significant corrosion within the first 26 weeks, with relatively little subsequent change in appearance. Corrosion product on the upper portion of the shield eventually became somewhat yellow in appearance.
- There is no consistent trend of change in shield dimensions that would suggest any creep deformation has occurred. This is consistent with literature data that indicate a small amount of creep might be expected over the 5 years aging experienced by LE3, but such deformation could easily be masked by the corrosion buildup and measurement uncertainties.

With the exception of the increased axial gap (to 1.48 inch), and modest staining / corrosion at some of the welds on the drum, package LE3 continues to conform to requirements and maintain its full functionality. It remains in test.

References

- [1] WSRC-TR-2005-00014, Rev. 2, "Task Technical and Quality Assurance Plan for Full-Scale Testing of Model 9975 Packages to Support Life Extension Model Development (U)", June 2008.
- [2] WSRC-TR-2001-0286, Rev. 4, "SRS Surveillance Program for Storage of Pu Material in KAMS".
- [3] SRNL-STI-2010-00185, "Model 9975 Life Extension Package 2 – Final Report", W. L. Daugherty, April 2010.
- [4] SRNL-STI-2011-00113, "Model 9975 Life Extension Package 1 – Final Report", W. L. Daugherty, March 2011.
- [5] OBU-NMM-2004-00060, "KAMS T-1 Temperature Readings", S. M. Herlihy, March 25, 2004.
- [6] SRNL-STI-2013-00020, "Status Report – Cane Fiberboard Properties and Degradation Rates for Storage of the 9975 Shipping Package in KAMS", W. L. Daugherty, January 2013.

- [7] SRNL-L7200-2008-00007, “Correlation between Cane Fiberboard Moisture Content and Relative Humidity”, W. L. Daugherty, December 10, 2008.
- [8] SRNL-STI-2012-00429, “Analysis of the Axial Gap vs Fiberboard Moisture Content in a 9975 Shipping Package”, W. L. Daugherty, September 2013.
- [9] “Deformation-Mechanism Maps, The Plasticity and Creep of Metals and Ceramics”, H. J. Frost (Dartmouth College) and M. F. Ashby (Cambridge University), web version <http://engineering.dartmouth.edu/defmech/>.

Table 1. Dimensional data for LE3 lower fiberboard assembly

Time at Temperature	Radial thickness at top (inch)	Change in thickness (%)	Diameter across bottom (inch)	Change in Diameter (%)	Height (inch)	Change in Height (%)
Baseline	4.757	0.00	18.029	0.00	26.861	0.00
43.7 wks	4.735	-0.46	18.047	0.10	26.160	-2.61
72.4 wks	4.747	-0.21	18.056	0.15	26.146	-2.66
95.9 wks	4.752	-0.11	18.036	0.04	26.063	-2.97
121.7 wks	4.784	0.57	18.048	0.11	26.016	-3.15
173.6 wks	4.748	-0.19	18.022	-0.04	25.891	-3.61
227.1 wks	4.755	-0.04	18.022	-0.04	25.822	-3.87

Table 2. Moisture content of LE3 fiberboard

Time at Temp (weeks)	Moisture Content (%WME)				
	Fiberboard sidewall ID	Fiberboard sidewall OD	Upper removable section	Lower removable section	Bottom of lower assembly
0.0	11.3	10.5	9.1	9.1	12
1.4			14.4	46	
4.4			12.2	67	
6.3			12.7	68	
12.0			12.4	100	
23.0	<6	11.7	11.1		
35.1	<6	10.3	10.1		
43.7	<6	9	9.9	100	100
54.6			8.7	44	
72.4	<6	8	10.1	100	100
80.6			10.3	91	
95.9	<6	<6	9.3	100	100
105.9			8		
121.7	<6	6.7	9	48.9	100
137.4			9.6	32	
154.4			7	46	
163.9			7.5	24.2	
173.6			7.8	14.7	
189.3			6.8	17.3	
210.1	<6	<6	<6	15.5	20.2
227.1	6.6	6.9	7.2	14.3	15
239.9	<6	<6	<6	12.5	

Table 3. Dimensional measurements of LE3 shield to investigate the potential for lead creep.

Time at Temperature	Avg Radial Thickness at bottom of shield (inch)	Avg Radial Thickness at top of shield (inch)	Difference (Bottom – Top) (inch)
Baseline	0.547	0.544	0.003
43.7 wks	0.558	0.560	-0.002
72.4 wks	0.560	0.570	-0.010
95.9 wks	0.570	0.565	0.005
121.7 wks	0.573	0.550	0.023
227.1 wks	0.560	0.568	-0.008

Table 4. LE3 data used to estimate changes in fiberboard radial thermal conductivity

Time at Temp. (weeks)	Mean Fiberboard Temperature (°F)	Avg Fiberboard Radial Temp. Gradient, ΔT (°F)	Fiberboard Radial Thickness, t (inch)	$\Delta T / t$ (°F/inch)	Normalized Thermal Conductivity, $4.829 / (\Delta T / t)$
22.3	142.6	32.7	4.757	6.873	1.000
35	143.2	32.4	4.757	6.811	0.991
66.7	144.4	32.1	4.756	6.749	0.982
76.3	142.4	32.2	4.756	6.770	0.985
115.6	141.5	32.5	4.755	6.835	0.994
129	142.7	33.4	4.755	7.025	1.022
169	142	33.5	4.754	7.047	1.025
183.3	142.6	33.9	4.753	7.132	1.038
205.4	144	32.3	4.753	6.796	0.989
227	143.8	32.7	4.752	6.881	1.001
235.7	145.3	31.8	4.752	6.692	0.974

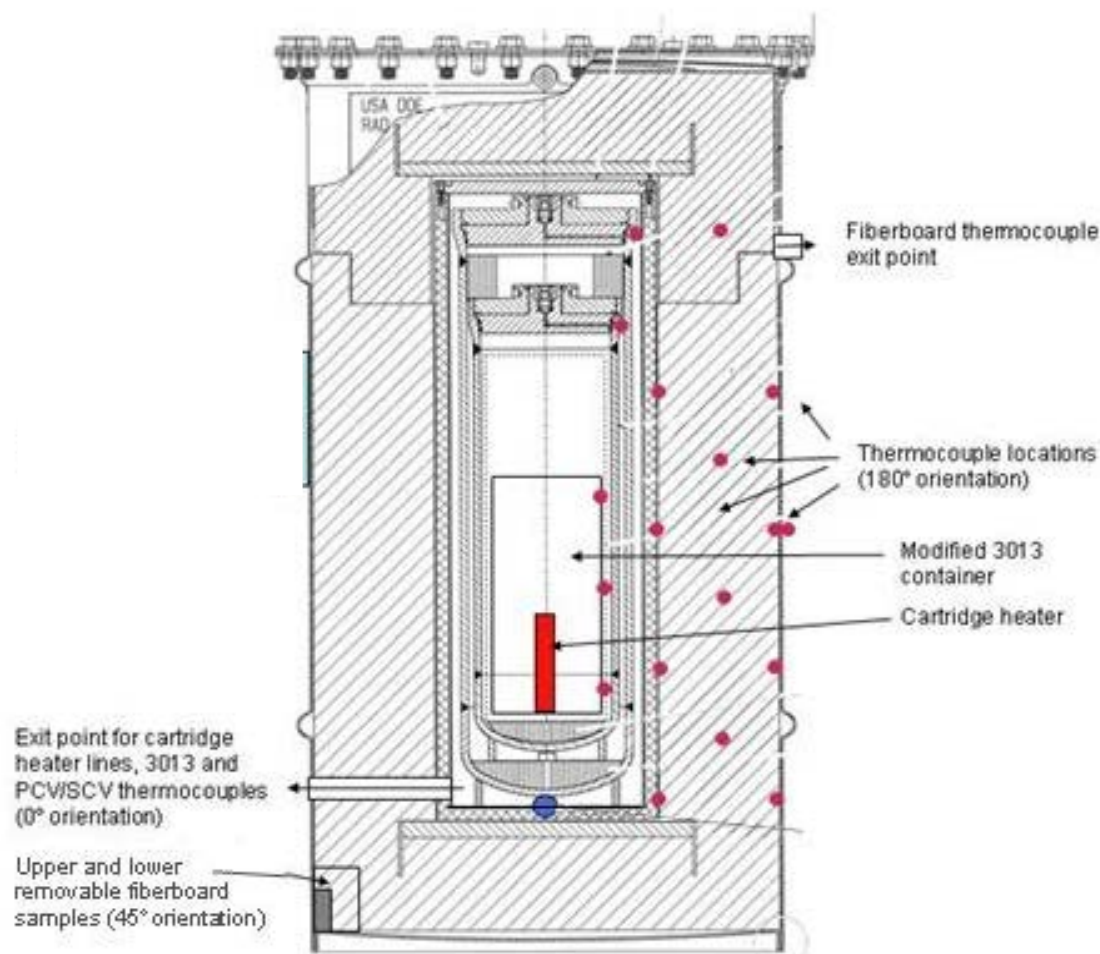


Figure 1. Cross section of 9975 package, showing added instrumentation and examination features.



Figure 2. Configuration of LE3 to provide external heat source. In (a) LE3 is seen with insulation on top, inside the modified 55-gal drum. In (b), the 55-gal drum is wrapped with a drum heater and insulation.



Figure 3. Liquid condensate on the underside of the drum lid and on the air shield, observed after 2 weeks in test.

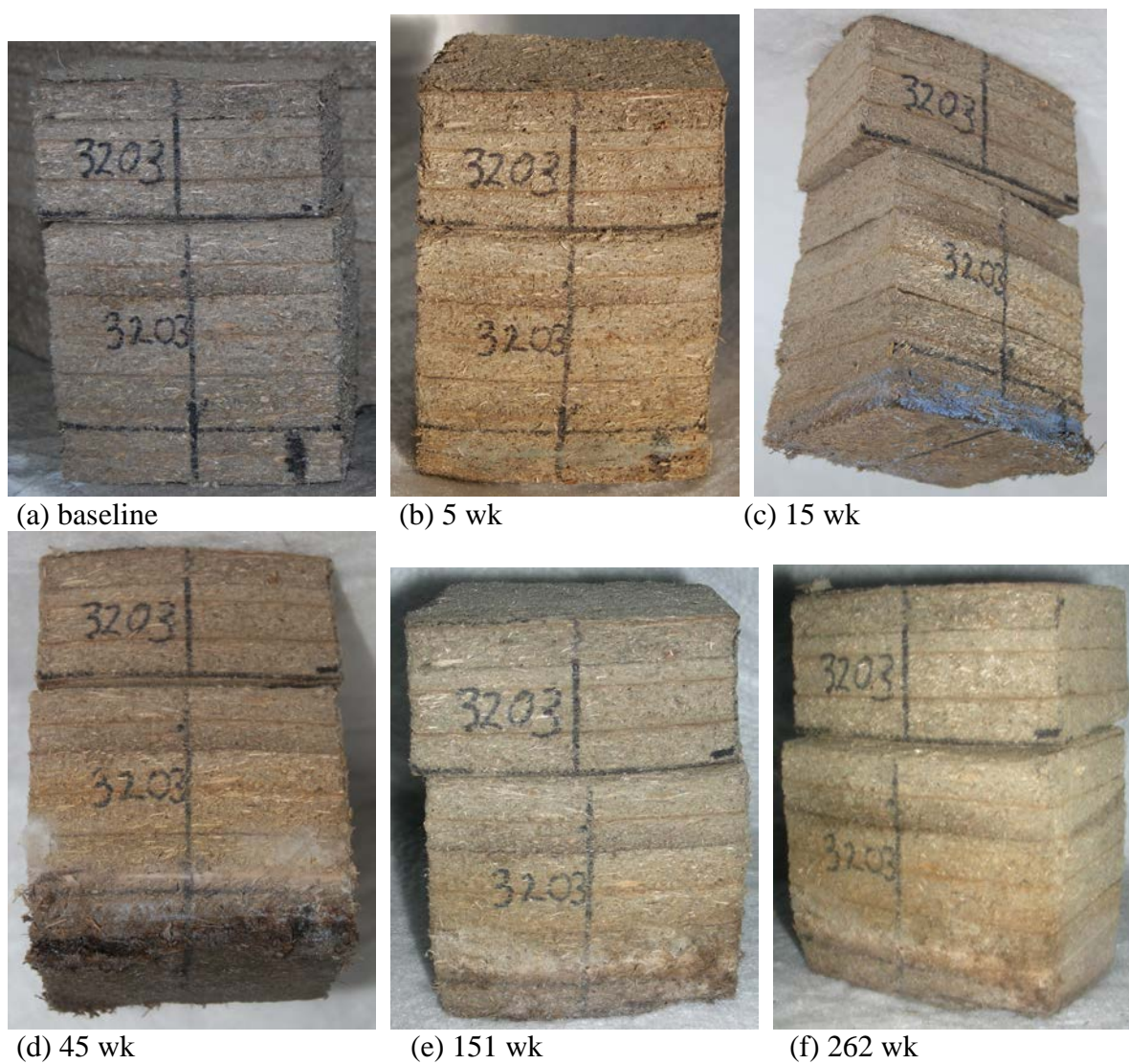


Figure 4. Sequence of photographs of small removable fiberboard sections from test LE3.



(a) Baseline



(b) 26 weeks



(c) 109 weeks



(d) 246 weeks

Figure 5. Progression of shield corrosion. Corrosion was uniformly heavy by week 26, and the upper portion (adjacent to the upper fiberboard assembly) gradually darkened somewhat by week 109. Very little change has been observed since that time.



Figure 6. Corrosion of the drum interior along the bottom crevice, observed after 109 weeks in test.



(a)



(b)

Figure 7. Corrosion of the drum exterior at a stitch weld along the bottom edge, observed after 119 weeks in test (a). After 151 weeks in test, some additional corrosion is observed at this location (b).



Figure 8. Corrosion / staining of the drum interior at the upper flange stitch welds, observed after 190 weeks in test.

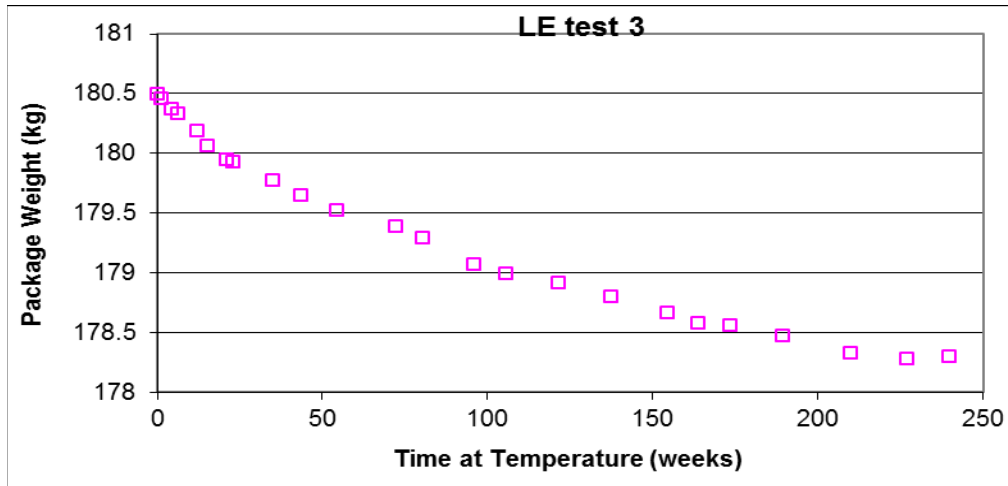
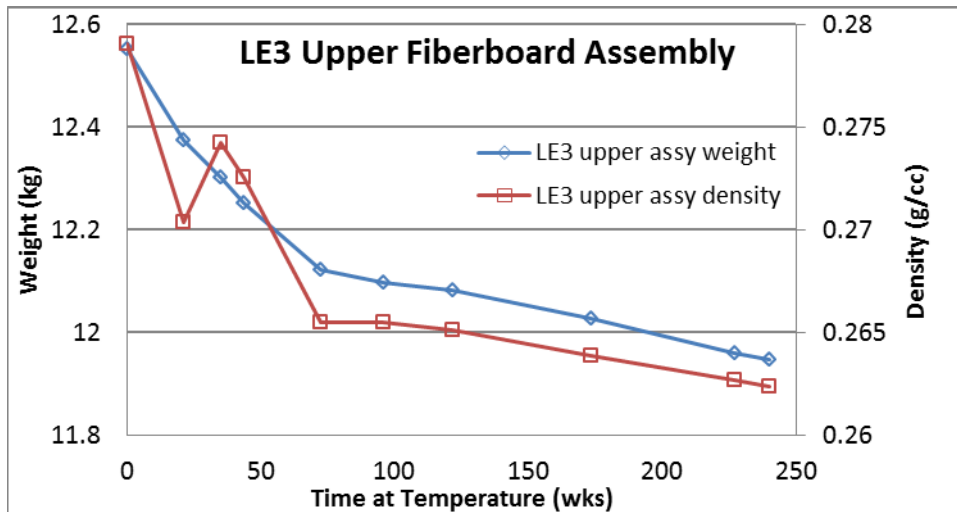
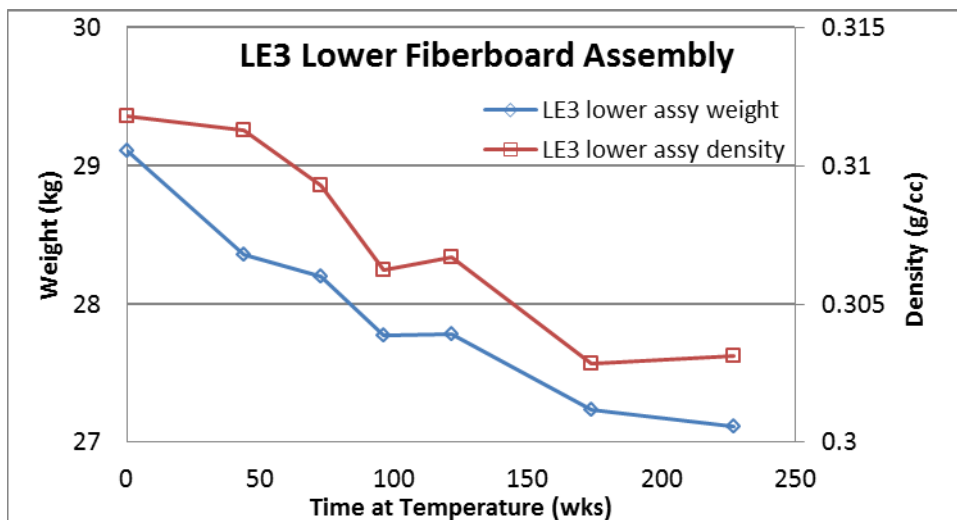


Figure 9. Package weight of LE3 over time.



(a) upper assembly



(b) lower assembly

Figure 10. Weight and density variation for LE3 fiberboard assemblies.

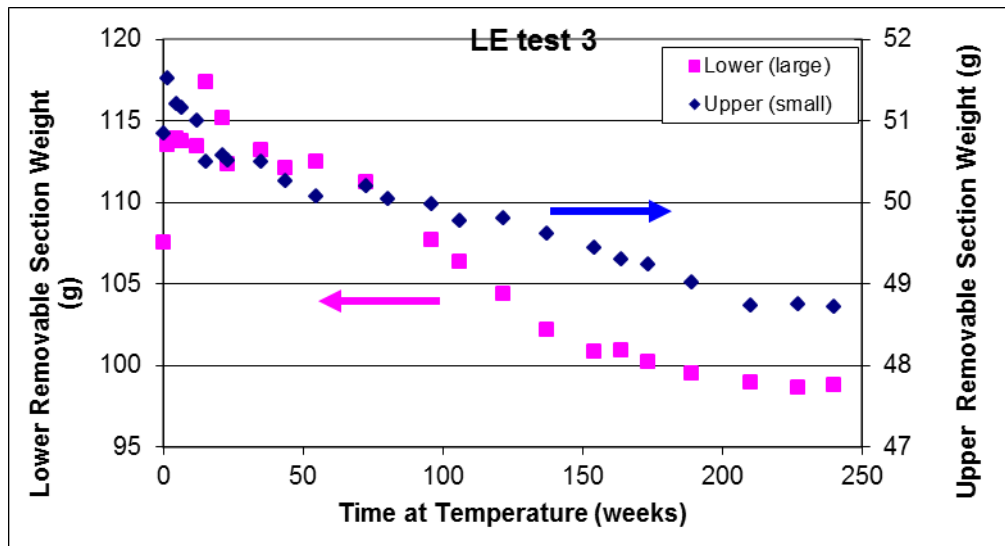


Figure 11. Weight of removable fiberboard sections from life extension test 3.

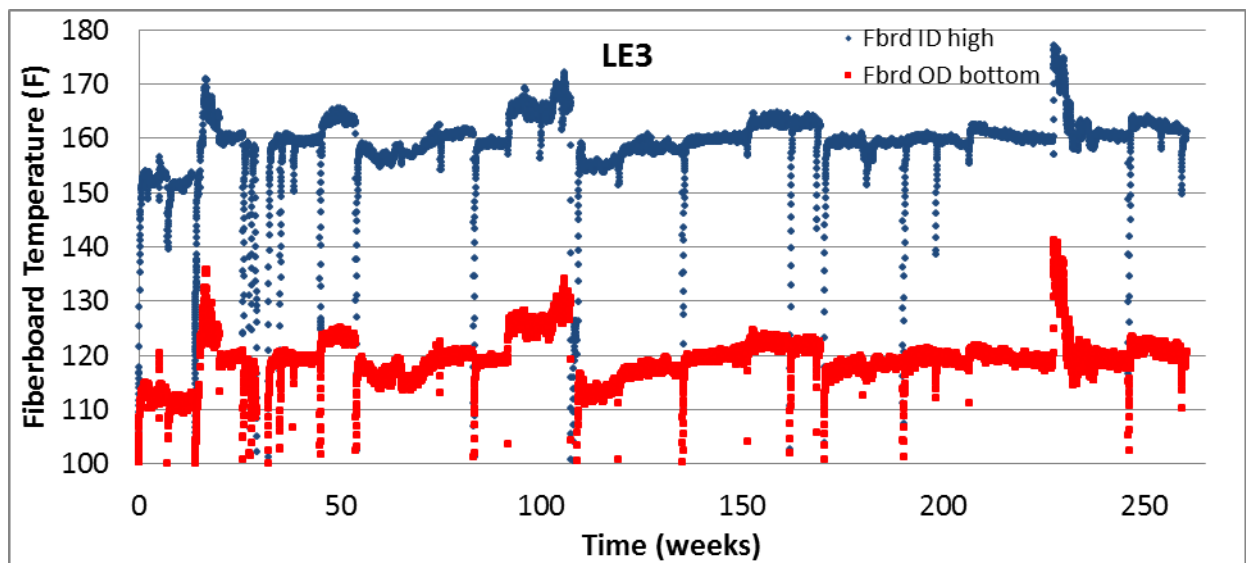


Figure 12. Temperature variation for package LE3 at the hottest (blue symbols) and coolest (pink symbols) instrumented fiberboard locations (highest fiberboard ID location and lowest fiberboard OD location – refer to Figure 1).

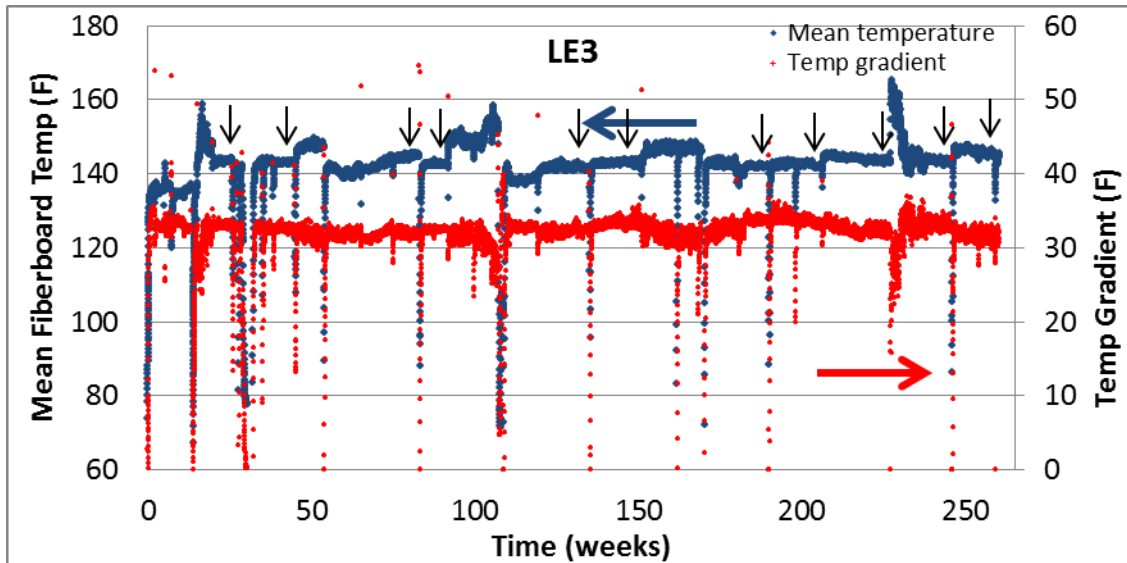


Figure 13. LE package 3 average radial temperature gradient (red symbols) in the fiberboard based on the upper 2 fiberboard ID thermocouples and the upper 2 fiberboard OD thermocouples, and average fiberboard temperature (blue symbols). The vertical arrows indicate times during steady state operation for which the relative thermal conductivity of the fiberboard was estimated.

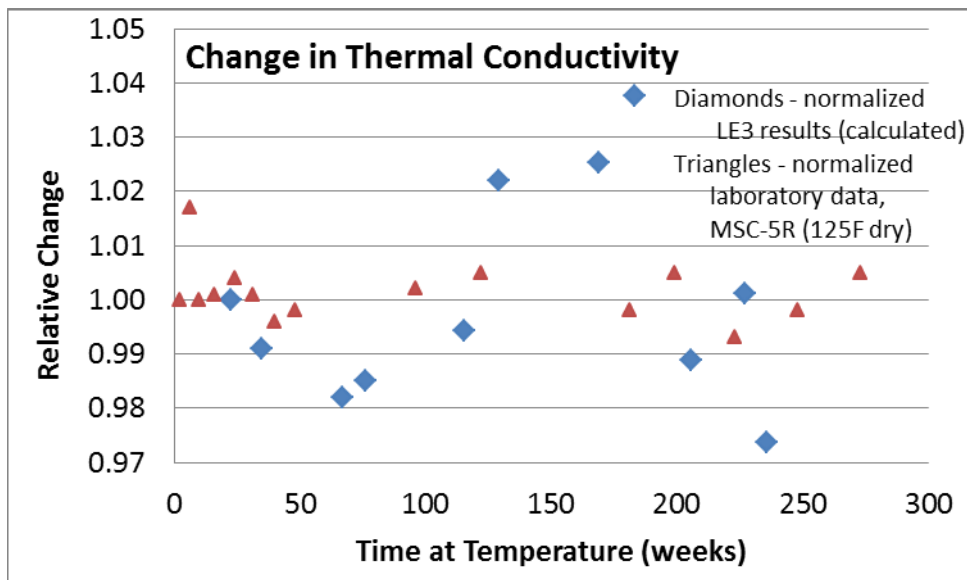


Figure 14. Relative change in radial thermal conductivity estimated from LE3 thermal gradient data. Comparable data for laboratory sample MSC-5R (conditioned at 125°F) are also shown. The mean test temperature for the laboratory sample was 122°F.

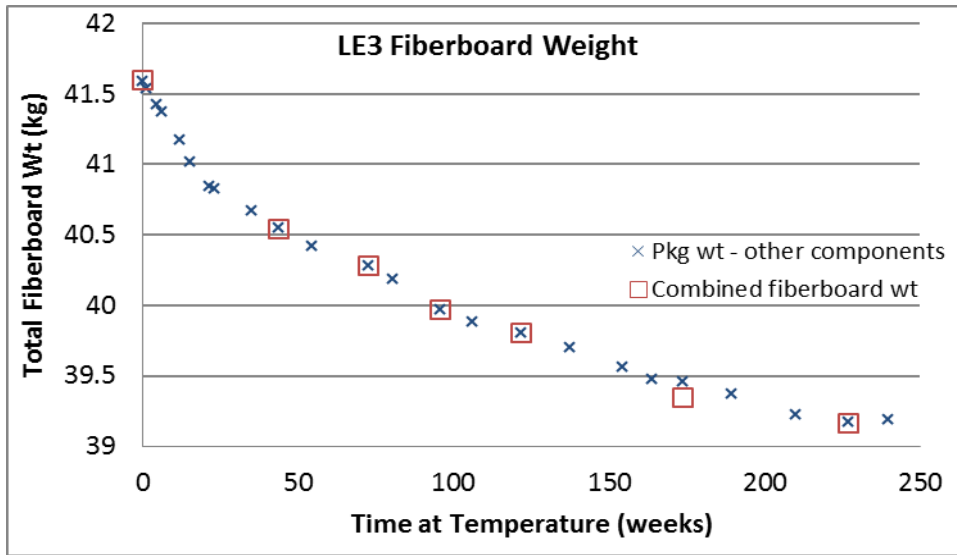


Figure 15. Variation in total fiberboard weight over time based on two methods. The combined fiberboard weight (red squares) is the total weight of all fiberboard sections. The blue “x”s show the difference between the total package weight and all non-fiberboard components.

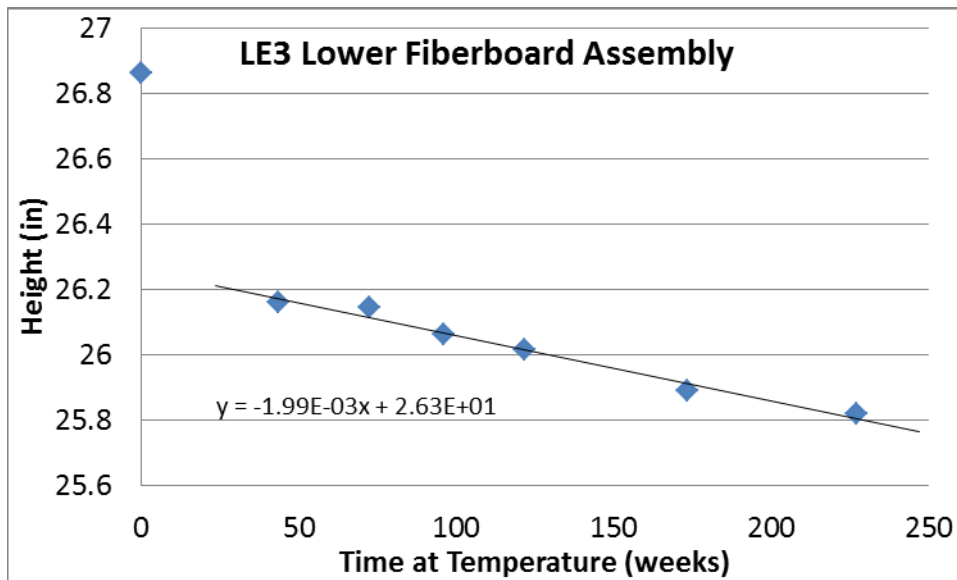


Figure 16. Change in height of the lower fiberboard assembly

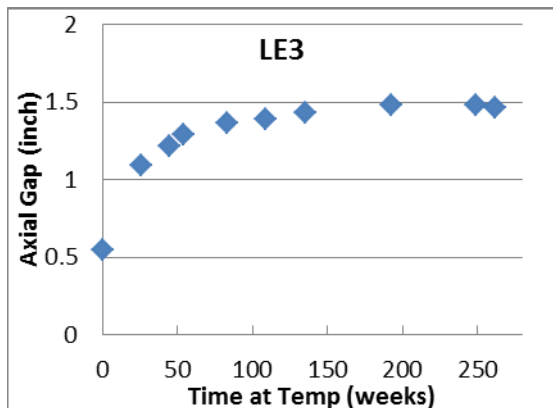


Figure 17. LE3 axial gap change over time. Interpolation indicates the axial gap exceeded 1 inch at ~20 weeks

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