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9975 Shipping Package – Performance of Alternate Materials for Long-Term Storage Application

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ABSTRACT

The Model 9975 shipping package specifies the materials of construction for its various components. With the loss of availability of material for two components (cane fiberboard overpack and Viton® GLT O-rings), alternate materials of construction were identified and approved for use for transport (softwood fiberboard and Viton® GLT-S O-rings). As these shipping packages are part of a long-term storage configuration at the Savannah River Site, additional testing is in progress to verify satisfactory long-term performance of the alternate materials under storage conditions. The test results to date can be compared to comparable results on the original materials of construction to draw preliminary conclusions on the performance of the replacement materials.

INTRODUCTION

Cane fiberboard wall sheathing is specified for thermal insulation and impact resistance in 9975 shipping packages (Figure 1). Softwood fiberboard manufactured by Knight-Celotex was approved as an acceptable substitute for transportation in 2008. Data in the literature have been summarized [1, 2] and the references cited show a consistent trend in thermal properties of fiberboard as a function of temperature, density and/or moisture content regardless of material source. Thermal and mechanical properties were measured for non-aged softwood fiberboard samples, and found to be sufficiently similar to those of non-aged cane fiberboard to support the acceptance of 9975 packages with softwood fiberboard overpack into the SRS K-Area Materials Storage (KAMS) facility for the long-term storage of plutonium materials.

The containment vessel O-ring compound historically used in the 9975 is Parker Seals V0835-75, based on Viton® GLT fluoroelastomer (DuPont Performance Elastomers). In 2006, DuPont announced that Viton® GLT was being replaced with Viton® GLT-S. The GLT-S polymer was developed in 2001 under the nomenclature of Advanced Polymer Architecture (APA), principally to improve flow processing characteristics. Two GLT-S polymers are currently produced (GLT-200S, GLT-600S). A primary difference between the two GLT-S grades is that the gum (Mooney) viscosity of the 200S grade is much lower (25 vs. 65). Both polymers significantly improve processing characteristics compared to the older GLT polymer with a Mooney Viscosity of ~ 90 [3, 4]. In 2006, Parker Seals formulated a new compound (VM835-75) based on GLT-S polymer and qualified the compound per AMS-R-83485 [5]. Based on review of polymer manufacturer data, O-ring compound qualification data and initial characterization of O-ring properties, the GLT-S based O-rings were approved as alternate seals for transportation and were accepted into the KAMS facility.

Efforts have been underway at the SRS for several years to assess the aging behavior of legacy fiberboard and Viton® GLT O-rings. Therefore, additional testing was needed to validate the continued acceptability of aged softwood fiberboard and GLT-S O-rings to meet KAMS storage requirements. These efforts to date are presented. Henceforth, the O-ring compounds will be designated as GLT and GLT-S for distinction.

Experimental Methods

Softwood Fiberboard

A lower fiberboard subassembly fabricated from softwood fiberboard for use in a 9975 shipping package was obtained from KAMS. Samples were removed from this subassembly for conditioning and testing to track the potential degradation in physical, thermal and mechanical properties. Samples were aged in 4 environments:

- 121 °C oven (nominal humidity of ~1%RH)
- 102 °C oven (nominal humidity of ~1%RH)
- 85 °C oven (nominal humidity of ~2%RH)
- 85 °C, 30%RH environmental chamber

The sample configurations and test methodologies are the same as used for aging and testing cane fiberboard samples [6, 7]. Samples for physical measurements are approximately 5 cm cubes, and receive periodic measurement of weight and dimensions. Two of these samples are conditioning in each of the 4 environments. Samples for compression testing are also approximately 5 cm cubes. These samples were placed in 3 of the environments (121 °C and 85 °C oven, and 85 °C 30%RH chamber). A few of these samples are removed periodically for testing. Since the compression test is destructive, these samples are not returned to the conditioning environment.

Testing for thermal properties includes both thermal conductivity and specific heat capacity. Thermal conductivity samples are approximately 18 x 18 x 3 cm. Two of these samples are conditioned in each of 3 environments (121 °C and 102 °C oven, and 85 °C 30%RH chamber) and tested periodically. In each sample pair, one is oriented for axial heat flow, and the other is oriented for radial heat flow. Thermal conductivity is measured at 3 mean temperatures – 25, 50 and 85 °C.

Specific heat capacity samples are cylindrical, approximately 2.5 cm diameter and 4 cm high. Three of these samples are conditioned in each of 2 environments (121 °C oven and 85 °C 30%RH chamber) and tested periodically. Specific heat capacity is measured at a mean temperature of 50 °C.

Thermal and physical property samples were characterized before conditioning, and separate compression samples were tested without conditioning to document baseline properties. The three oven environments have been maintained on an almost continuous basis, while the environmental chamber has experienced significant down-time. Therefore, the samples in the 85 °C 30%RH environment have not accumulated as much total exposure time as the other samples

GLT-S O-Rings

To accept the GLT-S O-rings as an alternate material for transportation and subsequent placement in KAMS, the nominal properties of GLT-S and GLT O-rings were compared. The majority of data for the GLT-S polymer and test compounds are available from the polymer manufacturer. AMS compound qualification data are available from Parker Seals. SRNL characterized several parameters of the new compound including Durometer hardness, structure and composition via FT-IR spectroscopy, differential scanning calorimetry (DSC), X-ray diffraction (XRD), neutron activation analysis (NAA), Dynamic Mechanical Analysis (DMA), tensile testing and compression stress-relaxation (CSR).

CSR is a measure of the retained sealing force over time as the elastomer relaxes at elevated temperature. CSR behavior of GLT-S O-rings was initially measured for a 1000 hours at 93 °C, 149 °C, 177 °C and 204 °C for direct comparison with GLT O-rings. Longer CSR tests are currently in progress at 79, 93, 121, 149 and 177 °C for both compounds to develop an aging model.

Actual seal performance is also being tracked via periodic helium leak testing of GLT and GLT-S O-rings in test fixtures being aged at temperatures ranging from 93-204 °C. O-ring temperatures in storage may reach temperatures as high as 79-120 °C, depending on payload and ambient conditions. Higher aging temperatures are used to accelerate the time to failure and to bound the storage condition. Leak test results will be reported in a separate paper.

Results

Softwood Fiberboard

The physical property samples are measured on an approximately weekly basis. Weight and density data are shown in Figure 2 on a normalized basis (each datum is divided by its corresponding value after the first conditioning period). This normalization allows for a direct comparison between samples with different starting values. The rates of change in the weight, density and dimensions of these samples are summarized in Table 1, along with comparable data for cane fiberboard samples.

Compression testing is performed with the load applied either parallel or perpendicular to the fiberboard layers. Stress-strain curves for softwood fiberboard samples are shown in Figures 3 and 4 for these two orientations, respectively. Because of variation in the shape of the stress-strain curve from one sample to another, two metrics have been used to provide a comparison of compression test performance. For samples of both orientations, the area under the stress-strain curve up to a strain of 40% provides a metric that is roughly proportional to the energy absorbed by the material. In addition, samples tested in the parallel orientation experience an initial stress peak as the layers start to buckle. This buckling strength provides a second metric for comparison of the parallel orientation samples. These metrics are summarized in Tables 2 and 3, along with data for cane fiberboard samples.

Thermal conductivity data for each sample are presented in Figure 5. Similar trends are seen for each of the three test temperatures – 25, 50 and 85 °C. Since the baseline thermal conductivity varies for each sample, normalized data are shown in Figure 6, and show the relative change from the first data point after 8 weeks conditioning. For comparison, comparable normalized data for cane fiberboard samples are also shown in Figure 6.

Specific heat capacity results are summarized in Figure 7. Due to the degree of scatter in individual results, results from each trial for all 3 samples in a given environment are averaged for each conditioning period. Comparable data for cane fiberboard samples are shown in Figure 7.

GLT-S O-Rings

The GLT-S O-rings are reportedly molded on the same tooling as the GLT O-rings. Dimensions were verified to be consistent. Durometer hardness is often used in O-ring and gasket specifications as a matter of quality control, usually specified as Durometer A scale per ASTM D2240. The hardness specified in the 9975 SARP is 75+/-5A. O-ring hardness is not verified prior to installation, so not an acceptance criterion. The A-scale method truly applies to O-rings >0.635 cm thick, whereas 9975 O-rings have a nominal thickness of 0.353 cm. O-ring hardness was measured using a micro-hardness (M-scale) tester per ASTM D1415. The M-scale hardness values for GLT-S O-rings (82-84.5M) were consistently slightly higher than values for GLT O-rings (76.5-81M). The correlation between A and M-scale hardness values is not well-defined, though the values tend to be close. Durometer A tests on GLT-S and GLT slab samples also showed a slightly higher hardness for the GLT-S compound, with most values just outside the 75+/-5A range. The slightly higher hardness observed for GLT-S O-rings is not considered critical to performance during transportation or storage.

The organic composition of GLT and GLT-S O-rings was analyzed via FT-IR spectrophotometry. The G in the designation indicates that the polymer is peroxide-curable (G), with LT designating low temperature, a primary reason for use in the 9975 as the seals must be leaktight to -40 °C. A proprietary cure site monomer (CSM) based on bromine was historically used in the GLT polymer to react end groups during the curing. The cure site monomer used in the GLT-S polymer is iodine-based, improving curing efficiency.

Samples of raw GLT-200S and GLT-600S polymers were analyzed in addition to as-received O-rings. FT-IR spectra for GLT-S and GLT O-rings are shown in Figure 8, with nearly identical results. The largest peak in the spectrum at ~ 1190 cm^{-1} is due to the absorbance of infrared light by the C-F part of the molecule and conversion of the energy to vibrations. The C-F stretch absorbs between 1300-1000 cm^{-1}. The small peak at ~890 cm^{-1} is also likely due to C-F stretching (bending occurs lower at ~650 cm^{-1}). The small peak at ~1390 cm^{-1} is likely due to C-H bending. The spectra for the O-ring compounds (bottom curves) are slightly influenced by the presence of carbon black.

From these data alone, it is very difficult to distinguish the materials. This is not unexpected, as the principal monomers used in the GLT and GLT-S polymers are reportedly the same and in the same proportion. The results suggest that the materials are chemically very similar and similar aging characteristics might be expected.

X-Ray diffraction (XRD) analysis was performed to identify possible inorganic variations in the compounds that may relate to aging performance. XRD analysis of the GLT-S compound detected barium sulfate, magnesium oxide, calcite (calcium carbonate) and hydrotalcite (Figure 9). XRD analysis of the GLT compound detected only zinc oxide and hydrotalcite (Figure 10). Per polymer manufacturer representatives, barium sulfate was not added to the legacy GLT polymer. Therefore, the presence of barium sulfate and/or magnesium oxide in the GLT-S compound may provide a possible way to distinguish between the materials if needed.

Energy-dispersive X-ray analysis was performed to identify possible elemental variations between the GLT and GLT-S compounds. Elemental spectra for the GLT compound indicate that the cure site monomer based on bromine can possibly be detected by this method.

Neutron activation analysis (NAA) was performed to determine the chloride content of the GLT-S compound. The total chloride content of one GLT-S sample was 151 ppm, as compared to 175 ppm for a GLT sample. Both values are below the 250 ppm limit imposed by SRS standards for materials in contact with austenitic stainless steels in certain environments.

GLT and GLT-S O-rings were analyzed via DSC to characterize thermal behavior and identify potential transitions in either the normal storage temperature range or the temperature range used for accelerated-aging. The DSC equipment used is not suitable for sub-ambient measurements so the glass transition temperature (T_g) could not be determined. However, the baseline DSC analysis (Figure 11) indicates that the GLT and GLT-S compounds are very similar and exhibit no significant thermal transitions in the normal storage range (4-93 °C) or the range used for accelerated-aging of GLT O-rings (79-177 °C). Additional DSC runs on aged O-rings will be performed and results reported at a later date. Sub-ambient DSC measurements are desirable for comparison to DMA results.

Dynamic Mechanical Analysis (DMA) is a useful tool to study polymers because the elastic and viscoelastic properties are significantly affected by morphological changes, such as glass and crystallographic transitions, cross-linking and chain scission, and approaching the melting point. The glass transition temperature (T_g) of elastomers is particularly of interest for low-temperature performance. Although not a storage criterion, the 9975 O-rings are required to remain leaktight to -40 °C during transport Rectangular pieces of GLT and GLT-S slabs were mounted in a single cantilever clamp. The width and thickness of the samples were approximately 12.7 mm x 3 mm. Per ASTM E 1640, the baseline T_g values for the GLT-S and GLT compounds were determined to be -32.4 °C and -30.9 °C, respectively (Figures 12-13). SRNL measurements are very close to polymer manufacturer values, with minor variations expected due to different techniques.

The T_g values of samples gamma-irradiated to 50 Mrad (500 kGy) are also very comparable, with only slight changes noted (Figures 14-15). In both irradiated samples, the T_g value was slightly increased. The 500 kGy dose was selected to easily bound a 50-year storage period and to challenge the O-rings within a reasonable exposure period for comparison. In service, the O-rings will only see a bounding dose rate of 0.02 Gy/hr.

Though not equivalent to leak testing at low temperatures, the T_g data suggest the GLT-S O-rings should perform the same or slightly better than GLT O-rings at low temperatures. Some references indicate marginal performance of GLT O-rings at -40 °C [6, 7], though the 9975 design has been qualified to -40 °C. The effects of long-term aging at elevated temperature on low-temperature performance have not been determined.

The baseline compression stress relaxation (CSR) behavior was determined for GLT and GLT-S O-rings for 1000 hours at temperatures of 93, 149, 177 and 204 °C per ASTM D6147. For reference, 177 °C is the PCV design temperature and 204 °C is the "continuous" upper temperature rating of the GLT O-rings. It is noted that most O-ring manufacturers quote an upper service temperature that is based on a limited exposure period (1000 hours) [8].

CSR data for GLT and GLT-S O-rings of 0.353 mm thickness, compressed 20% as expected in service and aged for 1000 hours per temperature are given in Figure 16. As shown, the CSR behavior of GLT and GLT-S O-rings is very similar at 149 °C, with the GLT O-rings showing a lower normalized sealing force. At 177 °C, the curves are more widely separated but seem to follow the same trend. These curves may converge over longer aging periods, but this is yet unknown. Longer times at temperature are required to determine this behavior.

At 204 °C, the CSR curves for both GLT and GLT-S O-rings are essentially equal at 1000 hours. The GLT samples appear to show a sharper initial drop in sealing force particularly at 177-204 °C, possibly attributed to the less efficient polymer network and non-functional ionic end groups allowing more physical relaxation. Based on these results, O-rings made from the GLT-S compound are expected to provide similar if not slightly improved aging behavior with regard to retained sealing force. It is possible that the data curves level off or converge at some point for all temperatures. Therefore, the use of short-term tests alone for life prediction is not sufficient. Long-term CSR behavior of GLT and GLT-S O-rings are being monitored at 79-177 °C to develop an aging model.

Discussion

Fiberboard

Overall, similar aging trends are observed for softwood and cane fiberboard samples. However, there are modest differences in some properties, which warrant further observation. In comparing the physical property data for the two materials (softwood and cane fiberboard), the results for the softwood fiberboard samples may not be fully representative since they have been conditioned for less than 1 year. Seasonal variation in ambient moisture level will also affect the physical properties, and this effect will superimpose on any change due to degradation. This is most pronounced for samples in the 85 °C oven, and has minimal effect in the other environments.

In calculating the rate of change for the density and dimension data in Table 1, the first 70 days of data were ignored, due to a significant degree of scatter during this period. That leaves ~8 months of data to consider. Since this time frame extends across a period of changing ambient humidity, seasonal variation may impact the observed change in properties. Longer-term data from softwood fiberboard samples will help eliminate this bias and demonstrate whether there is a significant difference in the physical behavior of the two materials.

For the physical property samples in the 85 °C 30%RH environment, the weight, density and height have decreased faster for softwood fiberboard than for cane fiberboard. The rate of weight loss for softwood samples in the 121 °C oven was greater than the rate for cane fiberboard (17%/yr vs 14 %/yr. However, the weight loss for cane fiberboard during its first year was 21%. It is likely that the reported differences between the two materials will decrease as additional data are collected.

The compression test data are compared to cane fiberboard results in Tables 2 and 3. In several cases, the compression test metrics (buckling strength, and area under the stress-strain curve to 40% strain) for softwood fiberboard fall below the comparable ranges reported for cane fiberboard, while many of the metrics fall within the range for cane fiberboard. While there is sample-to-sample variation for both materials, the softwood fiberboard appears weaker than cane fiberboard, on average.

The thermal data collected to date on softwood fiberboard appears similar to that for cane fiberboard. The normalized thermal conductivity data (Figure 5) show very similar trends for both materials in each environment. The biggest difference is seen at 121 °C, where the thermal conductivity of the softwood has not decreased quite as fast as that of the cane fiberboard. There is significant scatter in the specific heat capacity data for both materials, but no significant difference is seen between the two materials.

GLT-S O-rings

The properties and composition of GLT-S O-rings are similar to the GLT O-rings currently used in the 9975 shipping package. From literature review and baseline characterization, the GLT-S O-rings are expected to provide comparable if not superior performance. The GLT-S O-rings are therefore considered acceptable for an initial 10-year interim storage period in KAMS. Performance over longer storage periods is still be assessed.

The hardness of GLT-S O-rings was determined to be slightly higher than that of the GLT compound. This is consistent with O-ring manufacturer findings. The 75+/-5A hardness range quoted for the compound in the 9975 SARP does not technically apply to the specific size O-rings used. This is not considered an issue for shipping or interim storage and hardness verification is not required prior to use.

GLT-S O-rings exhibited higher initial sealing force and retained higher values of sealing force at test temperatures during the 1000-hour test period. The percentage of retained sealing force at 1000 hours is approximately the same for both compounds. These data indicate comparable and possibly superior aging performance of the GLT-S O-rings in KAMS, though this behavior may reverse at lower temperatures over longer aging times.

To validate these conclusions and provide a model for seal life prediction over longer periods, longer-term CSR tests on GLT-S O-rings are in process, similar to those currently being performed for GLT O-rings.

A limitation of CSR testing is that the time to reach target values of sealing force at all aging temperatures can be lengthy. This is more problematic for materials that normally function at room temperature, making valid extrapolations from elevated temperatures more difficult. If Arrhenius behavior occurs across the full aging range, elevated-temperature CSR data are more likely to reasonably predict behavior at lower temperatures. However, several studies have shown non-Arrhenius behavior in certain elastomers, which can invalidate life prediction by CSR alone [9, 10]. Non-Arrhenius behavior may occur due to oxidation at lower temperatures or other reasons. The potential for this behavior is being considered for future tests.

Conclusions

Overall, similar aging trends are observed for softwood and cane fiberboard samples. Some of the observed differences result from the limited exposure periods of the softwood fiberboard samples, and the impact of seasonal humidity levels. Testing following additional conditioning will continue and should eliminate this bias.

While the compression strength of the two materials generally falls within an overlapping range, the softwood fiberboard samples tested as part of this current effort tend to be weaker, on average, than cane fiberboard. As material from additional softwood fiberboard assemblies becomes available for testing, more information on sample variation can be developed.

Based on current data, GLT-S O-rings are overall expected to provide similar aging behavior at realistic storage temperatures as compared to legacy GLT O-rings. CSR and leak performance will continue to be monitored to validate these assumptions. Additional testing is planned to evaluate the potential for non-Arrhenius aging behavior.

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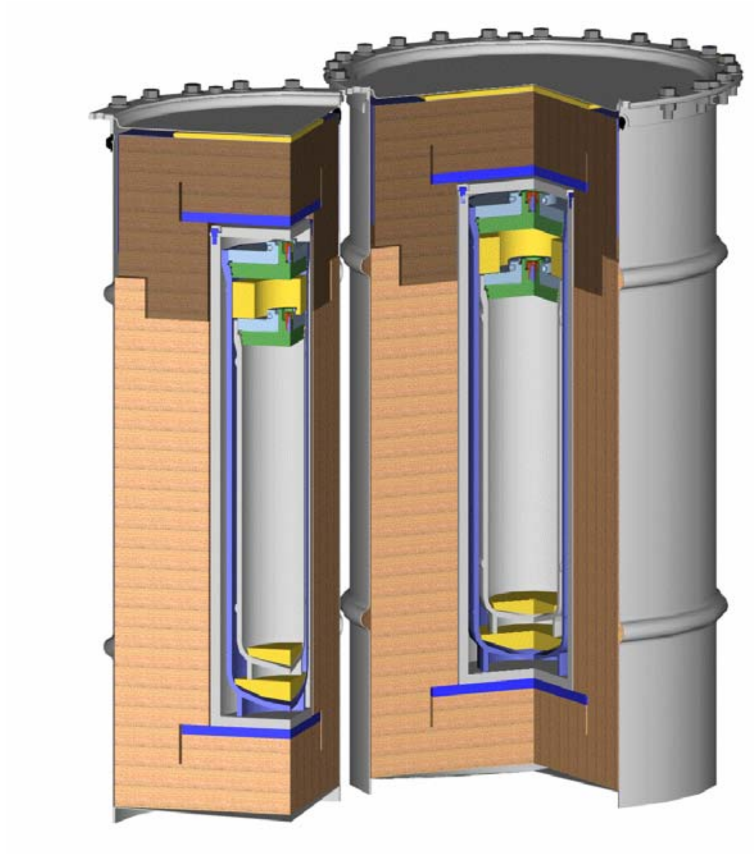


Figure 1. Illustration of 9975 package including fiberboard, lead shielding, and double containment vessels sealed with dual O-rings.

Table 1. Physical property changes in softwood vs cane fiberboard

		Softwood Fiberboard		Cane Fiberboard	
Property	Environment	Duration of Data (days)	Rate of Change (%/yr)	Duration of Data (days)	Rate of Change (%/yr)
Weight	121C, dry	303	-17.05	1104	-13.94
	102C, dry	307	-3.76	1116	-3.60
	85C, dry	314	-1.04	1106	-1.08
	85C, 30%RH	201	-7.70	666	-3.46
Density	121C, dry	230	-7.85	1104	-7.66
	102C, dry	234	-0.69	1116	-1.89
	85C, dry	241	+1.00	1106	-0.18
	85C, 30%RH	128	-4.20	666	-1.35
Height	121C, dry	230	-6.86	1104	-5.48
	102C, dry	234	-1.55	1116	-1.07
	85C, dry	241	-1.04	1106	-0.49
	85C, 30%RH	128	-2.87	666	-1.05
Length, Width	121C, dry	230	-0.60	1104	-1.81
	102C, dry	234	-0.58	1116	-0.40
	85C, dry	241	-0.52	1106	-0.21
	85C, 30%RH	128	-0.86	666	-0.58

Table 2. Summary of compression test metrics – parallel orientation

Condition	Area under Stress-Strain Curve to 40% Strain (kPa)		Buckling Strength (kPa)	
	Softwood Fiberboard	Cane Fiberboard	Softwood Fiberboard	Cane Fiberboard
Baseline	380	290 – 540	1750	1075 – 2825
85C, dry, 8 wks	195	255 – 260	1310	1070 – 1460
85C, dry, 32 wks	105	130 – 460	1560	550 – 2400
85C, 30%RH, 8 wks	205	185 - 460	1385	1560 – 2165
85C, 30%RH, 32 wks	105	195 - 295	960	1385 – 2000
121C, dry, 8 wks	95	75 – 285	1075	860 – 1770
121C, dry, 16 wks	55	75 – 125	740	750 – 835
121C, dry, 32 wks	95	110 - 145	915	650 – 930

Table 3. Summary of compression test metrics – perpendicular orientation

Condition	Area under Stress-Strain Curve to 40% Strain (kPa)	
	Softwood Fiberboard	Cane Fiberboard
Baseline	260	215 - 365
85C, dry, 8 wks	330	370 – 380
85C, dry, 32 wks	345	275
85C, 30%RH, 8 wks	270	330 - 365
85C, 30%RH, 32 wks	240	195 – 385
121C, dry, 8 wks	285	295 – 370
121C, dry, 16 wks	250	145 – 180
121C, dry, 32 wks	215	145 – 275

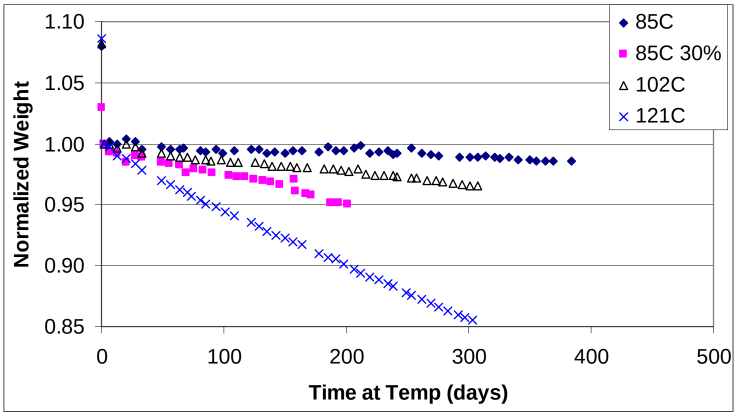
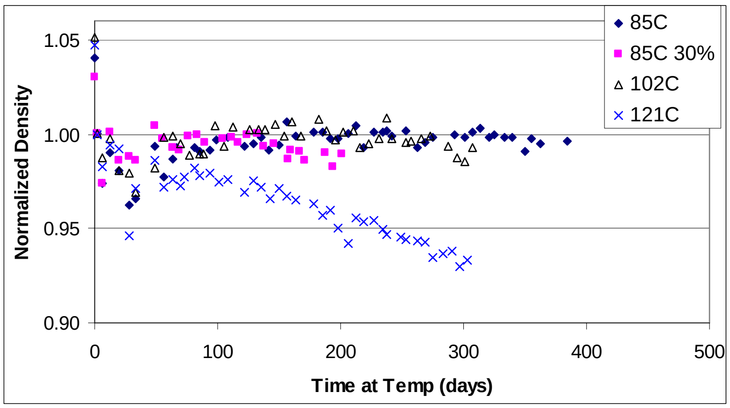


Figure 2. Normalized physical data for softwood fiberboard samples.

(a) Weight change



(b) Density change

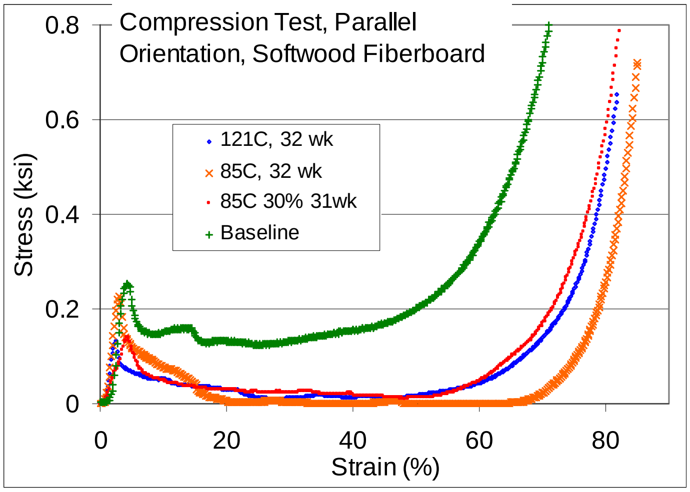


Figure 3. Compression stress-strain curves for softwood fiberboard samples, parallel orientation

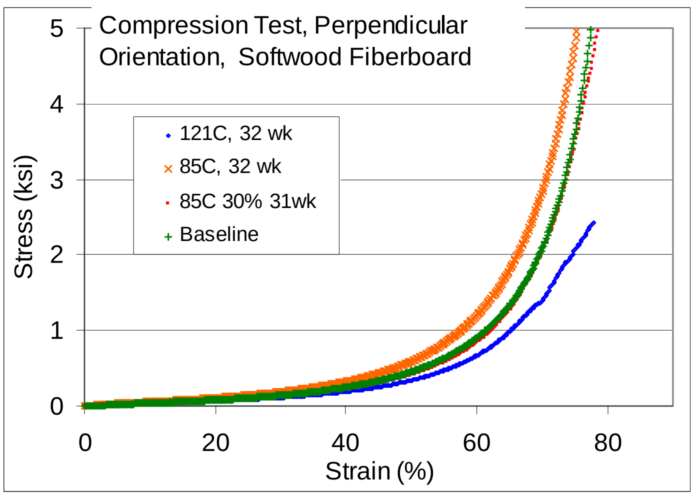


Figure 4. Compression stress-strain curves for softwood fiberboard samples, perpendicular orientation

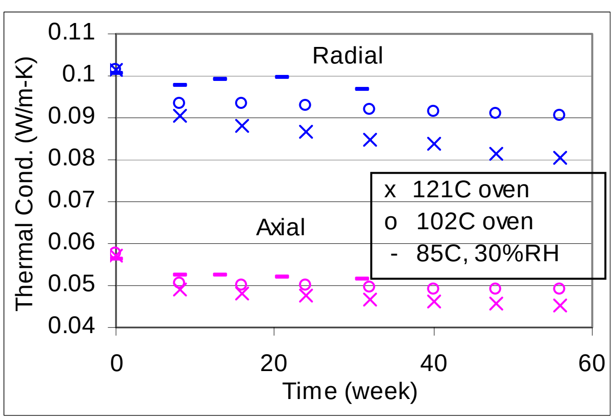
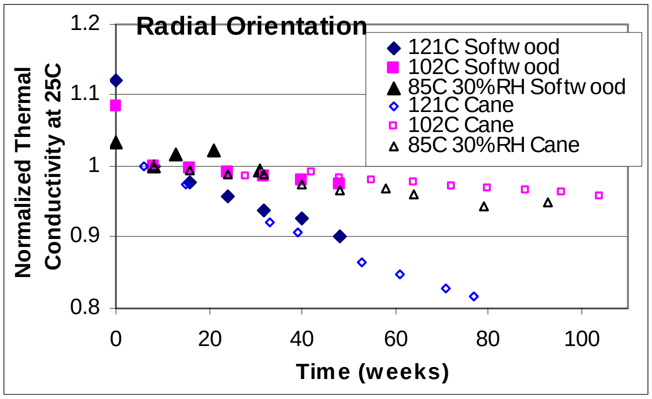
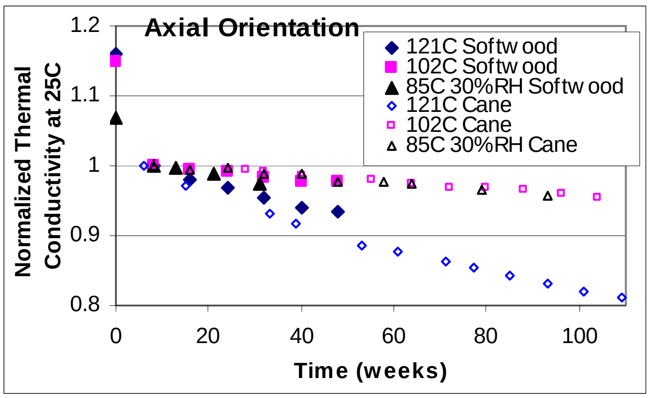


Figure 5. Softwood fiberboard thermal conductivity data at 25C mean temperature



(a) radial orientation



(b) axial orientation

Figure 6. Normalized thermal conductivity data for softwood fiberboard compared to data for cane fiberboard.

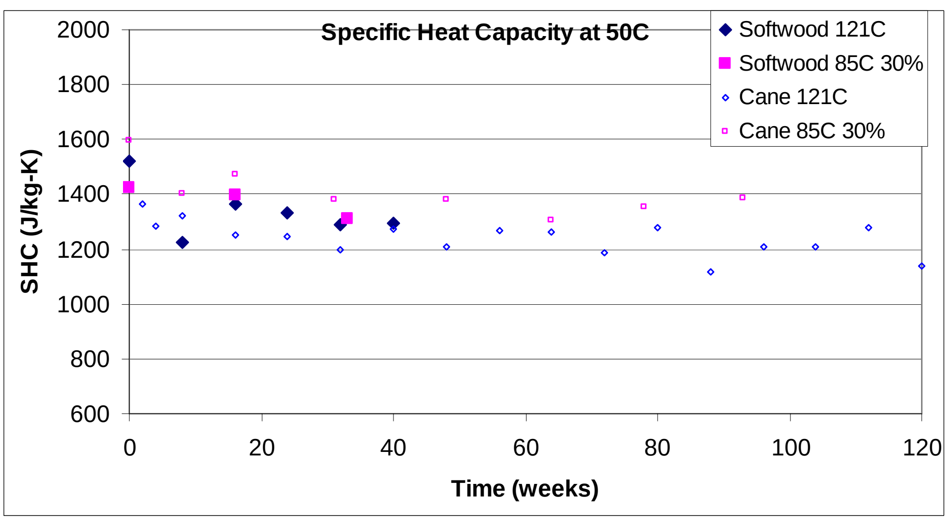


Figure 7. Specific heat capacity data for softwood fiberboard compared with cane fiberboard

50C mean temperature

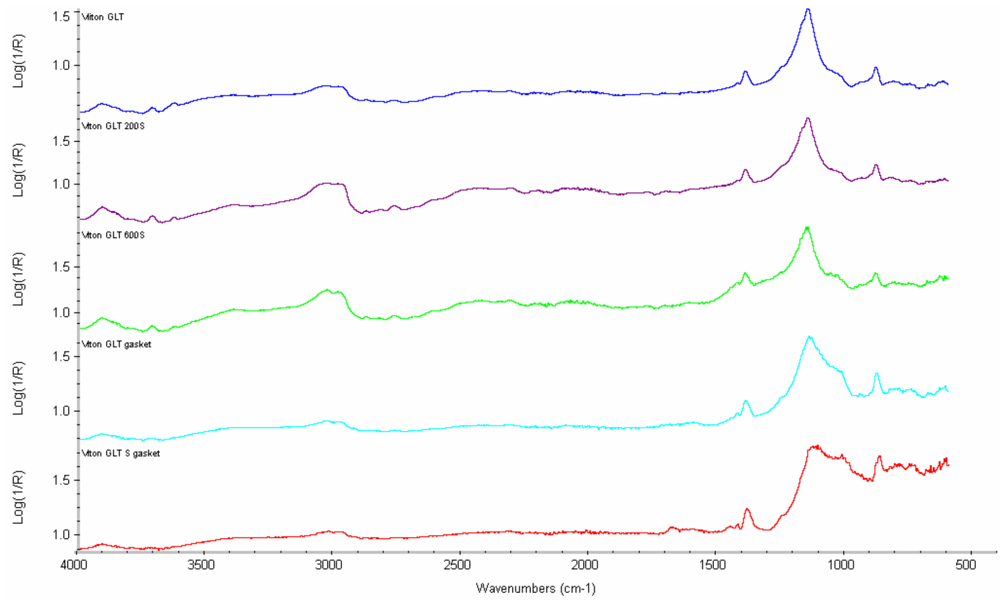


Figure 8. FT-IR spectra for GLT, GLT-200S, GLT-600S, V0835-75 and VM835-75 compounds

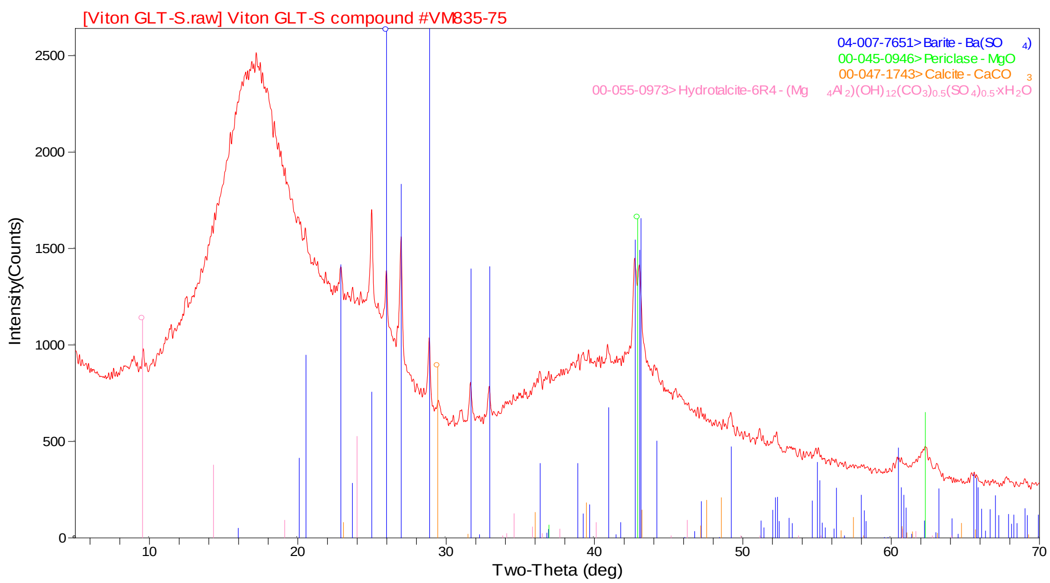


Figure 9. XRD spectra of Viton GLT-S (VM835-75) compound.
(barium sulfate, magnesium oxide, calcite, and hydrotalcite detected)

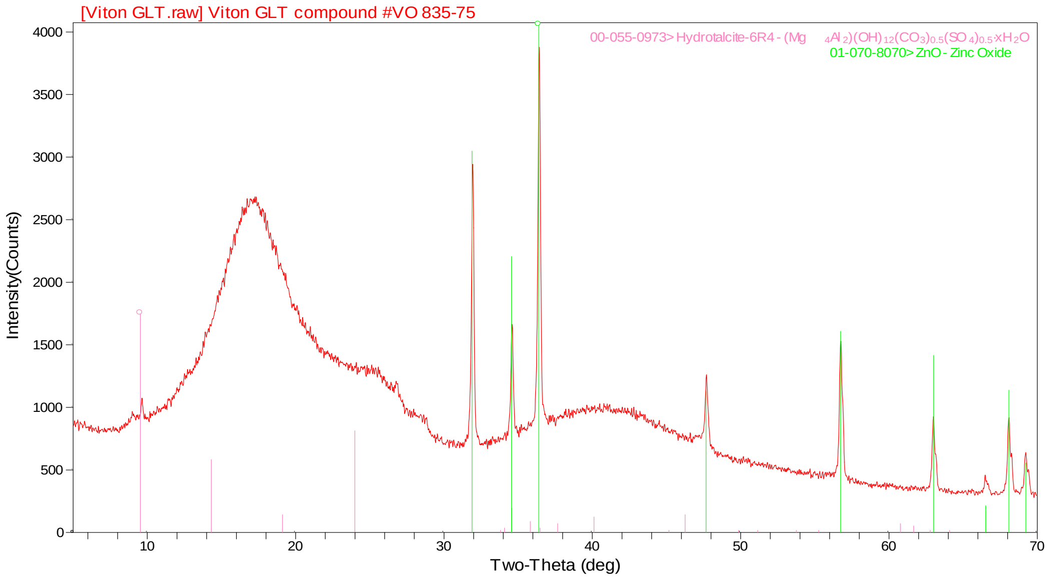


Figure 10. XRD spectra of Viton GLT compound (V0835-75)
(Zinc oxide and hydrotalcite detected, no barium sulfate or magnesium oxide)

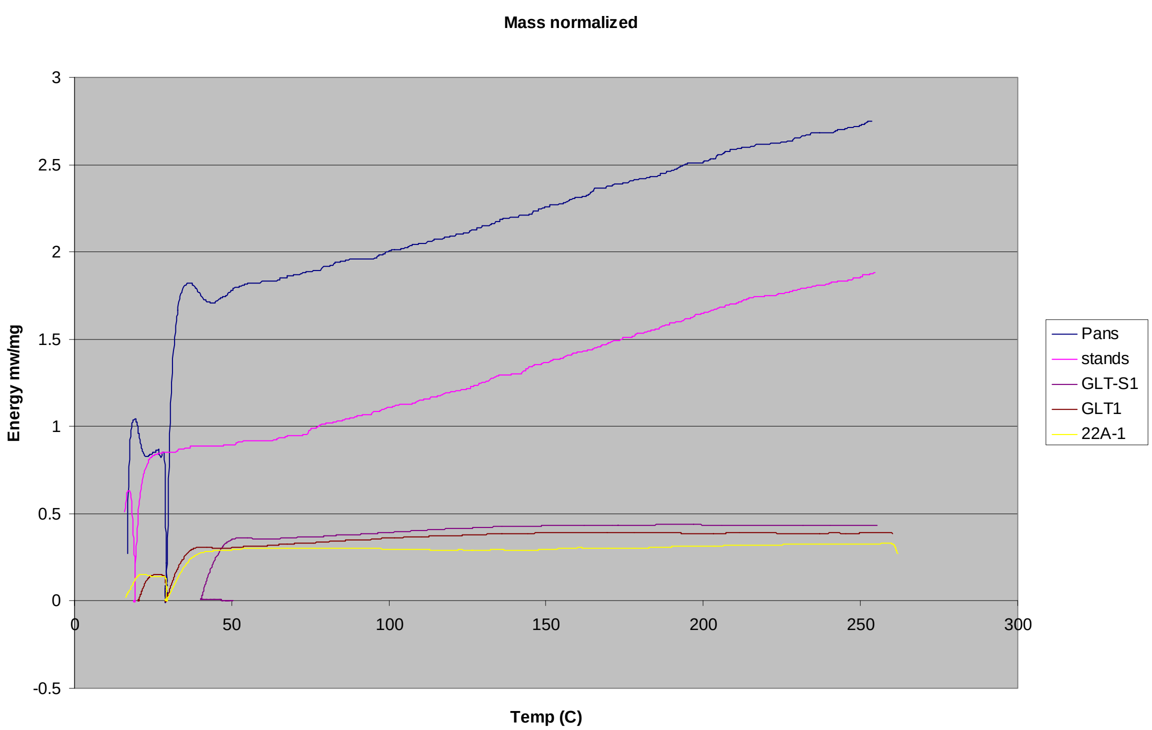


Figure 11. DSC curves for non-aged GLT and GLT-S Compounds
(no peaks or transitions in the 50-250 °C range)

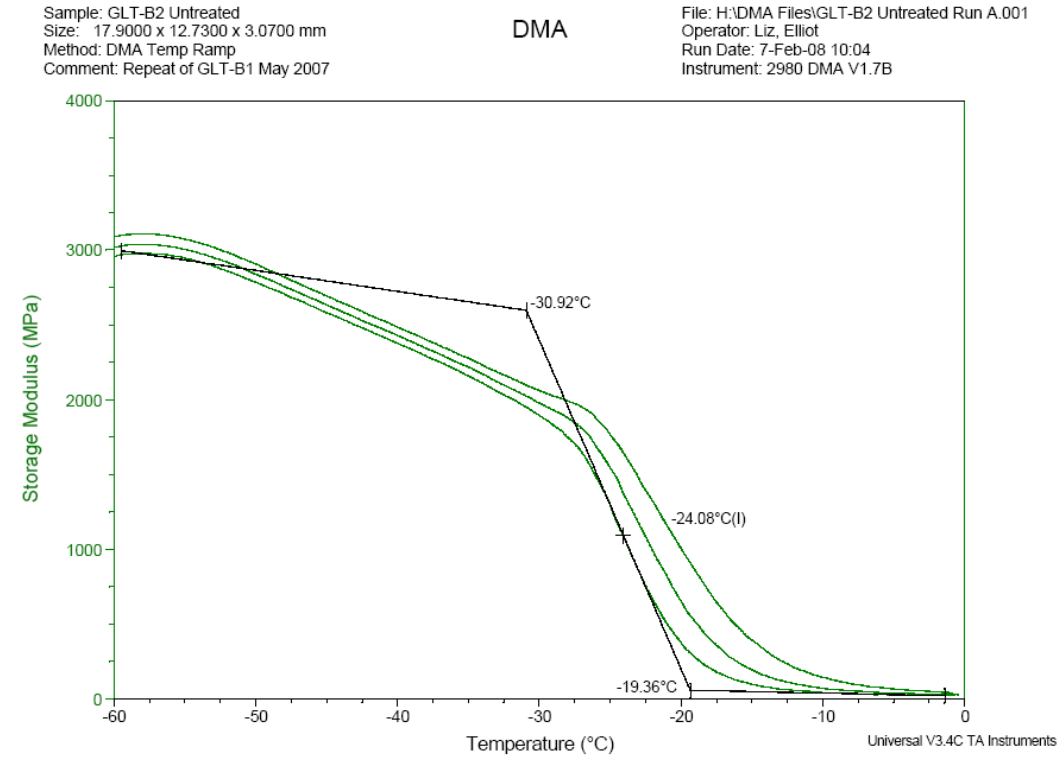


Figure 12. GLT Baseline DMA Curve

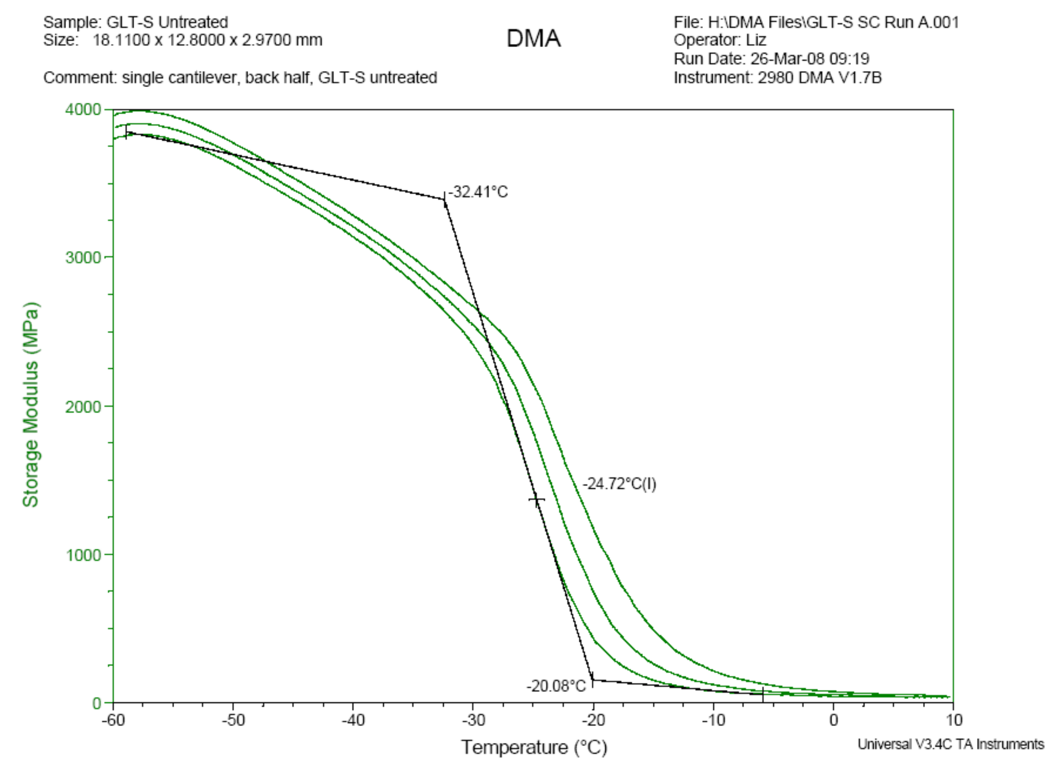


Figure 13. GLT-S Baseline DMA Curve

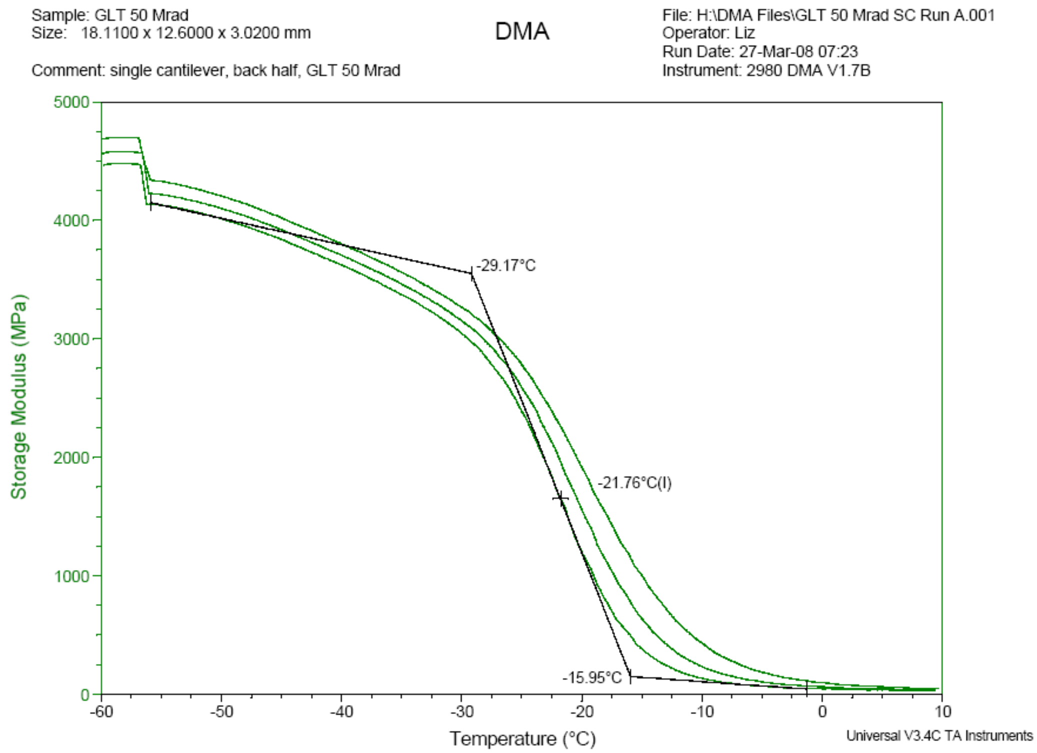


Figure 14. DMA Curve for GLT Irradiated to 500 kGy (50 Mrad)

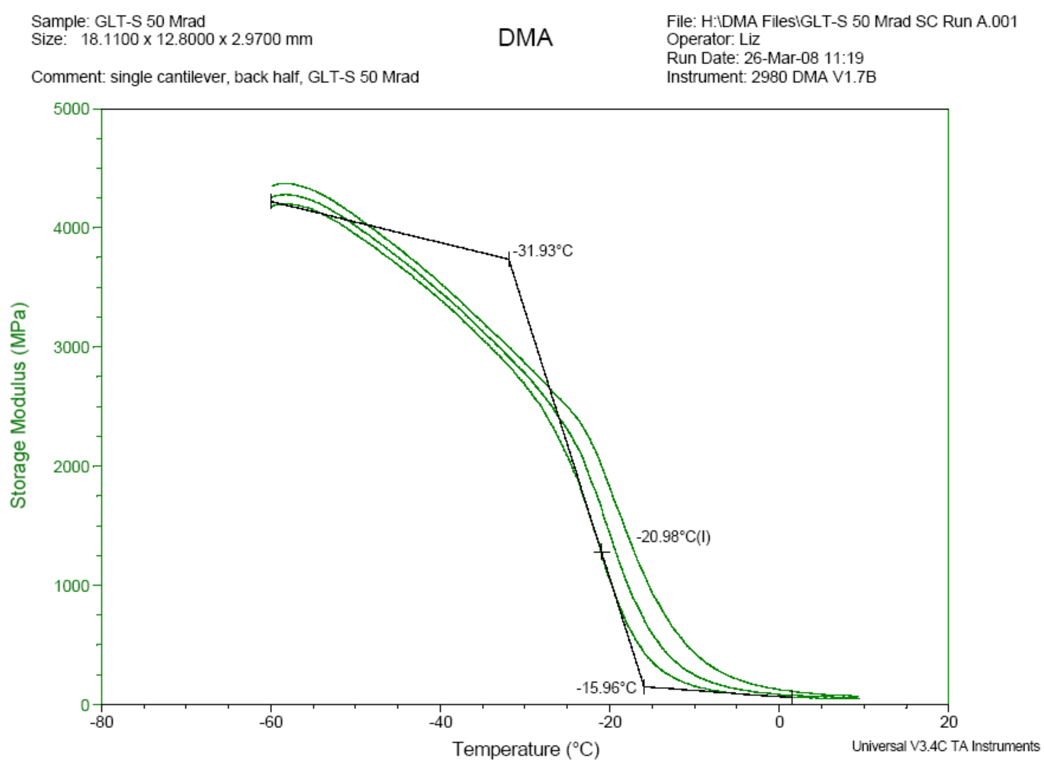


Figure 15. DMA Curve for GLT-S Irradiated to 500 kGy (50 Mrad)

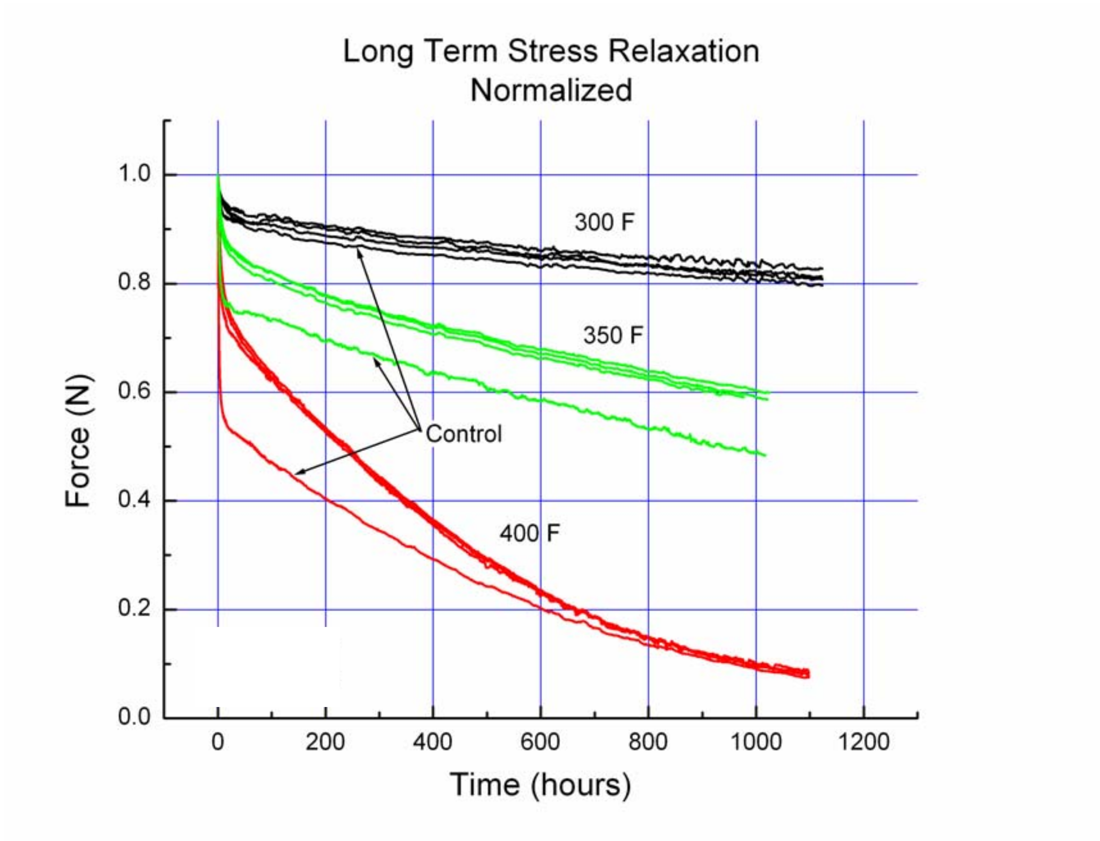


Figure 16. Normalized CSR Curves for GLT-S and GLT O-rings (20% compression)