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Thermal Aging of the Polyurethane Foam for the H1616 Shipping Container

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Abstract

A polyurethane foam used in the H1616 shipping container provides impact energy absorption and fire protection in hypothetical accident conditions. This study was undertaken to determine the estimated lifetime of the foam as a function of temperature. The foams were aged at temperatures ranging from 65°C to 95°C for periods of time ranging from 6 months to 6 years. Both destructive and nondestructive Dynamic Mechanical Analyses (DMA) were used to evaluate the performance of the foams as a function of time and temperature. In addition, color changes and weight loss were recorded. Three properties of the foam show a definite trend with aging time: weight loss, nondestructive G' (measured at 100°C), and glassy G'. A time temperature superposition analysis shows a reasonable trend with temperature for both the weight loss and glassy G'. The acceleration factors for weight loss and glassy G' did not correlate with each other, however. A prediction of the behavior of G' as a function of aging time at 25°C was derived from an extrapolated value of the acceleration factor. In addition to providing a quantitative estimation of the aging process, the curve also provides a description of the qualitative features of the aging process. First, the aging process appears to proceed smoothly as a function of aging time. There are no discontinuities or sharp breaks in the glassy G' as a function of aging time at any of the temperatures. Second, the rate of change of the glassy G' appears to decrease as the aging time increases.

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Nomenclature and Abbreviations

DMA =	dynamic mechanical analysis
pcf	pounds per cubic foot
T_g	glass transition temperature
G’	shear modulus
psi	pounds per square inch
a_T	acceleration factor

1. Introduction

A polyurethane foam used in the H1616 shipping container provides impact energy absorption and fire protection in hypothetical accident conditions. The H1616 shipping container is a Type B shipping container for Limited Life Components. This study was undertaken to determine the estimated lifetime of the foam as a function of temperature. The primary degradation mechanism of these foams is believed to be oxidation leading to stiffening of the foam. The foams were aged at temperatures ranging from 65°C to 95°C for periods of time ranging from 6 months to 6 years. Both destructive and nondestructive Dynamic Mechanical Analyses (DMA) were used to evaluate the performance of the foams as a function of time and temperature. In addition, color changes and weight loss were recorded. Three properties of the foam show a definite trend with aging time: weight loss, nondestructive shear modulus (measured at 100°C), and glassy shear modulus. Time-temperature analyses of weight loss and glassy shear modulus data were used to calculate acceleration factors. These factors were extrapolated to ambient temperatures using the Arrhenius relationship in order to provide an estimation of the foam's useful life.

2. Experimental

Materials. The foam is a rigid fire retardant polyurethane with a free rise density of 12 pcf and an installed density of 16.5 pcf. The foam was cut into pieces nominally 0.25" thick, 0.50" wide, and 2.25" long and placed in calibrated air circulating ovens at temperatures of 65, 80 and 95°C. Additional samples were stored in the laboratory at an average temperature of 22°C. Each sample was weighed before and shortly after removal from the oven.

Dynamic Mechanical Analysis. The thermal-mechanical properties of the rigid polyurethane foam material used in the H1616 shipping container were evaluated for changes induced by several isothermal aging temperatures. Using dynamic mechanical analysis (DMA), a *TA Instruments ARES* rheometer with a torsional rectangular test geometry operating at 1 Hz and a temperature ramp rate of 3°C/min was used to run these tests. From these tests, that measure shear modulus as a function of temperature, the glassy and rubbery modulus plus glass transition temperature (T_g) were determined. The scheme for determining the values is presented in Figure 1.

Two groups of specimens were tested for this study. The first group, the destructive test specimens, contains specimens that were tested to temperatures greater than 200°C for the purpose of obtaining T_g and the glassy and rubbery shear modulus. There were a different set of five samples tested for each aging time. The second group, non-destructive test specimens, was tested to a maximum test temperature of 100°C. This set of five samples was aged at 100°C pulled out for testing each year and then placed back in the 100°C oven for further aging.

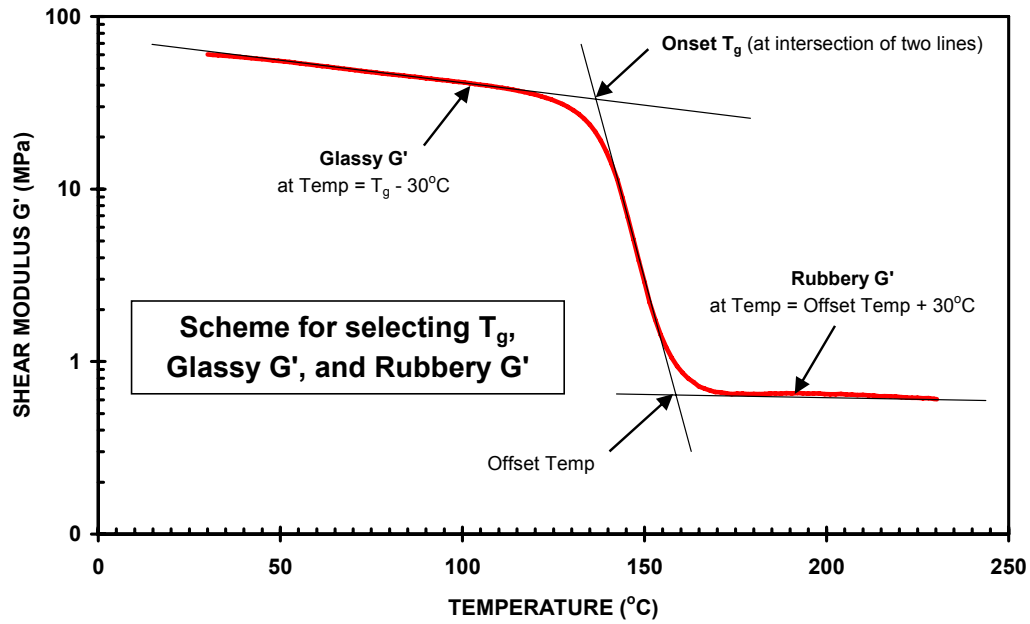

Figure 1. Representative curve from a torsional DMA test illustrating the procedure used to determine values of glassy and rubbery shear modulus G', and the glass transition temperature Tg.

3. Results and Discussion

We will first present the results for the four types of measurements that were performed on the samples:

1. observation of color changes
2. changes in sample weight
3. nondestructive DMA measurements to 100°C
4. destructive DMA measurements to 230°C

The changes in sample weight and the glassy shear modulus, G', show a consistent dependence on aging time and temperature. We performed a time/temperature superposition of these two quantities to derive an Arrhenius plot to display the aging rate vs. inverse temperature. An extrapolation of the aging rate of the glassy G' to ambient temperatures enabled us to make a prediction of the foam's properties vs. aging time.

Color Changes. Figure 2 shows a picture of the foam samples that are unaged and have been aged for 6, 18, 30, 42 and 72 months at 65, 80 and 95°C. Although the samples darken with aging time and temperature, color is probably not a useful indicator of a sample's condition. We also found that the samples continue to discolor for a period of time after removal from the oven. Because it would be difficult to quantify these color changes and because these changes may depend on the post aging storage conditions, we decided not to use the color changes as a predictive tool.

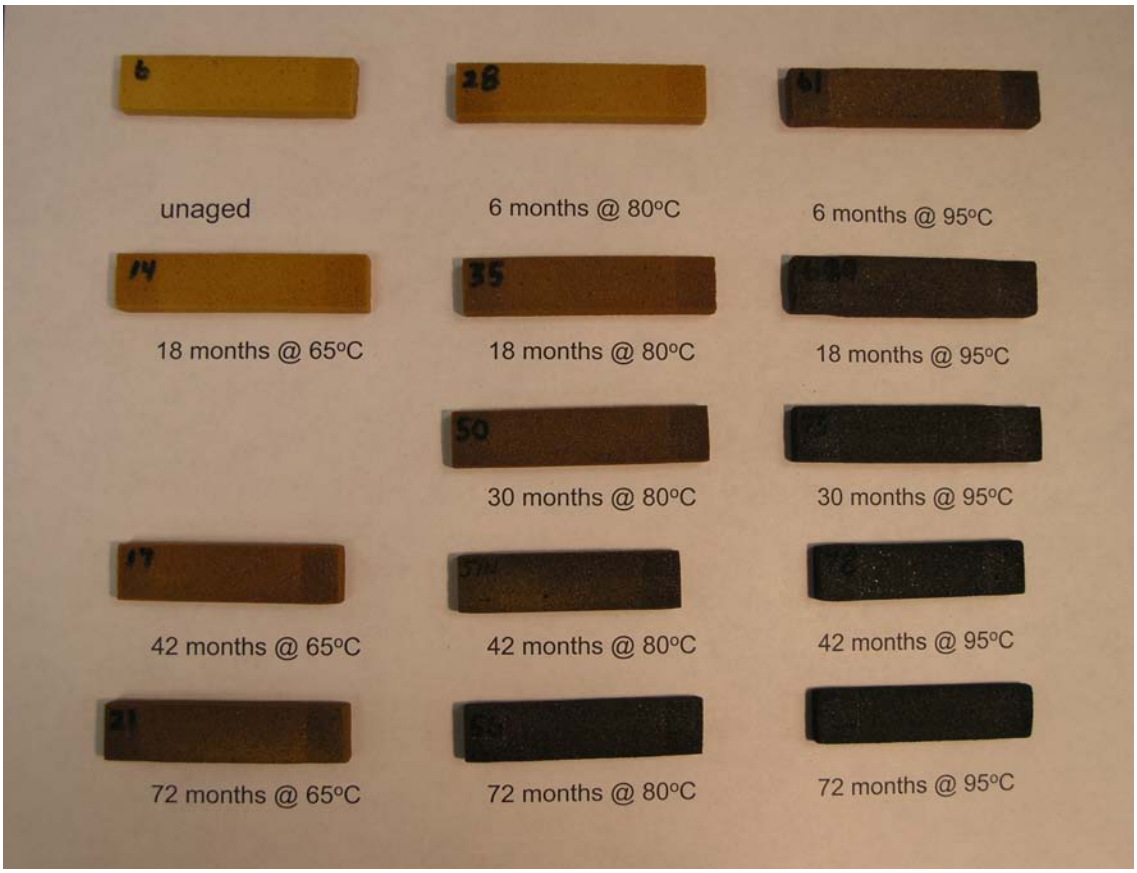

Figure 2. Substantial color changes as a function of aging time and temperature were observed for the foam samples.

Sample Weight. The sample weights as a function of aging time and temperature are shown in Table 1. The measurements at 6 and 18 months may have been complicated by the slight effect of the laboratory humidity on the sample weights. The high surface area foams evidently can adsorb several tenths of a percent weight water depending on the relative humidity of its surroundings. These complications were largely eliminated for the samples aged at 42 and 72 months by weighing the samples within 10 to 15 minutes after they were removed from the aging ovens. Weight measurements performed within this time window were shown to be consistent with the results for foams that were weighed under very low humidity conditions. Figure 3 shows the relative weights as a function of time and temperature in graphic form. The equations in the figure show a least squares fit of the weight data and will be used later to discuss the rate vs. temperature of the weight loss process.

Table 1. A summary of the foam weight loss data.

Time (months)	65°C	80°C	95°C
0	1.000	1.000	1.000
6		1.001	0.998
18		.986	0.970
32	0.996	.990	0.962
42	0.992	.982	0.943
72	0.991	.974	0.913

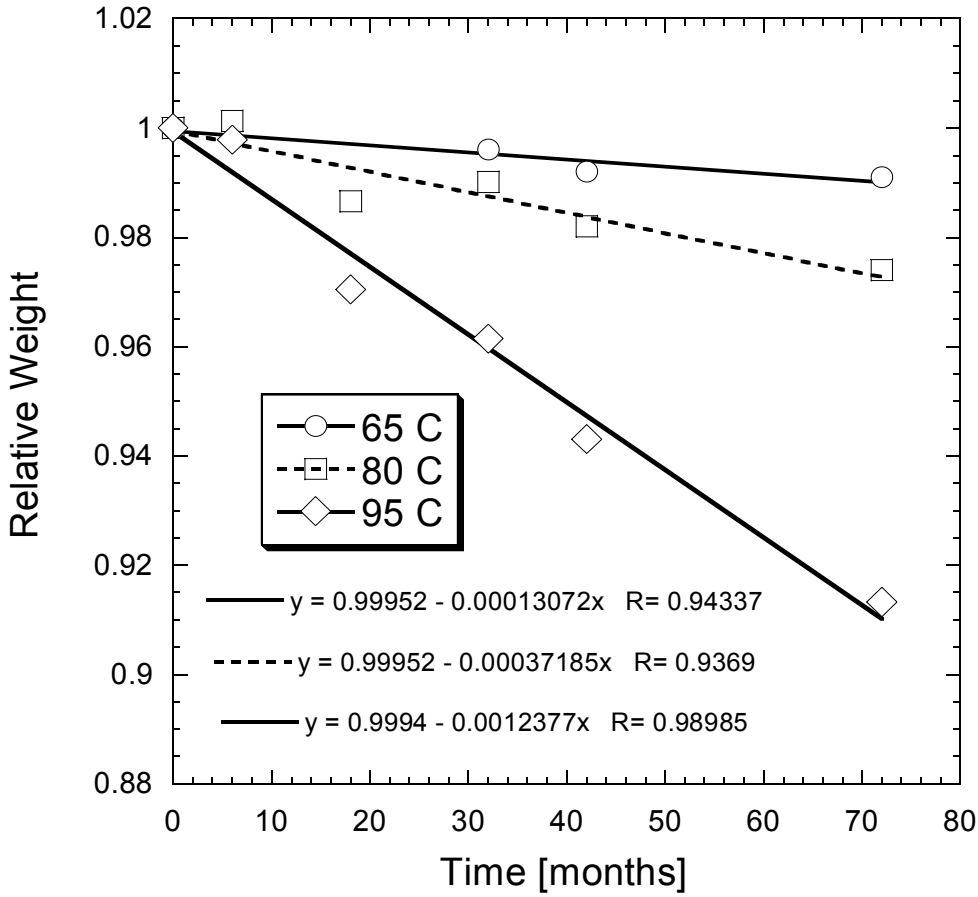

Figure 3. Relative weight of the foam samples as a function of aging time and temperature. The equations display a least squares fit of the data.

Nondestructive DMA Testing. Table 2 shows the results of the nondestructive DMA measurements of the shear moduli, G', conducted at 100°C for samples aged for 6, 18, 30, 42 and 72 months at 95°C. A plot of the change versus aging time at 95°C is shown in Figure 4. The line is a hand-drawn fit of the data. The shear moduli display a general increase with aging time.

Table 2. Nondestructive measurements of the shear moduli, G', conducted at 100°C for five samples aged at 95°C for the times indicated.

Specimen Number	Density (lb/ft^3)	G' at 100°C					
		Zero Time (psi)	6 Months (psi)	18 Months (psi)	30 Months (psi)	42 Months* (psi)	72 Months* (psi)
86	13.96	6015	7777	7670	9033	9047	10442
87	14.48	6490	8287	8786	10457	9599	11339
88	18.22	6665	8508	8854	10604	9260	11497
89	14.09	5845	7431	7641	9005	8120	9881
90	14.08	6125	7794	8096	9129	8725	10413
	Avg =	***6228***	***7960***	***8209***	***9646***	***8950***	***10714***
	Stdev =	***340.0***	***432.5***	***586.1***	***810.9***	***562.6***	***682.3***

*Tests were run on a new instrument (***TA ARES*** DMA)

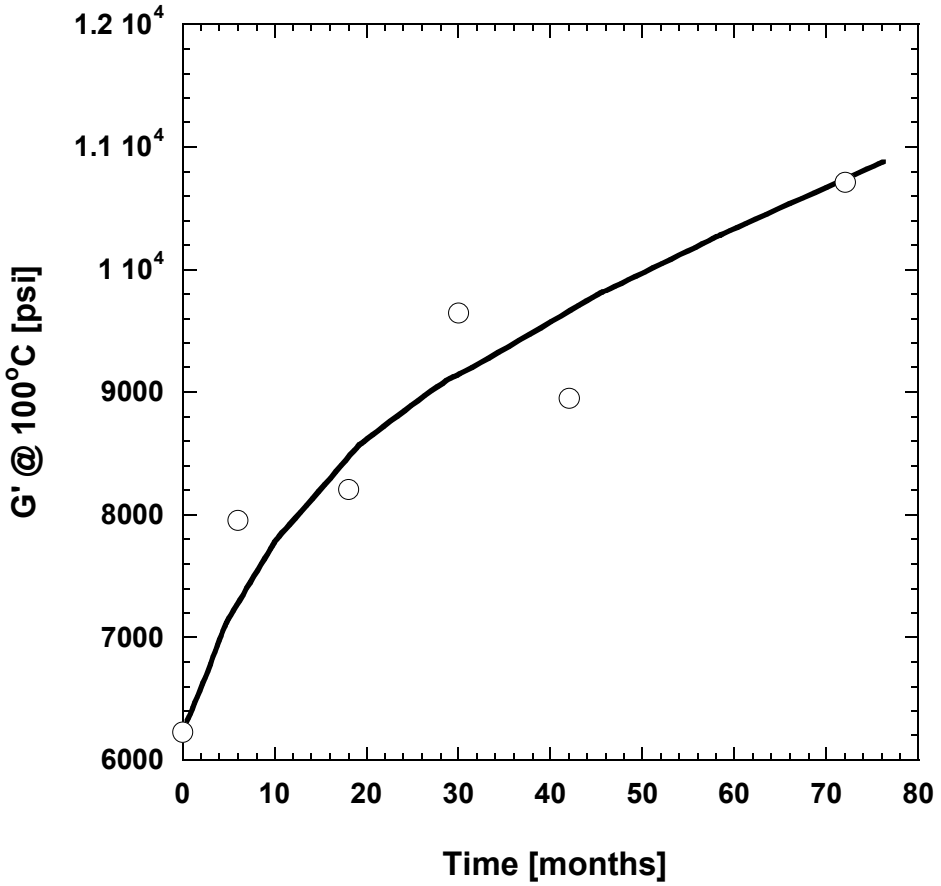

Figure 4. Increase in the shear moduli, G', measured at 100°C for samples stored at 95°C for the times indicated.

Destructive DMA Testing. The destructive DMA testing was performed on samples that were aged as a function of temperature as well as time. Since these data will be used to calculated predicted lifetimes at ambient temperatures, a more complete record of the results will be presented. Tables 3 through 6 show the glassy and rubbery shear moduli and the glass transition temperature for the five samples that were analyzed at each aging temperature and time. Table 7 shows a summary of the destructive DMA testing data.

Table 3. The glassy and rubbery shear moduli and the glass transition temperature for samples aged at 20°C.

Specimen Number	Aging Temperature (C)	Aging Time (Months)	Density (lb/ft3)	Shear Modulus G' Glassy (psi)	Shear Modulus G' Rubbery (psi)	Glass Transition Temperature, Tg (C)
45	20C	0	14.75	6197	95	137.2
46	20C	0	14.00	5427	92	143.9
47	20C	0	14.62	6402	99	136.6
48	20C	0	14.08	5407	93	148.0
49	20C	0	14.04	5504	94	146.6
			Avg =	*5788*	*94*	*142.5*
			Stdev =	*474.3*	*2.8*	*5.3*
1N	20C	18	15.85	5443	90	144.3
2	20C	18	14.19	5604	97	148.7
3N	20C	18	18.04	5320	89	144.2
4	20C	18	14.06	5427	96	149.2
5	20C	18	13.91	5810	98	148.7
			Avg =	*5521*	*94*	*147.0*
			Stdev =	*190.9*	*4.2*	*2.5*
6	20C	72	14.85	7004	119	138.5
7	20C	72	14.09	5786	95	136.6
8N	20C	72	14.35	5728	95	138.3
9	20C	72	14.20	5554	95	139.3
10	20C	72	13.87	5307	90	140.4
			Avg =	*5875*	*99*	*138.6*
			Stdev =	*657.5*	*11.6*	*1.4*

Table 4. The glassy and rubbery shear moduli and the glass transition temperature for samples aged at 65°C.

Specimen Number	Aging Temperature (C)	Aging Time (Months)	Density (lb/ft3)	Shear Modulus G' Glassy (psi)	Shear Modulus G' Rubbery (psi)	Glass Transition Temperature, Tg (C)
11	65C	18	14.21	6141	95	147.5
12N	65C	18	18.92	6273	92	143.6
13	65C	18	14.05	6071	94	147.3
14	65C	18	14.20	6499	95	144.1
15	65C	18	14.24	6163	94	145.8
			Avg =	*6229*	*94*	*145.7*
			Stdev =	*167.2*	*1.3*	*1.8*
16	65C	42	14.38	6750	91	134.5
17	65C	42	14.76	6695	91	135.7
18	65C	42	14.30	7001	94	134.2
19	65C	42	14.66	6845	94	136.4
20	65C	42	15.29	6651	90	135.3
			Avg =	*6788*	*92*	*135.2*
			Stdev =	*139.1*	*1.8*	*0.9*
21	65C	72	14.68	8178	91	131.3
22	65C	72	14.60	7381	89	134.3
23	65C	72	14.81	8106	100	134.8
24	65C	72	14.51	7758	95	135.4
25	65C	72	14.14	7569	91	135.2
			Avg =	*7798*	*93*	*134.2*
			Stdev =	*341.8*	*4.2*	*1.7*

Table 5. The glassy and rubbery shear moduli and the glass transition temperature for samples aged at 80°C.

Specimen Number	Aging Temperature (C)	Aging Time (Months)	Density (lb/ft3)	Shear Modulus G' Glassy (psi)	Shear Modulus G' Rubbery (psi)	Glass Transition Temperature, Tg (C)
26	80C	6	14.36	6506	95	144.3
27	80C	6	14.27	6296	93	144.6
28	80C	6	14.34	6789	94	141.8
29	80C	6	14.52	6686	104	147.6
30	80C	6	14.03	6484	96	146.6
			Avg =	*6552*	*96*	*145.0*
			Stdev =	*191.3*	*4.7*	*2.2*
31	80C	18	14.43	7053	92	144.9
32N	80C	18	16.31	6767	88	144.2
33	80C	18	14.08	6911	90	143.3
34	80C	18	14.05	6785	89	144.9
35	80C	18	14.12	6714	91	145.7
			Avg =	*6846*	*90*	*144.6*
			Stdev =	*136.5*	*1.8*	*0.9*
36	80C	42	14.72	7689	89	142.0
37	80C	42	14.04	7144	89	143.8
38	80C	42	14.80	7539	99	145.4
39	80C	42	13.91	6551	84	145.7
50	80C	42	14.17	6747	89	146.4
			Avg =	*7134*	*90*	*144.7*
			Stdev =	*490.4*	*5.5*	*1.8*
51N	80C	42	16.44	7534	86	134.8
52	80C	42	14.75	8591	92	131.8
53N	80C	42	15.19	8388	98	134.1
54	80C	42	14.50	8013	97	135.7
55	80C	42	14.33	7275	85	136.1
			Avg =	*7960*	*92*	*134.5*
			Stdev =	*555.8*	*6.0*	*1.7*
56	80C	72	14.18	8512	91	136.4
57	80C	72	14.01	8280	89	135.7
58	80C	72	14.58	8512	87	135.4
59N	80C	72	14.44	8207	85	135.7
60N	80C	72	14.90	8889	88	133.6
			Avg =	*8480*	*88*	*135.4*
			Stdev =	*266.3*	*2.1*	*1.1*

Table 6. The glassy and rubbery shear moduli and the glass transition temperature for samples aged at 95°C.

Specimen Number	Aging Temperature (C)	Aging Time (Months)	Density (lb/ft3)	Shear Modulus G' Glassy (psi)	Shear Modulus G' Rubbery (psi)	Glass Transition Temperature, Tg (C)
61	95C	6	14.71	7465	88	142.1
62	95C	6	14.21	6467	88	145.2
63	95C	6	14.21	6180	89	146.0
64	95C	6	14.28	7128	92	145.3
65	95C	6	13.93	6702	89	149.2
			Avg =	*6788*	*89*	*145.6*
			Stdev =	*513.3*	*1.3*	*2.5*
66	95C	18	14.43	7874	82	143.3
67	95C	18	14.13	7555	83	145.4
68	95C	18	14.12	6870	81	146.0
69	95C	18	14.53	7990	86	144.5
70	95C	18	14.17	7700	85	145.3
			Avg =	*7597*	*83*	*144.9*
			Stdev =	*439.1*	*2.3*	*1.0*
71	95C	30	13.90	7450	85	148.2
72	95C	30	14.38	8023	88	146.5
73	95C	30	14.21	8166	92	147.3
74	95C	30	14.00	7630	85	146.5
75	95C	30	14.35	8121	96	146.5
			Avg =	*7878*	*89*	*147.0*
			Stdev =	*319.4*	*4.7*	*0.8*
76	95C	42	14.28	8886	96	136.9
77	95C	42	14.18	9058	102	138.9
78	95C	42	13.85	8426	98	139.7
79	95C	42	14.54	8736	92	138.5
80	95C	42	14.09	8433	99	140.3
			Avg =	*8708*	*98*	*138.9*
			Stdev =	*278.4*	*3.5*	*1.3*
81	95C	72	14.07	9135	199	147.7
82N	95C	72	14.43	9208	194	144.0
83	95C	72	14.21	9686	184	144.8
84	95C	72	13.90	9237	170	147.1
85	95C	72	14.35	9759	181	143.9
			Avg =	*9405*	*186*	*145.5*
			Stdev =	*293.4*	*11.4*	*1.8*

Table 7. A summary of the glassy and rubbery shear moduli and the glass transition temperature for samples aged at temperatures ranging from 20 to 95°C.

Aging Temperature (C)	Aging Time (Months)	Shear Modulus G'		Glass Transition Temperature, Tg (C)
		Glassy (psi)	Rubbery (psi)	
20	0.0	5788	94	142.5
20	18	5521	94	147.0
20	72	5875	99	138.6
65	18	6229	94	145.7
65	42	6788	92	135.2
65	72	7798	93	134.2
80	6	6552	96	145.0
80	18	6846	90	144.6
80	30	7134	90	144.7
80	42	7960	92	134.5
80	72	8480	88	135.4
95	6	6788	89	145.6
95	18	7597	83	144.9
95	30	7878	89	147.0
95	42	8708	98	138.9
95	72	9405	186	145.5

In order to visualize the data and quantify trends, the glassy shear modulus, the rubbery shear modulus and the glass transition temperature are shown as a function of aging time in Figures 5, 6 and 7, respectively. The glassy shear moduli, Figure 5, of the samples aged at 65, 80 and 95°C show an increase with aging time. The rubbery shear moduli, Figure 6, exhibit significant fluctuation as a function of aging time and long-term trends are not apparent. The glass transition temperatures, Figure 7, also show significant fluctuation as a function of aging time and do not provide a useful measure of degradation for these aging times and temperatures. The 42 and 72-month aged DMA measurements were conducted on a new instrument compared to previous measurements. Calibration experiments showed that the glass transition temperatures measured by the new instrument were consistently 6°C lower than those measured by the previous instrument. Thus, the 42 and 72 month glass transition temperatures shown in Table 7 were adjusted upwards by 6°C when plotted in Figure 7.

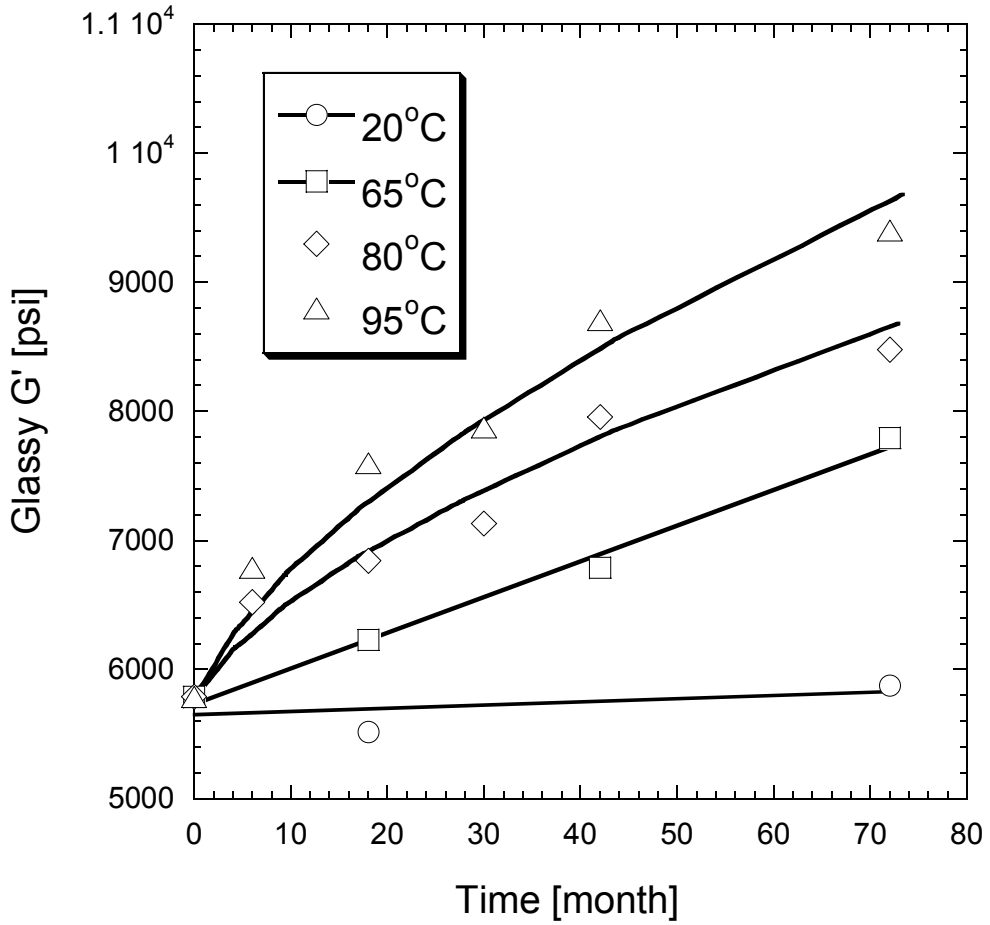

Figure 5. The dependence of the Glassy Shear Modulus, G', as a function of aging time at 20, 65, 80 and 95 °C.

The glassy shear modulus of the foam samples, shown in Figure 5, displays an increase with aging time, especially as the aging temperature is increased. The data at 20 and 65°C were fit with a straight line while the 80 and 95°C data were fit with a freehand curve. The rubbery shear modulus, shown in Figure 6, displays random scatter except for the sample aged at 95°C for 72 months. We are uncertain if this effect is real or an artifact. We will not analyze this data further since clear trends are not observed. We believe that the glass transition temperature data, shown in Figure 7, also display random scatter. While the 20 and 95°C data show a modest increase with aging time, the 65 and 80°C data show a modest decrease with aging time. In the next section we will perform a time temperature superposition of the glassy shear modulus data in order to derive lifetime predictions for this material. Fortunately this measurement, the foam stiffness below its glass transition temperature, probably best reflects the performance of the material as it is used to absorb impact energy in a shipping container.

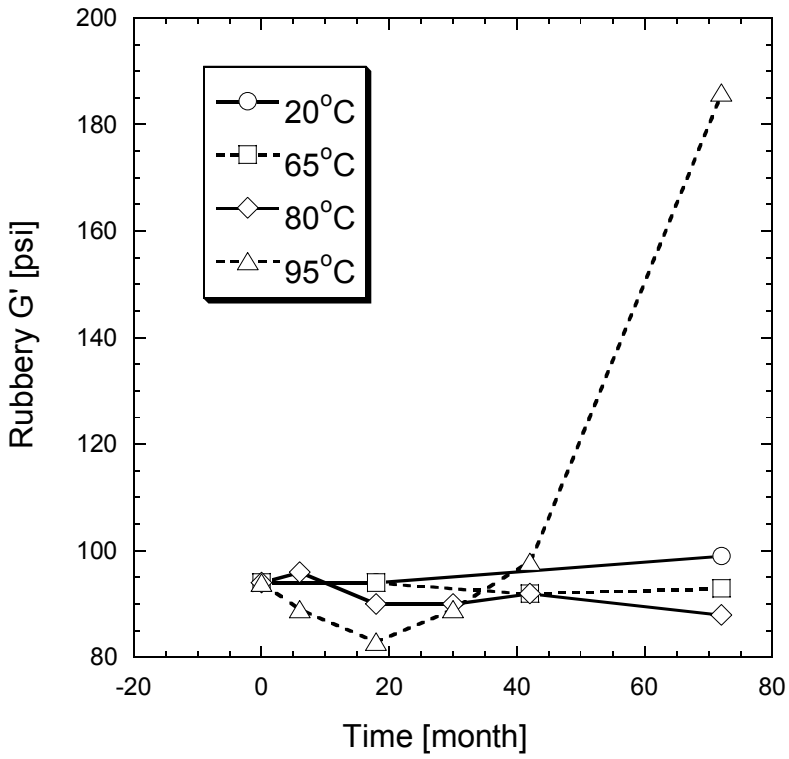

Figure 6. The dependence of the rubbery shear modulus, G', as a function of aging time at 20, 65, 80 and 95 °C.

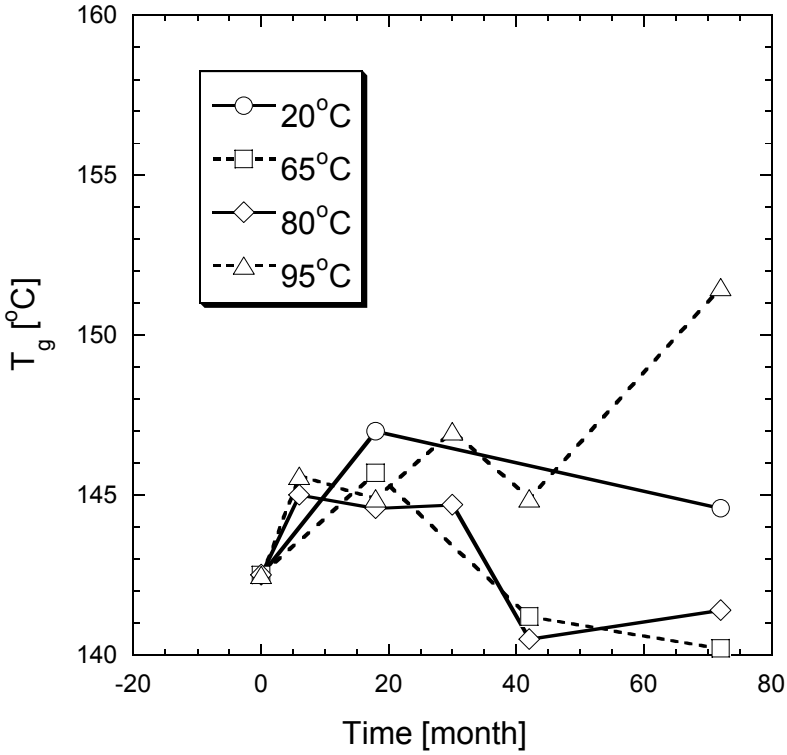

Figure 7. The dependence of the glass transition temperature, T_g, as a function of aging time at 20, 65, 80 and 95 °C.

Time-Temperature Analysis. The intent of these experiments is to predict the useful life of the foam materials. We use accelerated aging conditions (high temperatures) to measure property degradation in a short period of time and then extrapolate the results to the service temperature. The data are first subjected to a time-temperature superposition in order to derive acceleration factors for each temperature. The acceleration factor is the rate, relative to a reference temperature, by which the degradation process proceeds at each temperature. The acceleration factors are then plotted on an Arrhenius graph [1] and extrapolated to use conditions in order to predict the degradation rate under those conditions. The uncertainty of the extrapolation process must be balanced against the length of time for which the laboratory experiments can be conducted.

Three of the measurements have shown a significant change with aging: weight loss, nondestructive G' and glassy G'. The nondestructive G' was performed on samples aged at a single temperature so they cannot be used for the extrapolation. They do confirm that the G' is increasing significantly at 95°C. The weight loss data in Figure 3 were analyzed by a linear fit that was forced through 1.0 at time zero. The slope of the line represents the aging rate. If the aging rate at 95 °C is assigned a value of 1.0, then the rate at 80°C is 0.305 and the rate at 65°C is 0.112.

The glassy G' measurements show curvature as a function of time. Rather than attempting to derive a fitting function, we used time-temperature superposition [1] to compare the aging rates at the two temperatures. Again, the aging rate at 95 °C is assigned a value of 1.0. The time axes of the 65 and 80°C data are then adjusted so that the three aging curves superimpose. This analysis is shown in Figure 8. The limited number of data points for the three temperatures appear to follow the same hand-drawn curve. The acceleration factors for the 80 and 65°C data are 0.50 and 0.28.

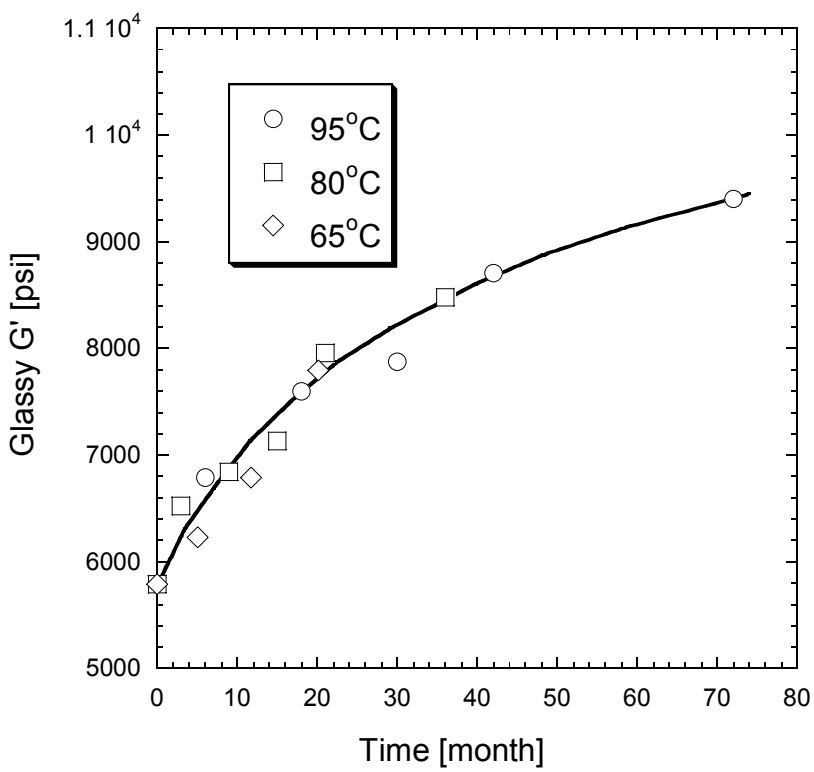

Figure 8. The time temperature superposition analysis for the glassy shear modulus of the foam for the H1616 shipping container.

Figure 9 shows an Arrhenius plot of the acceleration factors for both weight loss and glassy shear modulus. The solid lines are extensions of a linear fit of the data. The acceleration factors for the weight loss and glassy shear modulus diverge widely as the temperature is lowered (larger values of $1000/K^{-1}$). This means that the predicted rates for the two quantities will be quite different. Often times the mechanical properties of a material can be related to specific chemical processes [2,3]. In these instances, the acceleration factors have the same temperature dependence and either mechanical or chemical properties can be used to perform lifetime predictions. The weight loss (a chemical process) and glassy shear modulus (a mechanical property) appeared to track each other during the early stages of the aging study short term aging study. If this relationship could be established, it could have offered alternative methods to characterize the aging process. The recently acquired 72 month data, however, provided a more severe test of this relationship and it appears that the correlation breaks down. For the remainder of the study we will focus our attention of the glassy shear modulus. It is the property most closely associated with the performance of the foam. In addition, the process has a lower activation energy so any lifetime predictions based on the glassy shear modulus will be more conservation than those based on the temperature dependence of the weight loss.

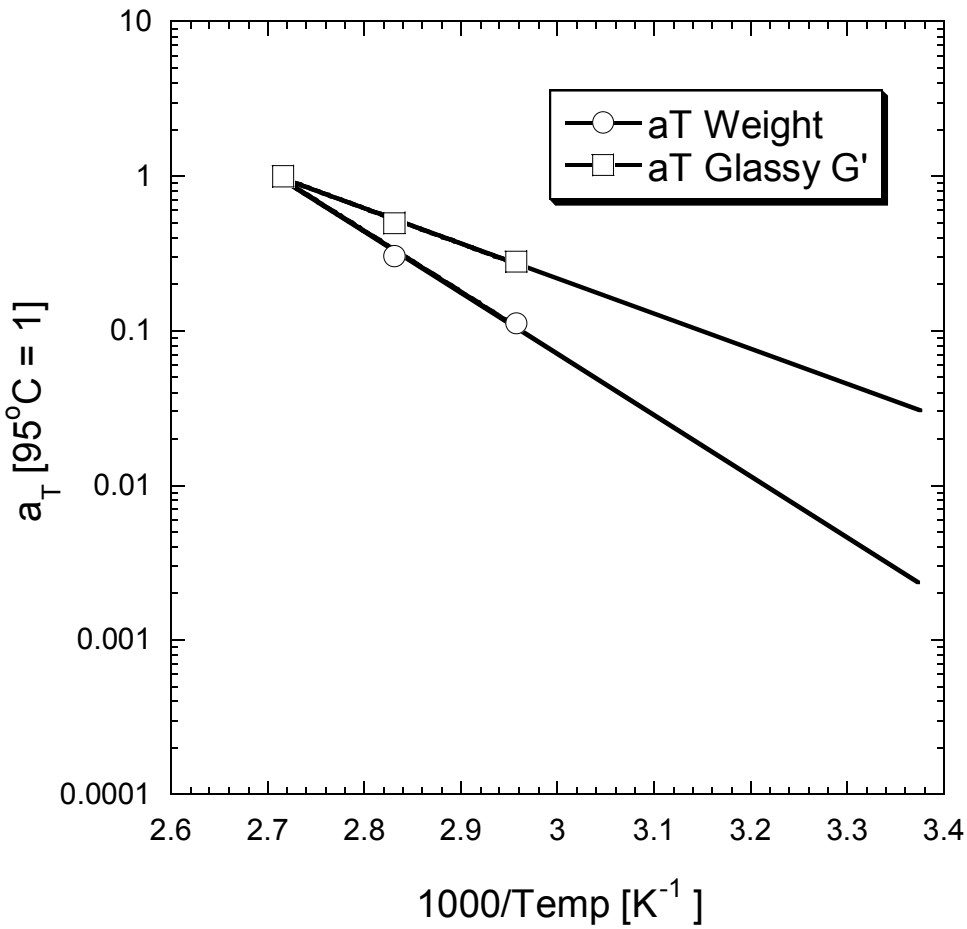

Figure 9. The Arrhenius plots of the acceleration factors for weight loss and the glassy shear modulus.

The acceleration factor for the glassy shear modulus at 25°C obtained by extrapolation is 0.034. This means that the predicted rate of degradation at 25°C is a factor of 1/0.034 or 29.4 times slower than that observed at 95°C. An alternative method of thinking about the data is to plot the change of the glassy shear modulus at 95°C as a function of time and multiplying the time axis by a factor of 29.4. Such a plot is shown in Figure 10 and predicts how the glassy shear modulus behaves as a function of time at 25°C.

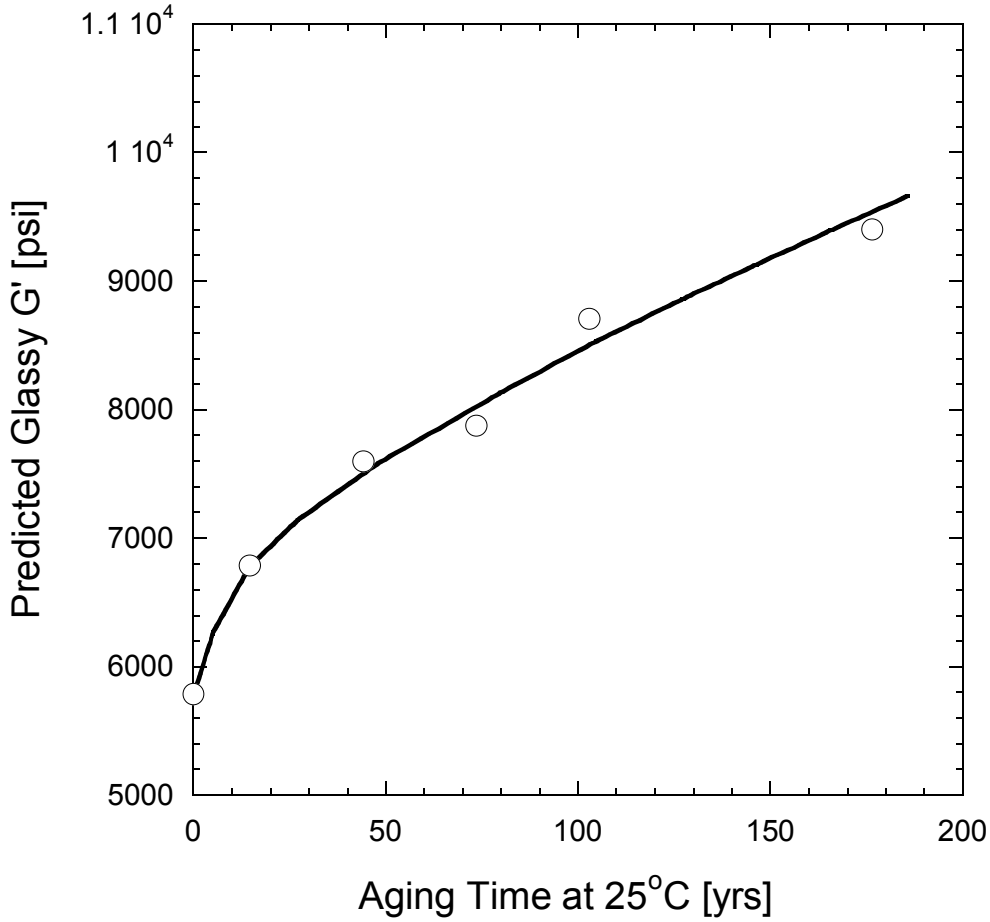

Figure 10. The predicted behavior of the glassy shear modulus vs. aging time of the foam at 25°C. The model is based on an extrapolated acceleration factor from an Arrhenius plot.

This prediction obviously has considerable uncertainty. This uncertainty arises from several factors:

1. The acceleration factors are not determined with sufficient accuracy.
2. The extrapolation range from 65°C to 25°C is quite large.
3. The extrapolation may not be linear.

The first two factors could introduce a random error into the analysis, while the third depends on whether the acceleration factors follow a linear pattern, curve upward or curve downward. The most common trend is for the acceleration factors to curve upward [4] meaning that the predictions underestimate the rate of aging at 25°C. Arguing against this trend, however, is the very low activation energy of 10.5 kcal/mole calculated from the slope of the acceleration factors for the glassy shear modulus shown in Figure 9. This is in the lower range of activation energies observed for chemical processes so we expect that any upward curvature would be relatively mild.

4. Conclusions

Three properties of the foam show a definite trend with aging time: weight loss, nondestructive G' (measured at 100°C), and glassy G'. A time temperature superposition analysis shows a reasonable trend with temperature for both the weight loss and glassy G'. The acceleration factors for weight loss and glassy G' did not correlate with each other, however. This finding suggests that the weight loss of the foams is not closely related to the chemical processes responsible for the increase in glassy G'. A prediction of the behavior of G' as a function of aging time at 25°C was derived from an extrapolated value of the acceleration factor. In addition to providing a quantitative estimation of the aging process, the curve also provides a description of the qualitative features of the aging process. First, the aging process appears to proceed smoothly as a function of aging time. There are no discontinuities or sharp breaks in the glassy G' as a function of aging time at any of the temperatures. Second, the rate of change of the glassy G' appears to decrease as the aging time increases. For example, if we follow the smooth curve drawn through the data points in Figure 10, we predict that the glassy G' would increase from 5800 psi at time 0 to 6950 psi after 20 years to 7400 psi after 40 years. The smooth, downward curving trend implies that the glassy G' will not exhibit any abrupt changes and that the rate of change will decrease with aging time.

5. References

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