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Materials Assessment of Insulating Foam in the 9977 Shipping Package for Long-Term Storage - Annual Report

A. J. McWilliams

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REVIEWS AND APPROVALS

Materials Assessment of Insulating Foam in the 9977 Shipping Package for Long-Term Storage -Annual Report

APPROVALS:

A.J. McWilliams ____________________ Date ____________________
Author, Materials Science and Technology

W. L. Daugherty ____________________ Date ____________________
Technical Review, Materials Science and Technology

B. L. Garcia-Diaz ____________________ Date ____________________
Manager, Materials Science and Technology

J. M. Jordan ____________________ Date ____________________
NMM Engineering

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1.0 Executive Summary

The 9977 shipping package is being evaluated for long-term storage applications in the K-Area Complex (KAC) with specific focus on the packaging foam material. A rigid closed cell polyurethane foam, LAST-A-FOAM® FR-3716, produced by General Plastics Manufacturing Company is sprayed and expands to fill the void between the inner container and the outer shell of the package. The foam is sealed in this annular space and is not accessible. During shipping and storage, the foam experiences higher than ambient temperatures from the heat generated by nuclear material within the package creating the potential for degradation of the foam. A series of experiments is underway to determine the extent of foam degradation. Foam samples of three densities have been aging at elevated temperatures 160 °F, 160 °F + 50% relative humidity (RH), 185 °F, 215 °F, and 250 °F since 2014. Samples were periodically removed and tested. After approximately 80 weeks, samples conditioned at 160 °F, 160 °F + 50% RH, and 185 °F have retained initial property values while samples conditioned at 215 °F have reduced intumescence. Samples conditioned at 250 °F have shown the most degradation, loss of volume, mass, absorbed energy under compression, intumescence, and increased flammability. Based on the initial data, temperatures up to 185 °F have not yet shown an adverse effect on the foam properties and it is recommended that exposure of FR-3716 foam to temperatures in excess of 250 °F be avoided or minimized.

Testing will continue beyond the 96 week mark. This will provide additional data to help define the long-term behavior for the lower temperature conditions. Additional testing will be pursued in an attempt to identify transition points (threshold times and temperatures) at the higher temperatures of interest, as well as possible benefits of aging within the relatively oxygen-free environment the foam experiences inside the 9977 shipping package.

2.0 Introduction

A review of the materials in the 9977 shipping package was undertaken to identify potential concerns with using this package for long-term storage of plutonium materials in the K-Area Complex (KAC). A preliminary assessment of the 9977 materials degradation potential was performed in 2010.[1] A literature review was performed based on that preliminary assessment that evaluated the primary materials of construction in the 9977 as well as the storage environment.[2] The literature survey identified a dearth of aging behavior data for the FR-3716 foam used in the 9977 shipping container and recommended testing to fill this knowledge gap. The FR-3716 foam serves several roles in the 9977; it is flame retardant, protects the containment vessel (CV) against fire, and provides impact absorption.[3, 4]

Representative flame retardant foam for the 9977 was procured from General Plastics Manufacturing Company in FY12 and received in August 2012 at SRNL. Aging testing started in 2014. One concern is that a change in the foam weight or density could impact criticality control as well as the density is expected to vary some degree through the package as a result of the foam formation.[5] Three densities of foam were tested with approximate densities of 12, 16, and 20 lb/cu ft denoted by the product name FR-3712, FR-3716, and FR-3720, respectively, to judge the impact of this variable due to density variations in the foam in the 9977 package.[1] Testing commenced with accelerated aging of LAST-A-FOAM® FR-3712, FR-3716, and FR-3720 for 9977 related surveillance and aging studies and the progress of those studies is reported herein. Testing temperatures include 160 °F, 160 °F + 50% relative humidity, 185 °F, 215 °F, and 250 °F and are within the product temperature use range (-320 °F – 250 °F). [3]

3.0 Current Exposure and Testing Progress

Representative samples were cut from the three different density foams and different foam-rise orientations. The foam designations: FR-3712, FR-3716, and FR-3720 correspond to the approximate density of 12, 16, and 20 lbs/ft^3, respectively. The samples were labeled, dimensioned, and massed. The samples were loaded into five conditioning furnaces in two different sets approximately one month apart due to furnace availability. The first sample batches were tested at temperatures of 185, 215, and 250 °F for 20.6 months (between 10/2014 and 6/2016). A second set of sample batches was loaded into a furnace set at 160 °F, an environmental chamber at 160 °F with 50% relative humidity and additional samples were added to 185, 215, and 250 °F temperatures for an exposure 19.1 months (between 11/2014 and 6/2016). Samples, **Table 1**, were sent to General Plastics for flammability, chloride leaching, phosphorous leaching, and intumescence testing.

Table 1: Temperature testing and lifetimes of samples sent to General Plastics Manufacturing Company for testing.

Temperature (°F)	160	160 + 50% R.H.	185	215	250
Lifetime (hours)	13,514	13,537	14,490	14,562	14,490

4.0 Data and Analysis

4.1 Thermal Conductivity

Baseline thermal conductivity measurements were conducted per ASTM C-518 on as-received foam from each of the three foam densities and two foam-rise directions 3/2014 as shown in Figure 1 and presented in

Table 2. The thermal conductivity tests were conducted at three different temperature points with a 20 °C difference between the upper and lower plates and the thermal conductivity data are presented at the average of the test temperatures. The thermal conductivity increases with increasing foam density and testing temperature, which agrees with other reported data from polyurethane foams.[6, 7] The foam-rise direction does not significantly influence the thermal conductivity as FR-3716-Parallel (0.05626 W/m-K) is only slightly higher than the FR-3716-Perpendicular (0.05584 W/m-K) by less than 0.8%.

The thermal conductivity measurements for aged samples, Figure 2, Figure 3, and Figure 4 at 25 °C, 55 °C, and 85 °C, respectively, show a fairly stable trend with increasing lifetime. It should be noted that the 250 °F data end at 6,200 hours due to physical deformation of the samples such that the samples no longer fit the testing device. The surfaces first expanded in a bulging fashion and were machined flat to allow for continued testing and then followed by the sample faces collapsing towards the center of the sample so only the edges would be in contact with the testing device.

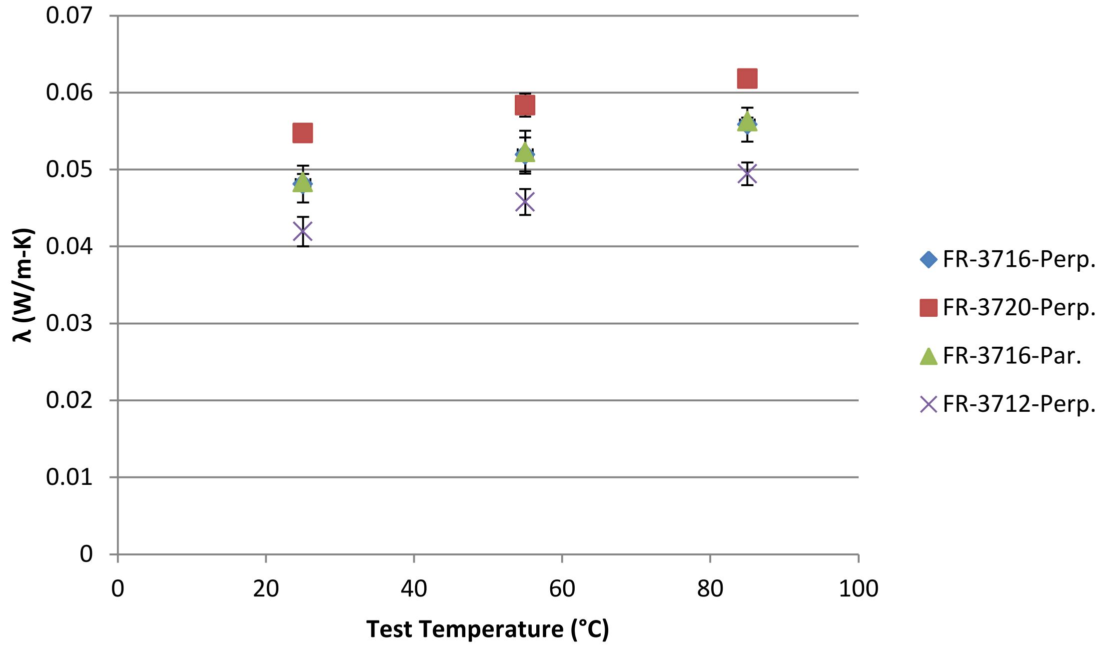

Figure 1: Thermal conductivity (λ) values for the three foam densities (3712, 3716, & 3720) and two different foam-rise directions: perpendicular (Perp.) and parallel (Par.), with 3720 and 3712 foam-rise direction being perpendicular. Error bars are set to two standard deviations.

Table 2: Tabulated thermal conductivity (λ) values for the different foam densities and foam-rise orientations.

Foam ID	Number of Samples	λ (W/m-K)	25 (°C)	55 (°C)	85 (°C)
3716-Perp.	12	Median	0.04812	0.05196	0.05584
		Std. Dev.	0.001198	0.001099	0.001108
3720-Perp.	4	Median	0.05474	0.05836	0.06179
		Std. Dev.	0.000548	0.00075	0.000331
3716-Par.	4	Median	0.04835	0.05226	0.05626
		Std. Dev.	0.000539	0.001394	0.000245
3712-Perp.	4	Median	0.04193	0.04577	0.04944
		Std. Dev.	0.000952	0.000841	0.000732

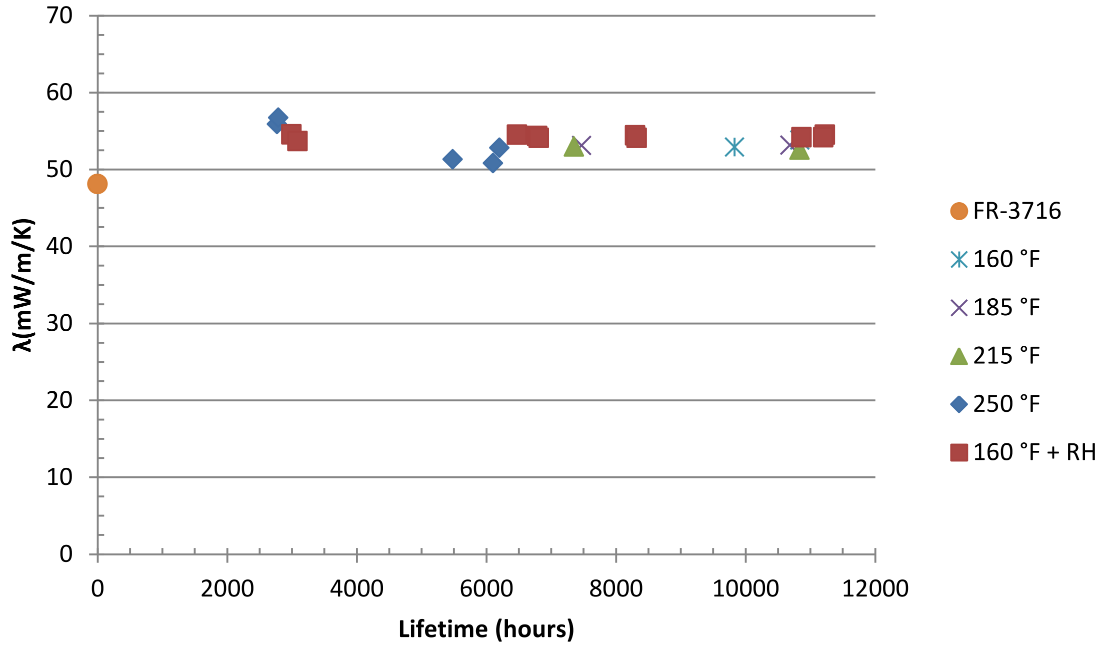

Figure 2: Thermal conductivity measured at 25 °C of FR-3716 at various aging temperatures as a function of lifetime.

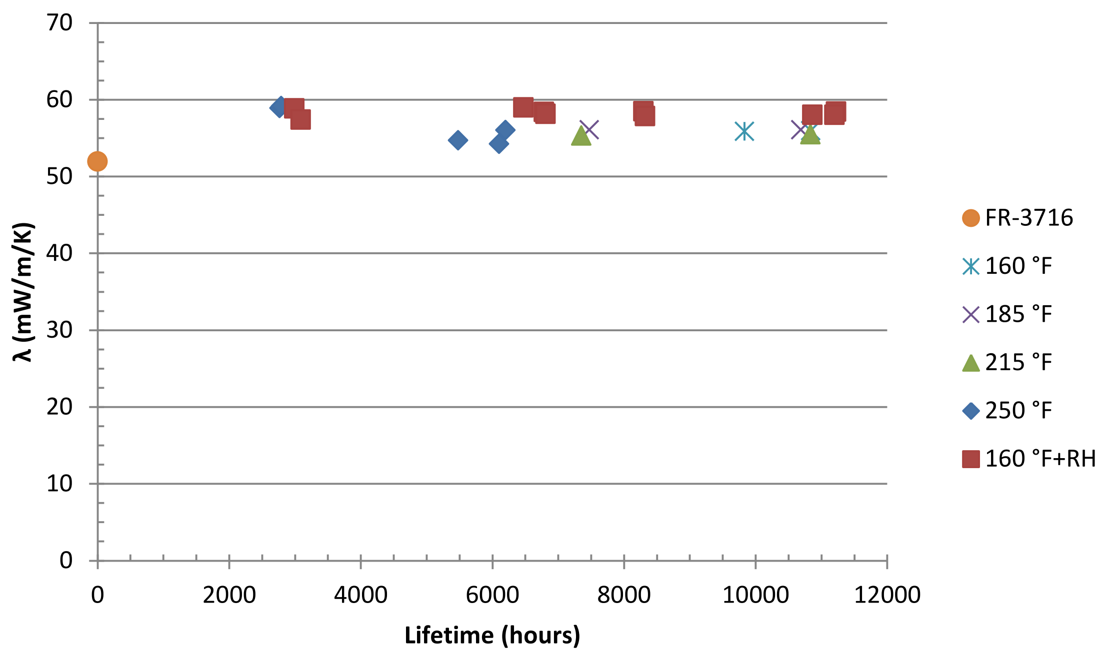

Figure 3: Thermal conductivity measured at 55 °C of FR-3716 at various aging temperatures as a function of lifetime.

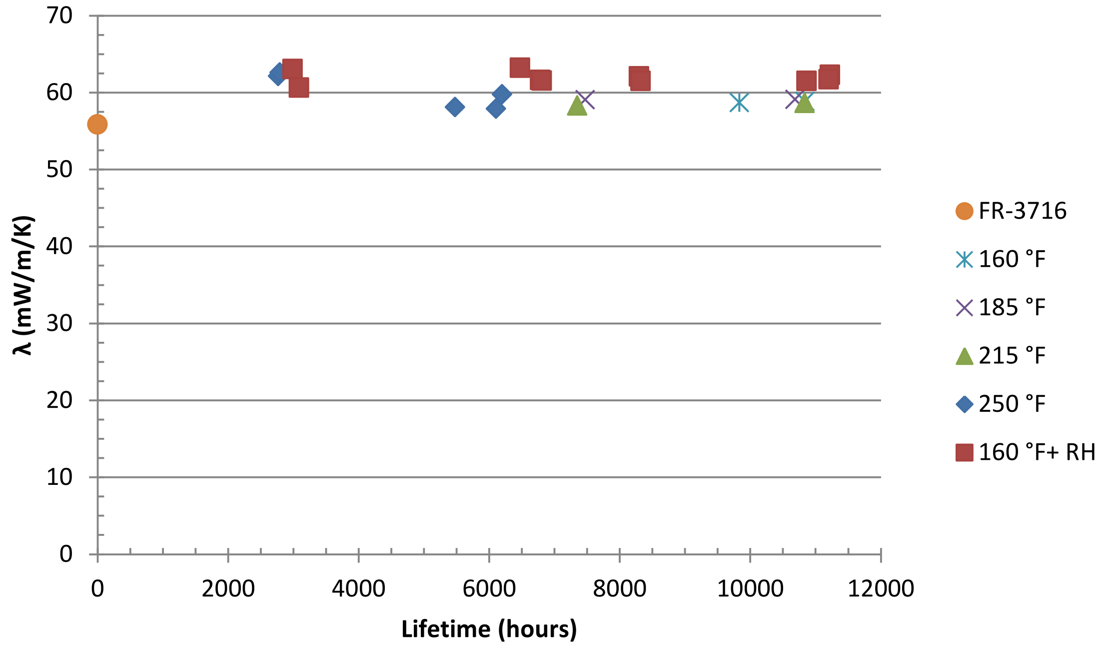

Figure 4 Thermal conductivity measured at 85 °C of FR-3716 at various aging temperatures as a function of lifetime.

4.2 Specific Heat Capacity

Specific heat capacity values, C_p (J/g-K), were obtained using differential scanning calorimetry (DSC), following ASTM E1269-11. Testing shown in Figure 5 indicates a fairly stable heat capacity over time. A reference value for polyurethane foam is included from [8] that was reported to have been measured using a laser flash technique showing that the C_p for the initial FR-3716 foam condition is comparable to the referenced foam.

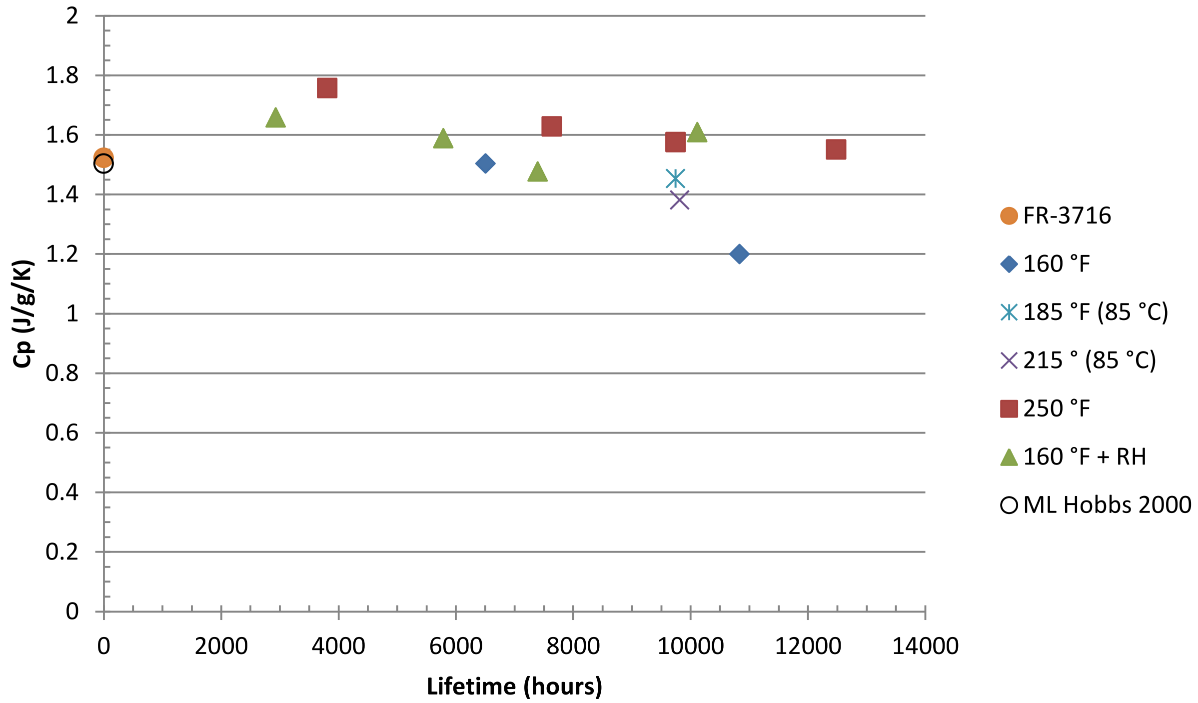

Figure 5: Heat capacity of FR-3716 as calculated at 85 °C for the testing temperatures as a function of lifetime.

4.3 <u>Compression Testing</u>

Due to the use of the FR-3716 foam for impact absorption, it is necessary to determine what, if any, changes occur to the foam compression behavior from conditioning. The compression direction relative to the foam rise direction is depicted in Figure 6. Baseline compression data conducted on as-received foam reveal increasing strength with increasing foam density as shown in Figure 7. The compression strength is slightly larger when performed parallel to the foam rise direction as shown in Figure 8 and given in Table 3.

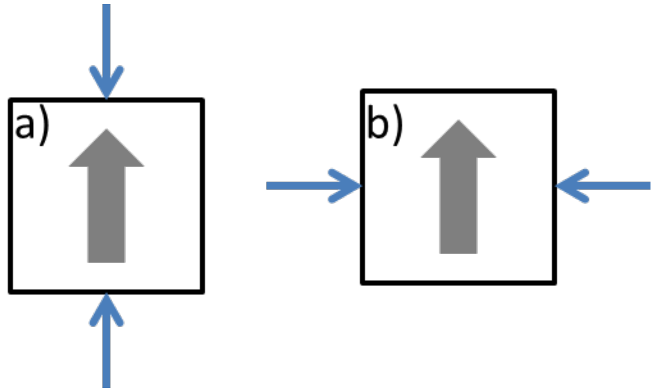

Figure 6: Compression direction indicated by external arrows and rise direction indicated by internal arrows for a) parallel and b) perpendicular orientations with respect to the foam rise direction.

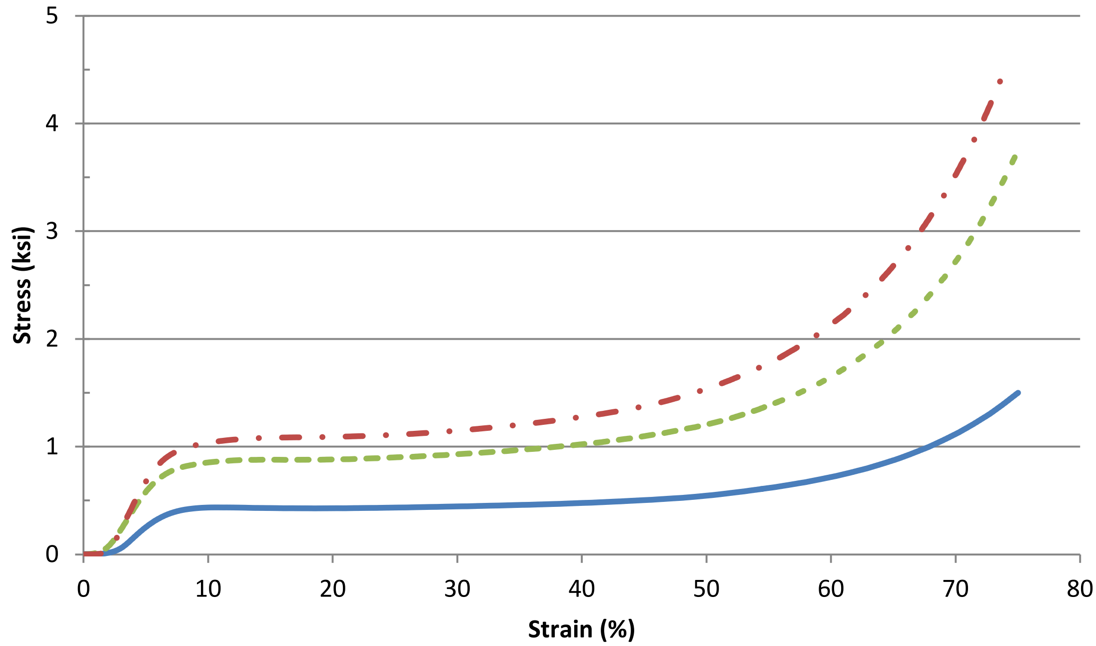

Figure 7: Baseline compression data of the three densities of foam with parallel foam-rise orientation FR-3712 (——), FR-3716 (- - - -), and FR-3720 (— · —).

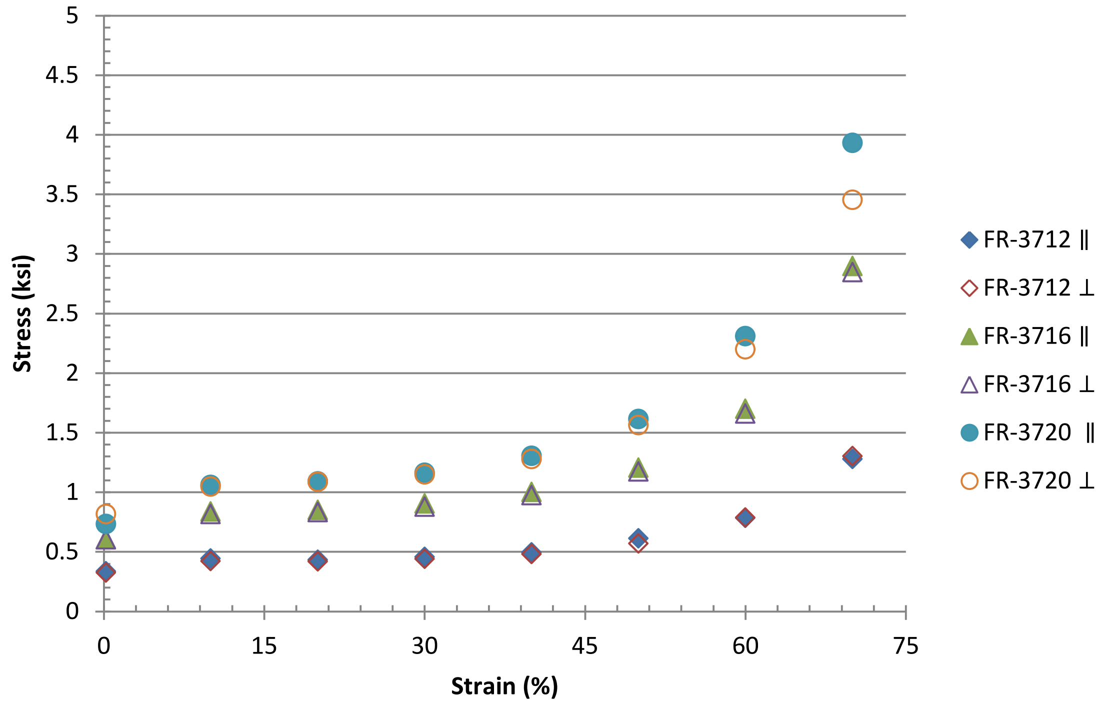

Figure 8: Compression data of three densities of foam with parallel and perpendicular foam rise orientations: FR-3712 ∥ (◆), FR-3712 ⊥ (◊),FR-3716 ∥ (▲), FR-3716 ⊥(△), FR-3720 ∥ (●), FR-3720 ⊥ (○). The initial strain offset differs from the other analysis; however, the curve shape remains similar. The data show the foam density trend of increasing stress at a given strain and minimal difference between foam rise direction.

Table 3: Compression data of three densities of foam with parallel and perpendicular foam-rise orientations.

Foam ID	3712	3712	3716	3716	3720	3720
Orientation	Parallel	Perpendicular	Parallel	Perpendicular	Parallel	Perpendicular
Sample Size	2	2	4	4	2	2
0.2% σ (PSI)	336.6	323.8	605.1	605.5	733.5	815.2
Std. Dev.	18.9	3	32.7	39.4	14.9	35.2
10% σ (PSI)	441.9	420.9	839.2	815.3	1061	1046.8
Std. Dev.	10.7	11.8	25.1	17	6.1	8.7
20% σ (PSI)	432.3	419.3	851.5	831.8	1091.1	1086.8
Std. Dev.	3.2	7.7	28	22.7	8.8	1.2
30% σ (PSI)	456.5	440.7	904.1	878.3	1163.6	1149.5
Std. Dev.	0.6	4.5	30.6	24	9	3.5
40% σ (PSI)	492.9	479.9	1002.8	973.8	1304.1	1279.5
Std. Dev.	4.9	4.7	33.1	25.9	12.2	10.7
50% σ (PSI)	613	569.2	1204.8	1173.1	1614.4	1562
Std. Dev.	49.7	5.2	42.2	38.2	17.5	31.5
60% σ (PSI)	783.6	785.6	1699.8	1660.8	2308.5	2200.3
Std. Dev.	4.4	7.8	63.5	61.7	30.1	88.7
70% σ (PSI)	1277.5	1302.6	2897.6	2846.3	3930.6	3454
Std. Dev.	18.2	5.3	120.9	123.7	87.9	*

*only one sample tested

Rigid closed-cell foams under compression experience three main deformation mechanisms as depicted in Figure 9. The first region deforms in a linearly elastic manner through cell wall bending and cell face stretching, where the initial slope is the Young's modulus. The collapse plateau is indicative of the cells elastic buckling, formation of plastic hinges, and crushing if the cell walls are brittle with little increase in stress. Once the cells have collapsed to the point that opposing walls touch, further strain compresses the foam and densification occurs leading to the sharp increase in stress.[9, 10] The parameters used for analysis of the stress-strain curves are depicted in Figure 10.

Compression testing data are given for foam aged at 250 °F (Figure 11) and at 160 °F with 50% relative humidity (Figure 12). These data indicate a positive correlation between foam density and increased yield stress and the onset of densification at lower strain values and are similar to trends present in the literature.[9, 11] The Young's modulus for FR-3716 at the various testing temperatures (Figure 13) show a noticeable increase followed by a relatively stable period except for samples at 250 °F that decrease back near the original value. This decrease could be indicative of the physical deformations of the samples. The yield stress (Figure 14) is similar in that there is an initial increase and then remains relatively stable. The energy absorbed, (Figure 15 (lbf-in) and Figure 16 (kJ)) as calculated by integrating curve ABCDF from Figure 10, shows little change over time with the exception of the samples aged at the highest testing temperature of 250 °F that have decreasing energy absorption.

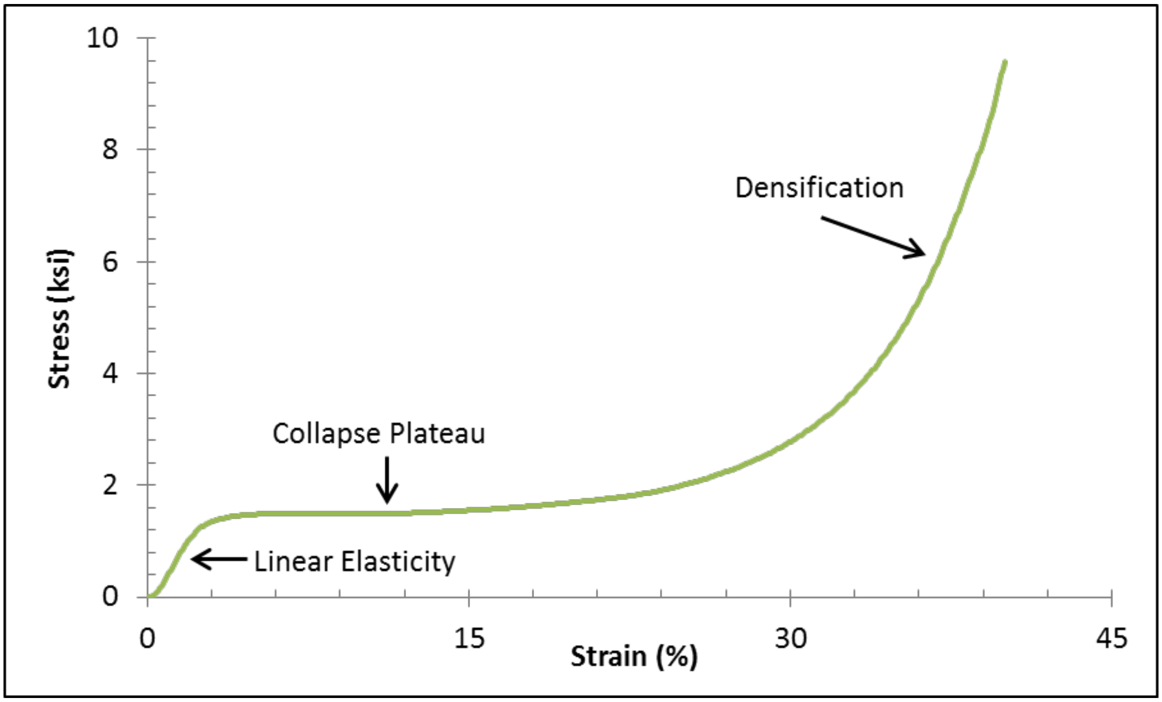

Figure 9: Stress-strain curve for an elastic-plastic foam under compression, illustrating the three deformation mechanism regions

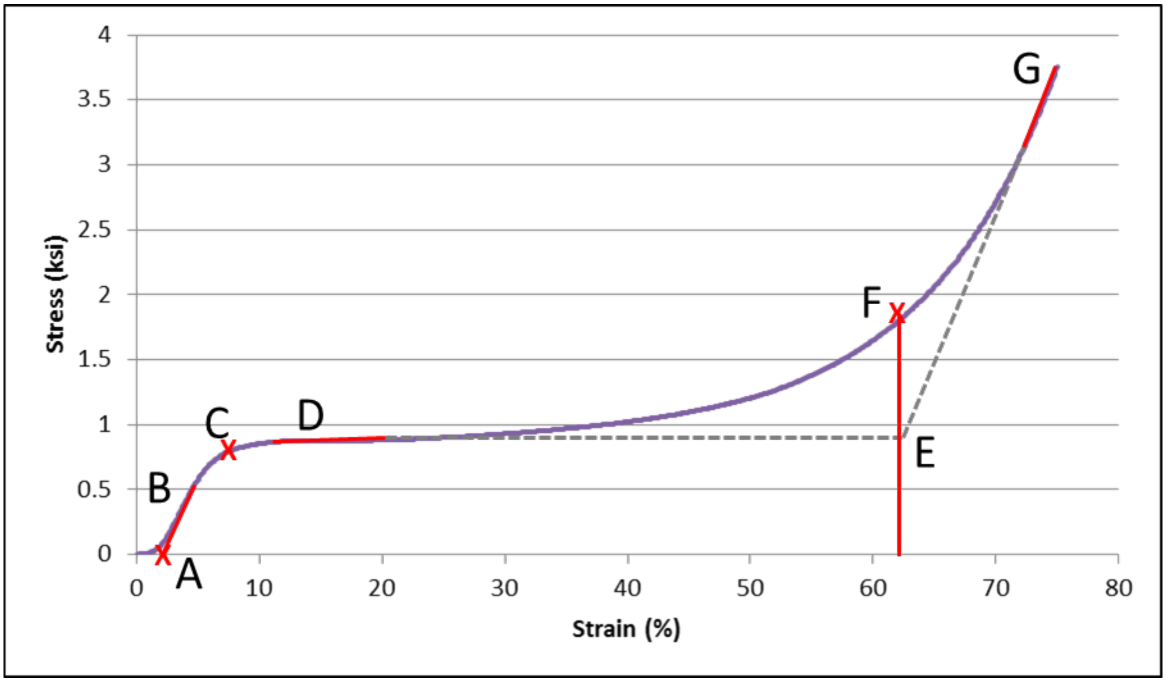

Figure 10 Compression analysis: A) zero-strain offset, B) Young's Modulus, C) Yield point, currently approximated by maximum stress – ASTM D1621: 1st point that increases in strain but not stress, D) slope of collapse plateau, E) intersection point of plateau slope and densification slope, F) point used to calculate absorbed energy extended from point E), G) densification slope.

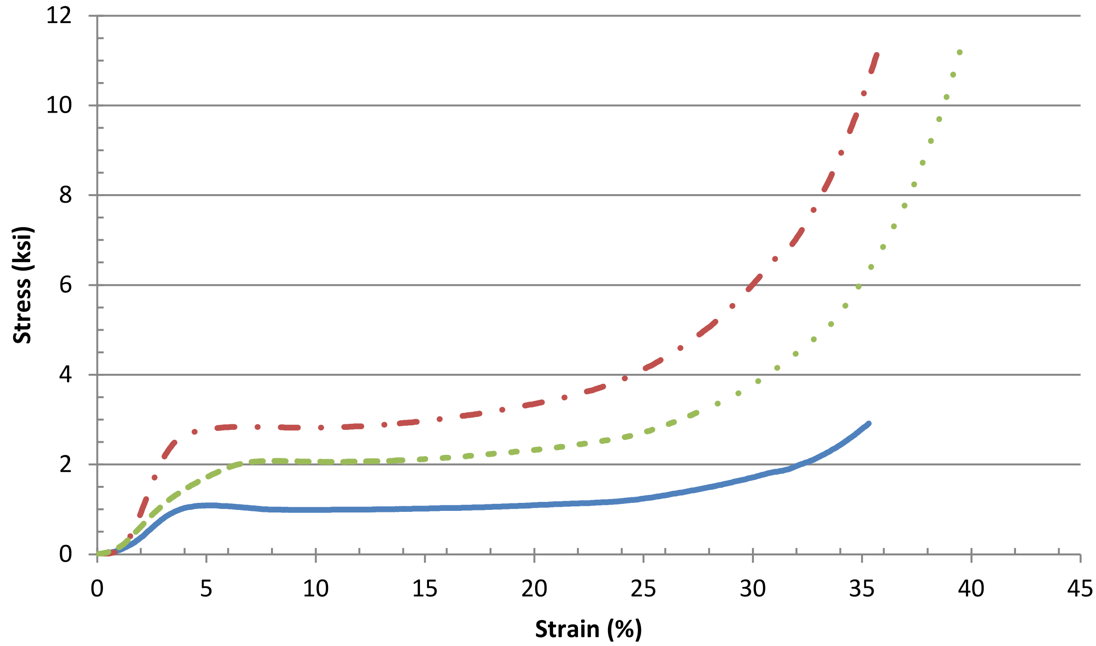

Figure 11: Compression data for foam aged at 250 °F for approximately 3,883 hrs, FR-3712 (———), FR-3720 (– · –), and FR-3716 (- - - -).

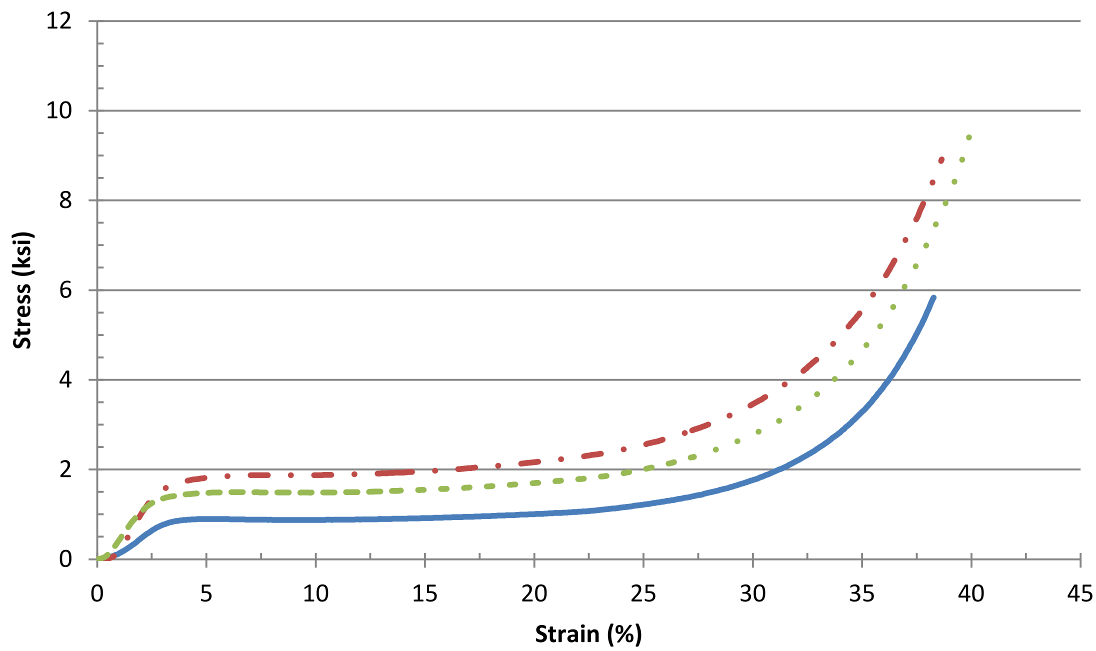

Figure 12: Compression data for foam aged at 160 °F at 50 % RH for approximately 2,930 hrs, FR-3712 (———), FR-3720 (– · –), and FR-3716 (- - - -).

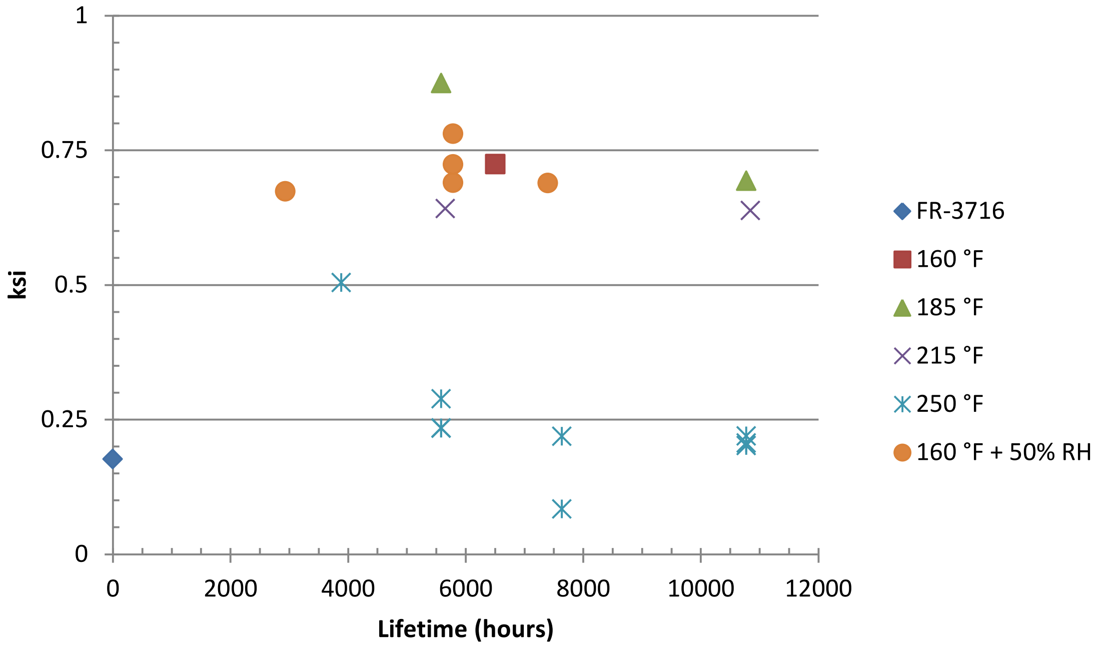

Figure 13: Young's Modulus for FR-3716, including both perpendicular and parallel rise direction for 160 °F + 50% RH and 250 °F.

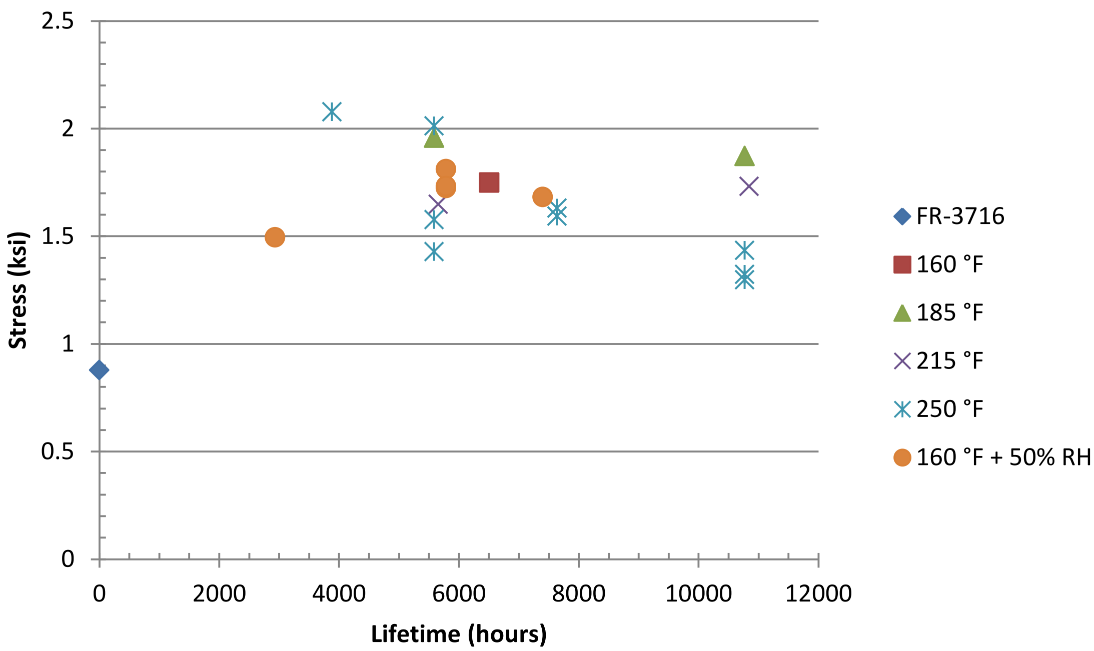

Figure 14: Yield stress for FR-3716, including both perpendicular and parallel rise direction for 160 °F + 50% RH and 250 °F.

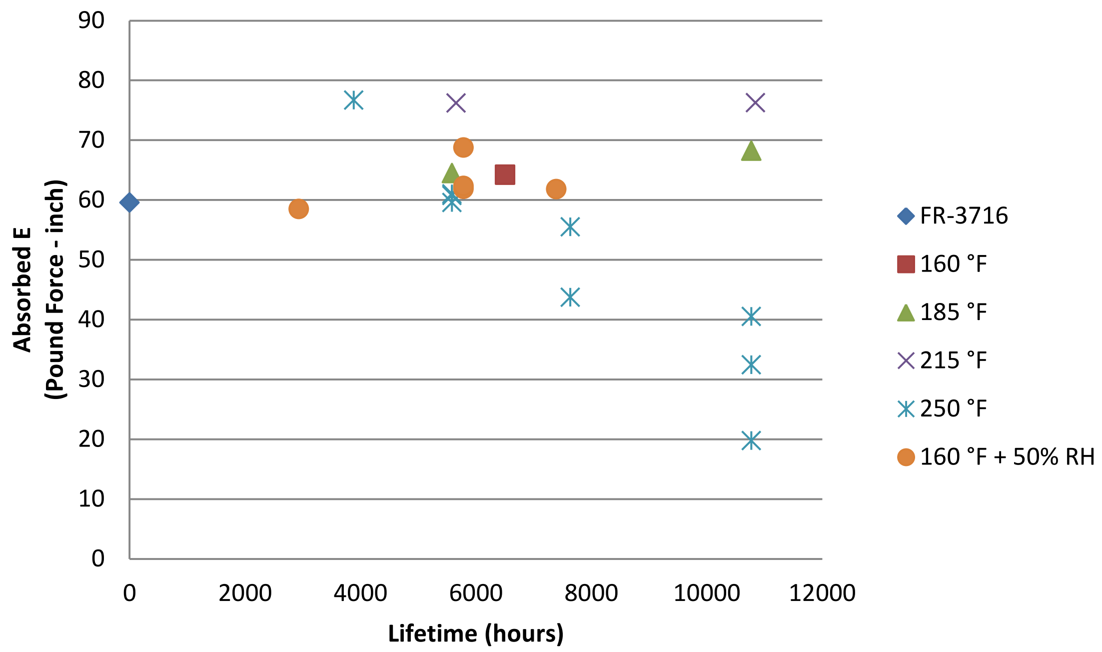

Figure 15: Energy absorbed in pound force-inch, including both perpendicular and parallel rise direction for 160 °F + 50% RH and 250 °F.

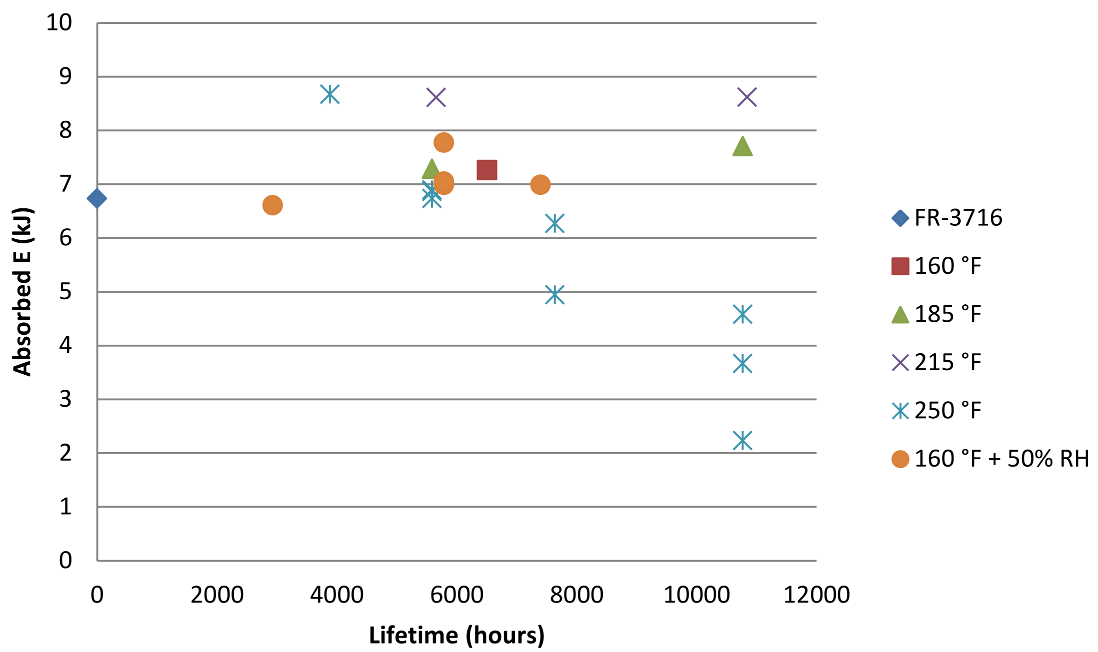

Figure 16: Energy absorbed in kJ, including both perpendicular and parallel rise direction for 160 °F + 50% RH and 250 °F.

4.4 Spectroscopy

Optical spectroscopy characterization using FTIR and Raman has yielded inconclusive data. The inconsistencies with both spectroscopy methods, particularly the varying peak intensity, make even qualitative comparison for samples at different conditioning lifetimes ambiguous. Both spectroscopy methods are surface characterization techniques that are highly dependent on the condition of the exposed material. Any variations in exposure to ambient conditions, humidity, or even dust particles would alter the chemical signature, and any vibration that occurred during spectra acquisition would cause varying degrees of light scatter from the high surface roughness of the samples. It is possible that the peak intensities are accurate and the samples have a high degree of variability, which would similarly make characterization of the bulk material inconclusive. These data were collected for baseline and through ~4,000 hrs of conditioning. Based on results to that point further efforts were discontinued.

4.5 Dimensional

All the samples being tested were dimensioned to 0.001" and massed to 0.001 g except for the flammability samples. The dimension and mass information was used to calculate the as-received density for all samples currently undergoing conditioning and is presented with the standard deviation in Table 4.

Table 4: Density values (lb/ft^3) for as-received foam samples

Foam ID	Median	Std. Dev.	Sample #
FR-3712 "A"	11.906	0.1145	56
FR-3720 "B"	18.940	0.2055	56
FR-3716 "C/E"	16.370	0.2949	308

The volume and density of the foam were tracked as the sample aged at the given temperatures. The density of the FR-3716 foam is shown in Figure 17, and appears quite stable over the tested lifetimes. The only samples to demonstrate noticeable deformation were aged at 250 °F, an approximation of the physical deformation is depicted in Figure 18. As an approximation the volume calculations were an average between $V = d_x * d_y * d_z$ and $V' = d'_x * d'_y * d'_z$. It should be noted that the samples were selected at random and not all samples exhibited this magnitude or direction of dimensional instability. As the mechanism is unclear under the current aging and sample parameters, additional testing would be required to identify the conditions required for the dimensional changes, determine if air exposure is a factor, and the likely impact on a sealed package.

Tracking of the mass change of the individual samples is given in Figure 19 as calculated: $\Delta m = \frac{m_f}{m_i}$ and shows little change for aging temperatures up to 215 °F and a pronounced mass reduction to 86% for samples aged at 250 °F. The sample volume changes $\Delta V = \frac{V_f}{V_i}$, Figure 20, show a similar trend such that there is little change for aging temperatures up to 215 °F and considerable loss of volume for samples aged at 250 °F to 54% of the initial volume.

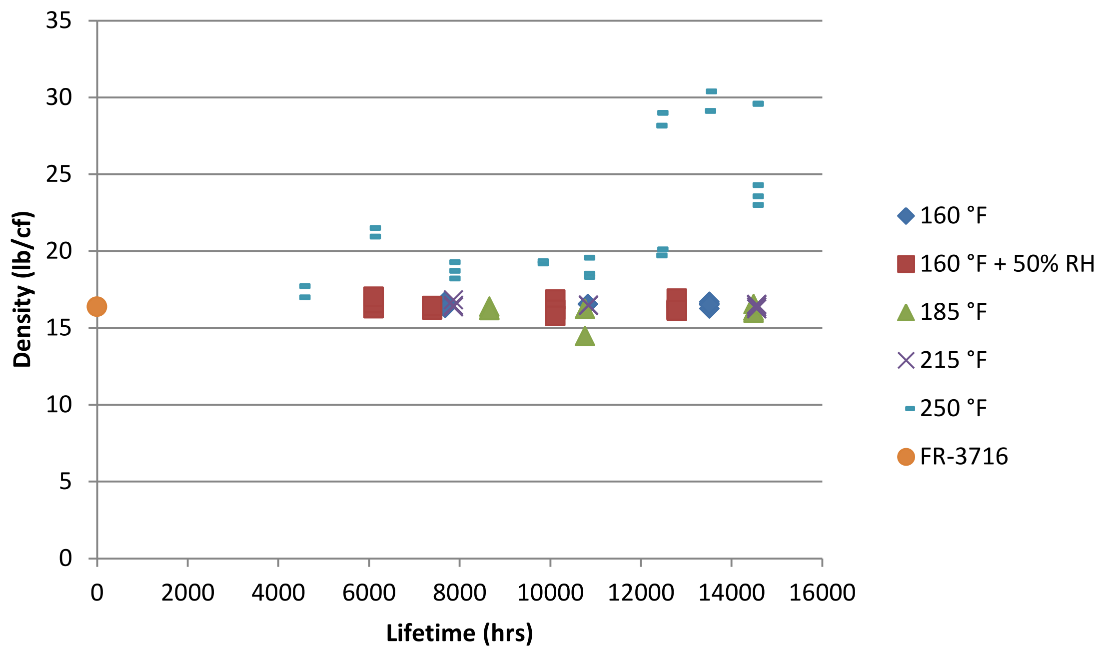

Figure 17: Density of FR-3716 foam as a function of aging temperature and lifetime.

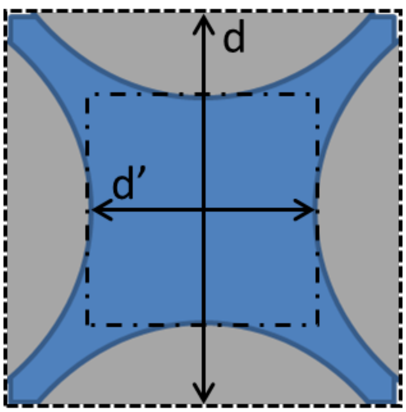

Figure 18: A depiction of the physical deformation seen in the 250 °F samples, where (d) represent the distance between the outer edges, (d') represents the distance between two concave surfaces, initial volume V (----), and final volume V' (– · –).

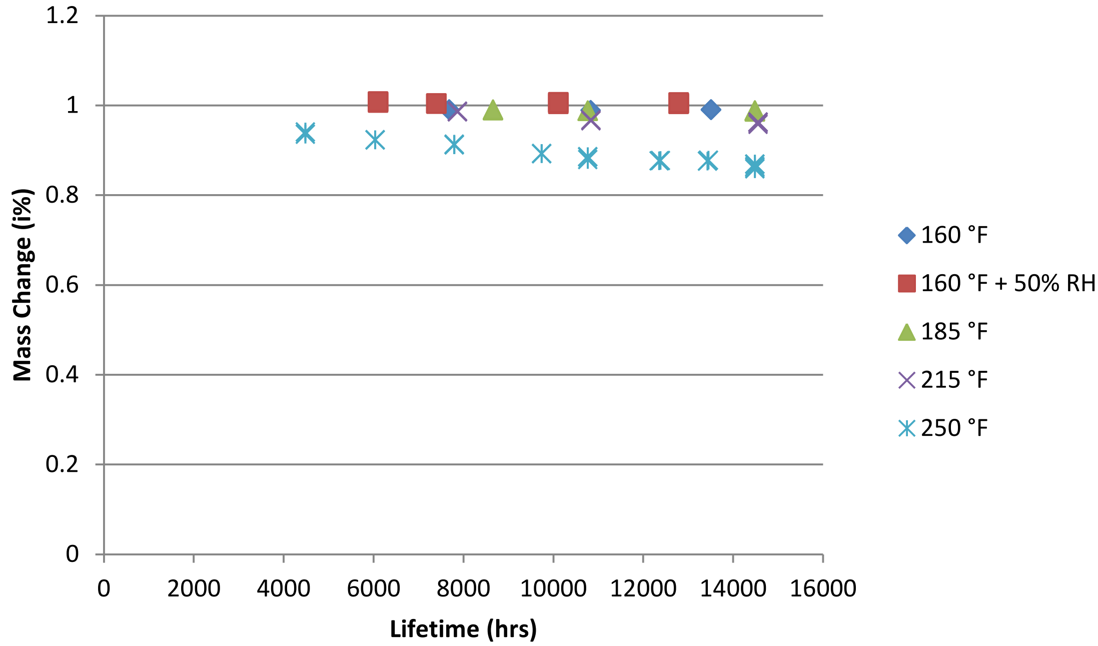

Figure 19: Mass change for samples as measured at the initiation of testing and at final removal.

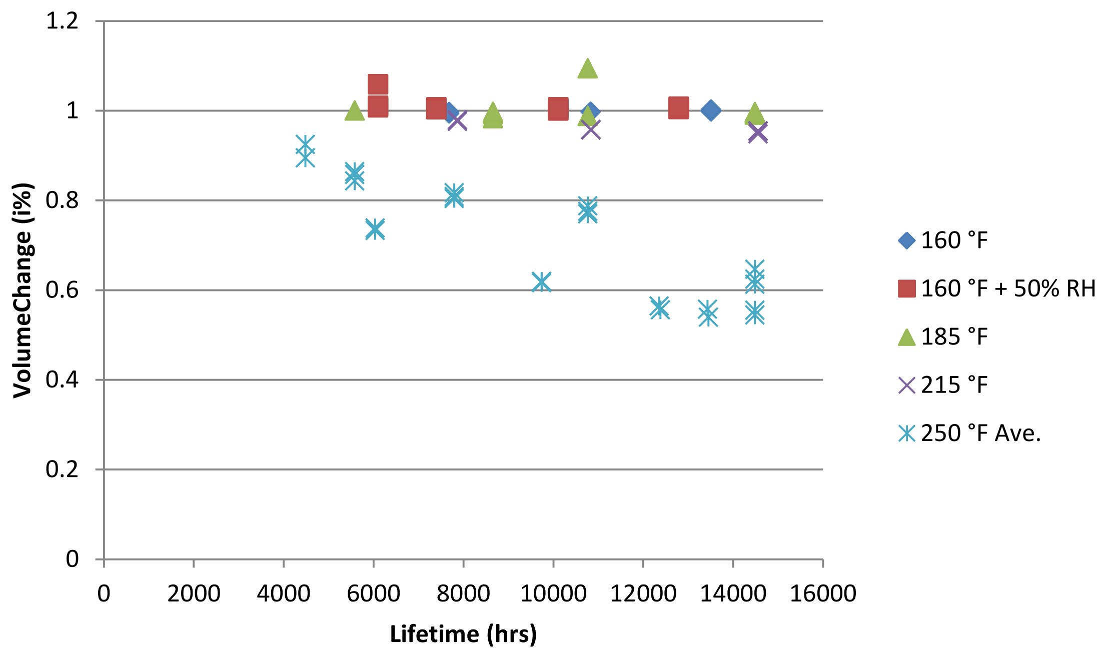

Figure 20: Measured volume of FR-3716 foam as a function of aging temperature and lifetime.

Flammability, intumescence, chloride leaching, and phosphorus leaching measurements were performed at General Plastics Manufacturing Company or subcontracted out after fabrication of the foam, prior to foam shipment to SRNL and are presented in

Table 5. Physical data for aged FR-3716 samples are presented in Table 6. The individual intumescence and burn lengths for FR-3716 are given in Table 7 to show the range of sample-to-sample variation. The intumescence of aged samples does not significantly change for the lower temperatures (160 °F, 160 °F +50% RH, and 185 °F) and then drops significantly at 215 °F and drops to nearly zero at 250 °F. The leached chloride remains unchanged while the phosphorous increases comparably among all aging temperatures. Additional samples are being analyzed to determine if the source of phosphorus is an aging effect or some other cause such as cutting and handling. The burn length remains constant for the lower aging temperatures and nearly doubles for the 250 °F sample. Conditioned samples will be periodically sent back to the manufacturing company to rerun these tests.

Table 5: Initial data from General Plastics Manufacturing Company for as-shipped foam. Flammability was tested per ASTM F-501, phosphorous content was tested per GLI Procedure ME-70, and intumescence, chloride content, and density did not have testing standards listed.

Foam ID	Ave. Burn Length (in)	Ave. Intumescence %	Density (pcf)	Phosphorus (mg/sample)	Chloride Content
FR-3712	2.5	241.8	12.18	1.24	Non-Detect.
FR-3716	2.3	229.5	16.64	1.42	Non-Detect.
FR-3720	2.1	222.4	19.68	1.17	Non-Detect.

Table 6: Physical data for aged FR-3716 samples.

Furnace Temp (°F)	Lifetime (hrs)	Burn Length (in)	Intumescence (%)	Phosphorous (mg/L)	Chloride (mg/L)
Initial	0	2.3	229.5	1.42	non-detect
160	7685	2	218	6.76	<1.0
185	8661	2.1	196.3	7.82	<1.0
215	7870	2.1	67.8	8.2	<1.0
250	7798	4	0.5	9.68	<1.0
160+RH	6101	1.9	231.3	7.18	<1.0

Table 7: Individual values for initial intumescence and burn lengths for each FR-3716 sample, showing the range of sample-to-sample variation.

Initial FR-3716	
Intumescence (%)	Burn Length (in)
255.7	2.2
191.8	2.4
241.1	2.3

5.0 Conclusion and Recommendations

Accelerated aging at elevated temperatures continues for LAST-A-FOAM® FR-3712, FR-3716, and FR-3720. No major changes were observed for the thermal conductivity, heat capacity, compressive properties, and physical properties examined for the lower aging temperatures from 160-185 °F. The exception is the increase in leached phosphorus for all temperatures. The samples aged at 215 °F appear to be showing initial signs of deteriorating properties such as a slight decrease in volume and 70.5% reduction of intumescence. Samples aged at 250 °F show considerable deterioration primarily in the physical characteristics as summarized in Table 8.

Table 8: Property changes of samples aged at 250 °F

Property	**Change**
Volume	-50%
Mass	-15%
Density	+100%
Energy absorbed	-30%-50%
Modulus	Lower than other temperatures, but similar to initial
Yield Stress	Nominal
Thermal conductivity	Unchanged & Terminated
Heat capacity	Nominal
Intumescence	-99.8%

Based on the available data, it is recommended to avoid prolonged exposure of FR-3700 to 250 °F or higher temperatures. It is noted that current storage conditions in KAC should keep foam temperatures within the 9977 package well below 250 °F, although transient conditions could result in short-term exposures approaching this value. Insufficient aging durations have been accumulated to date to determine whether similar extreme changes might eventually be expected at lower temperatures typical of actual storage conditions.

As the furnaces still have an excess inventory of samples, testing is planned to continue beyond the 96 week mark. The additional data will help define the long-term behavior for the lower temperature conditions and will be incorporated into time-temperature-superposition analysis.

Conditioning and testing of foam samples has been initiated for accelerated aging and long-term exposure tests to determine any possible degradation that may occur from expected storage conditions. This testing will continue, as scheduled. Additional suggested testing will be pursued in an attempt to identify the following:

- Onset of physical changes prior to ~7,000 hr mark for the 250 °F condition
- Onset temperature of physical changes between 215 °F and 250 °F
- Impact of anaerobic or depleted oxygen environment on 250 °F condition (since the foam in the 9977 shipping package is effectively isolated from the ambient storage atmosphere)

Projected deliverables remain the issuance of final report upon completion of testing.

6.0 References

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