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Characterization of Solid Polymers, Ceramic Gap Filler, and Closed-Cell Polymer Foam Using Low-Load Test Methods

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Abstract

Various solid polymers, polymer-based composites, and closed-cell polymer foam are being characterized to determine their mechanical properties, using low-load test methods. The residual mechanical properties of these materials after environmental exposure or extreme usage conditions determines their value in aerospace structural applications. In this experimental study, four separate polymers were evaluated to measure their individual mechanical responses after thermal aging and moisture exposure by dynamic mechanical analysis. A ceramic gap filler, used in the gaps between the tiles on the Space Shuttle, was also tested, using dynamic mechanical analysis to determine material property limits during flight. Closed-cell polymer foam, used for the Space Shuttle External Tank insulation, was tested under low load levels to evaluate how the foam's mechanical properties are affected by various loading and unloading scenarios.

Introduction

In order to determine the most suitable materials for a specific structural application, the possible influence of extreme environments and vigorous usage on bulk mechanical properties must be given careful consideration. The most common environmental factors that can affect mechanical properties are temperature and moisture. In some cases, extreme exposure to high temperatures or humidity can alter the mechanical response of a material significantly and render it useless in particular applications. For certain solid polymers, the mechanical properties may be diminished after prolonged exposure to heat or humidity to a point of being incapable of performing to expectations. Therefore, it is necessary to employ test methods, specifically designed to mechanically characterize such polymers, to evaluate the effects of extreme environmental exposure on bulk properties. One such method is dynamic mechanical analysis (DMA), which can acquire accurate mechanical property data on a small scale so as to provide more efficient usage of test material.

Thermal protection tiles are installed on the outer surface of the Space Shuttle Orbiter to shield the

vehicle from the intense heat, experienced during re-entry. These tiles are protected from the bending of the aluminum skin of the orbiter by a Strain Isolator Pad (SIP) and filler bar system. Their installation results in a gap between adjacent tiles. The dimensions of these gaps are limited to preclude sub-surface inner mold line heating during re-entry. Because the gaps between some tiles can exceed the dimensional limits, silica-based ceramic gap fillers are used to maintain allowable tolerances. There is a risk that these gap fillers detach from their bonding surfaces during the ascent and re-entry trajectories, resulting in a protuberance in the local flow field. These protuberances are known to disturb boundary layer flow which can lead to significant increases in surface convective heating and, potentially, asymmetric aerodynamic effects if only one side of the windward surface is affected. Gap filler material was characterized, using the DMA, to assist in predictions for a shuttle mission that was in progress. This characterization was necessary to predict the gap filler deflection and boundary layer transition, needed by the Shuttle Program Mission Management Team to support reentry flight decisions. [1]

Understanding the mechanical behavior of the closed-cell polymer foam, which covers the Space Shuttle External Tank as part of its thermal protection system, is of critical importance. Additionally, knowing the effect on the mechanical properties of different loading and unloading sequences and various environmental conditions can lead to improvement in material performance in future applications. Thus, it is imperative that full material characterization simulates the range of loads and environments this foam would see during its regular usage.

The objective of this report is to determine experimentally the effects of extreme environmental exposure on the storage modulus of selected structural polymers using DMA, the mechanical characterization of Ames ceramic gap filler material, and both the effect of varied environmental and mechanical loading sequences on material response of closed-cell polymer foam. The solid polymers were exposed to either elevated temperatures or moisture for various increments of time prior to mechanical testing. For proper evaluation, the storage modulus of each individual exposed solid polymer sample was compared to that of a baseline, as-received sample. The tile gap-filler material was tested to failure under a static bending load, using DMA, at the request of Langley Research Center's Structural Dynamics Branch. The Shuttle Tank foam was tested to determine the effects of the thermal and mechanical loading sequence on the stiffness.

Materials

Four distinct solid polymers, selected for environmental exposure and characterized using DMA, are considered typical materials for elevated temperature aerospace applications. Polymer 8320 is a polyarylsulfone thermoplastic manufactured by the Amoco Corporation. LaRC-SI is a thermoplastic polyimide with a 5% stoichiometric offset made by Imitech Inc. K3B is a thermoplastic polyimide made by DuPont, and 5260 is a modified bismaleimide thermoset polymer made by BASF Corporation. The gap-filler material consists of a woven fabric, coated with a silicon carbide. The foam used on the Space Shuttle External Tank is BX-265 Closed-Cell Polyurethane applied with a CFC-11 chlorofluorocarbon blowing agent.

Experimental Procedure

Dynamic Mechanical Analysis of Solid Polymers

In order to obtain material stiffness as a function of temperature, dynamic mechanical analysis (DMA) was performed on all solid polymer samples. Briefly, DMA provided a measure of the dynamic storage modulus over a range of temperatures. DMA is a standard thermal analysis method, and explanations of the theory and operation for DMA can be found in [2] and [3].

There were three bar samples cut for each polymer tested in this study. From these three, one was to be used as a baseline, one for isothermal elevated-temperature aging, and one for boiling in water. The aging and boiling conditions for each polymer are given in Table 1. The bar samples were cut to be between 5 and 15 mm in width and 55 and 60 mm in length, using a calibration standard template provided with the DMA apparatus, to conform to the dimensional limits required for the test fixture. The average thickness for each bar sample was based on four separate measurements, taken along the length of the specimen with a digital micrometer, not to exceed 5 mm. A photograph of the DMA 3-point bend clamp is shown in Figure 1.

The DMA test procedure was as follows: After preconditioning either by elevated-temperature aging or boiling, a sample was mounted across the 3-point bend supports, and the moveable clamp was placed in position at the center of the bar. The furnace was then sealed, and the sample was heated at 3°C/min. from ambient temperature to beyond its approximate glass transition temperature. All samples were tested with an applied preload force of 0.5 N, using a dynamic force with a single frequency oscillation of 1 Hz and amplitude of 20 μm. The storage modulus for each run was calculated as a function of increasing temperature, using thermal analysis software included with the DMA apparatus.

Dynamic Mechanical Analysis of Gap-Filler Composite

Three-point bend testing of gap-filler specimens to failure was conducted at room temperature using DMA. The test method was a force ramp while recording the load vs. deflection. The distance between the rollers for the three-point bend was 20 mm (0.8 in) for this experiment. The average specimen size was approximately 0.5-in wide and 1.6-in long, and a total of fourteen (14) specimens were tested.

Mechanical Testing of Closed-Cell Foam

Standard tension specimens of the closed-cell polyurethane foam were tested with and without initial cracks and subjected to various thermal and mechanical loading sequences. Prior to testing, a speckle pattern was spray-painted on one side of each specimen to allow strain measurements to be taken with a visual imaging system. Each specimen was then placed in a cryogenic bath, which was constructed as part of a horizontal tension test frame with an environmental chamber for the purpose of cryogenic testing. Figure 2 shows the sample mounted in the cryogenic bath assembly in the horizontal test frame. Also shown in this figure is the control station, which consisted of load frame and environmental controls,

data acquisition, and both low- and high-speed video image acquisition. Four separate test scenarios were considered:

Tension test to failure at room temperature,

Cool down to -325°F and then tension test to failure,

Tension to 0.8Pcr at room temperature, cool down to -325°F at constant axial load, unload to 0.125Pcr, and then tension test to failure,

Tension test to 0.2Pcr at room temperature, cool down to -325°F at constant axial load, and then tension to failure,

where Pcr is the expected room temperature failure load. For the cryogenic phase of the test, the cryogenic bath assembly was filled with liquid nitrogen to a level just covering the sample to facilitate maximum saturation. All tests were run below 100 psi axial stress. All tests were recorded using low- and high-speed video with images being captured at various intervals of time throughout each test. Visual image analysis was performed to aid correlation of strain values. Comparison of data from specimens subjected to different loading sequences was made to identify changes in the response.

Results and Discussion

Dynamic Mechanical Analysis of Solid Polymers

Figures 3 through 6 are graphical representations of temperature versus storage modulus for each polymer, tested using DMA. Each DMA test run is shown by a single line and labeled according to that particular sample's pre-conditioned state. In all the polymers, the isothermally aged specimens registered a higher storage modulus than the baseline. However, the storage modulus for the boiled specimens fell below that of the baseline in all polymers with the exception of K3B. For the 8320 specimens, the changes in the storage modulus by isothermal aging or boiling were minimal when compared with the baseline. For LaRC-SI 5%, the storage modulus for the thermally aged sample was only slightly higher than the baseline, but the boiled specimen's storage modulus was much lower. This same trend was also observed for the 5260 specimens with a more diminished storage modulus for the boiled sample than the baseline, but the thermally aged sample's storage modulus only minimally higher. The most change was seen in the case of K3B, where the storage modulus for the isothermally aged sample was much higher than that of the baseline. Additionally, the boiled sample expanded to approximately three times its original size as moisture escaped the polymer while being heated during the DMA test, and its storage modulus was slightly elevated above the baseline.

Dynamic Mechanical Analysis of Gap-Filler

For the gap-filler composite material, most bending loads were approximately linear with displacement until failure occurred. Various loads and loading rates were used to determine changes in

the deflection and the failure limit of this material. A failed specimen in the test apparatus is shown in Figure 7.

Mechanical Testing of Closed-Cell Foam

In Figure 8, a representative digital image of a closed-cell foam sample is shown under tension load. The center circle is the area upon which strain analysis was performed via a digital image correlation technique. These data were later correlated to the mechanical data and recorded as the specimen was loaded to failure. Each image, captured for a specific interval of time, was analyzed in this manner, and the strain, experienced by the sample during the test, was plotted. A graphical example of the load vs. strain response for the four particular loading scenarios is displayed in Figure 9. As can be seen, the cooling down in three of the four scenarios reduced the foam samples' strain as compared to the specimen that was loaded to failure at room temperature. For cases 3 and 4, where the samples were loaded at room temperature and then cooled before loading to failure, the initial strains were similar to that of the room temperature only samples. However, regardless of the point in the loading at which cooling was introduced, the end result of a reduction in strain still occurred.

Summary and Conclusions

Four distinct polymers were characterized, using DMA, after exposure to extreme environments. More specifically, the polymers were either aged at high temperature or boiled in water and then evaluated to determine the storage modulus. As seen in the analysis of the data, the aged specimens displayed a storage modulus higher than the baseline, while that of the boiled samples fell below the baseline in nearly every case except one. It was determined that the prolonged exposure of the various polymers to elevated temperature, and therefore the removal of most of the residual moisture, succeeded in boosting the storage modulus. On the other hand, the storage modulus was adversely affected by the boiling of each polymer except the K3B. Thus, it can be theorized that the presence of extra moisture diminishes the storage modulus of the majority of the polymers in this study.

The unique testing abilities of the DMA proved to be exceptionally valuable for the evaluation of the gap-filler composite material. This material required a method of analysis, using very low loads, which would allow the proper determination of the material's true failure limits. Utilization of the three-point bend test apparatus of the DMA provided the desired result.

The closed-cell polymer foam, used on the Space Shuttle External Tank, was subjected to varied thermal environments and mechanical loading sequences. The effect of these scenarios on the mechanical properties, particularly the strain, of the foam was measured both mechanically and visually. It was shown that altering the environment and loading of the foam resulted in a definite change in the material stiffness. Most notably, modifications in those loading cases in which extreme cooling was involved resulted in a lessening of the strain and failure response of the foam.

References

1. Campbell, C. H., Driver, D. M., Alter, S. J., Fasanella, E. L., Wood, W. A., and Stone, S. A.: "Orbiter Gap Filler Bending Model for Re-entry," AIAA Paper No. AIAA-2007-0413, 45th AIAA Aerospace Sciences Meeting and Exhibit, Reno, Nevada, 8-11 January 2007.

2. Menard, K. P., Dynamic Mechanical Analysis: A Practical Introduction. 1999, Boca Raton: CRC Press LLC.

3. Thompson, C. M, Herring, H. M. Gates, T. S., Connell, J. W., Preparation and Characterization of Metal Oxide/Polyimide Nanocomposites, Composite Science and Technology, Special Issue on Modeling and Characterization of Nanostructured Materials, ed. Thomas S. Gates, Elsevier, 2002.

Table 1. Sample Polymer Aging and Boiling Conditions

Polymer	Aging Temp./Time	Boiling Temp./Time
8320	215°C/~88Hrs.	100°C/~10min.
K3B	235°C/~88Hrs.	100°C/~10min.
5230	237°C/~88Hrs.	100°C/~10min.
LaRC SI 5%	230°C/~88Hrs.	100°C/~10min.

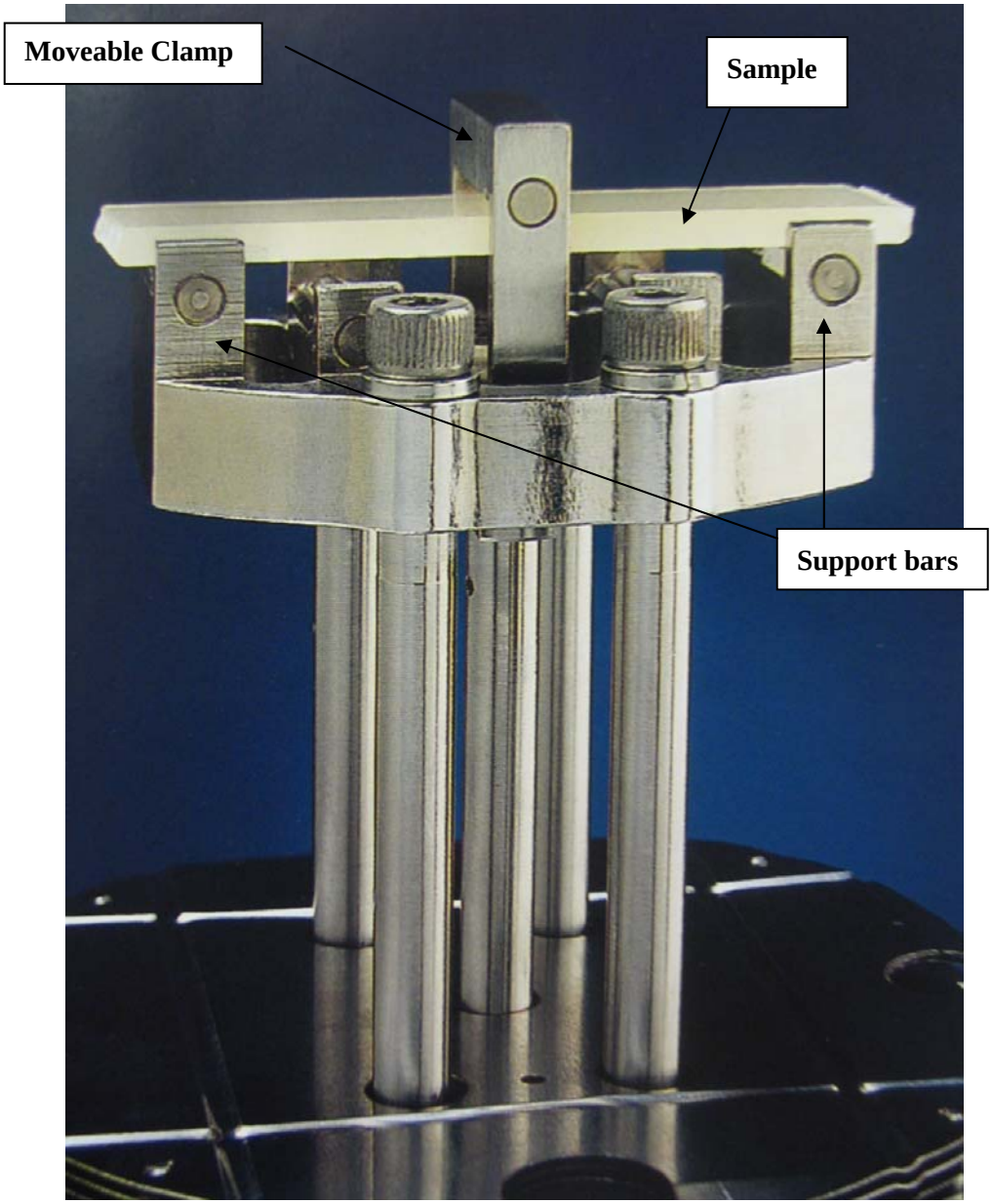

Figure 1. Dynamic Mechanical Analyzer with 3-Point Bend Clamp Installed

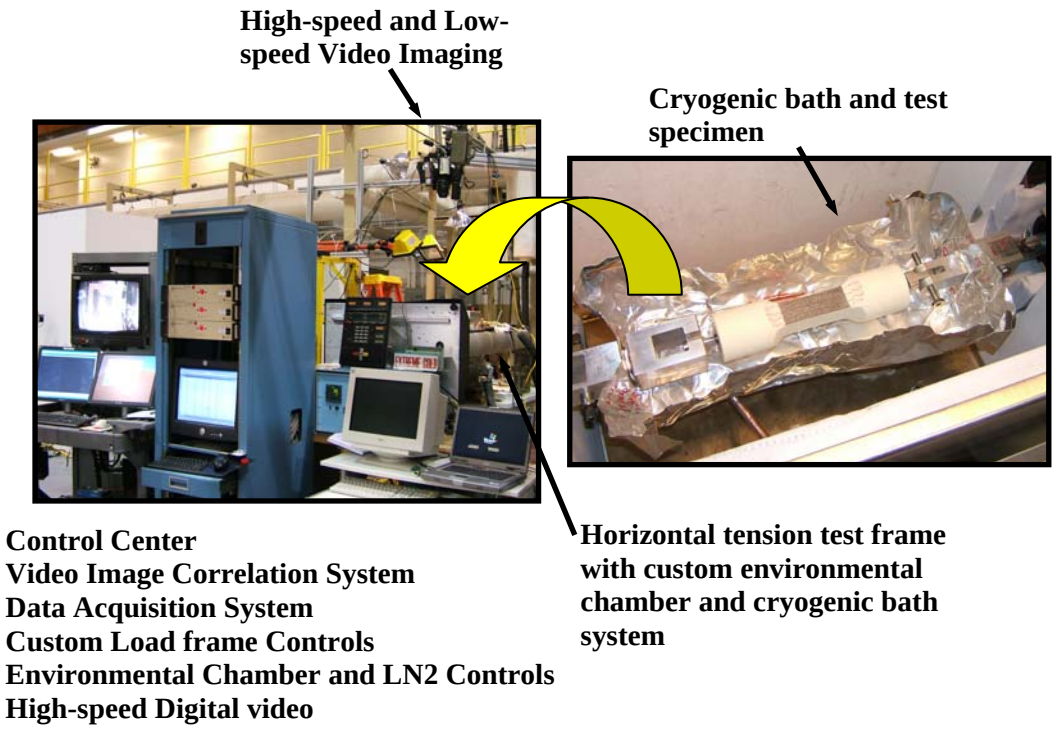

Figure 2. Tension Stand and Control Station Apparatus for Closed-Cell Foam Tests and Specimen in Cryogenic Bath

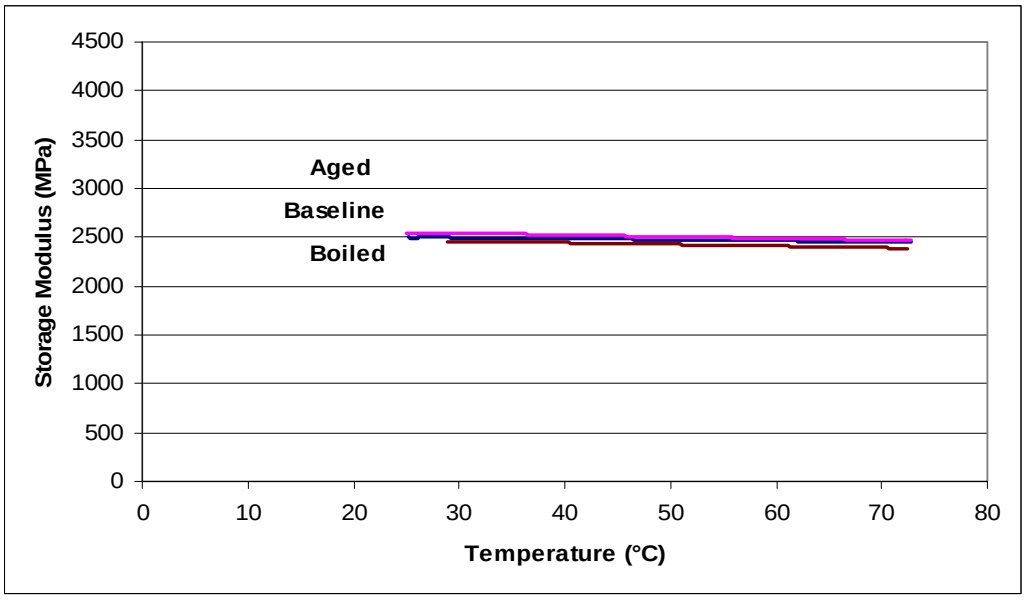

Figure 3. Dynamic Mechanical Analysis of 8320

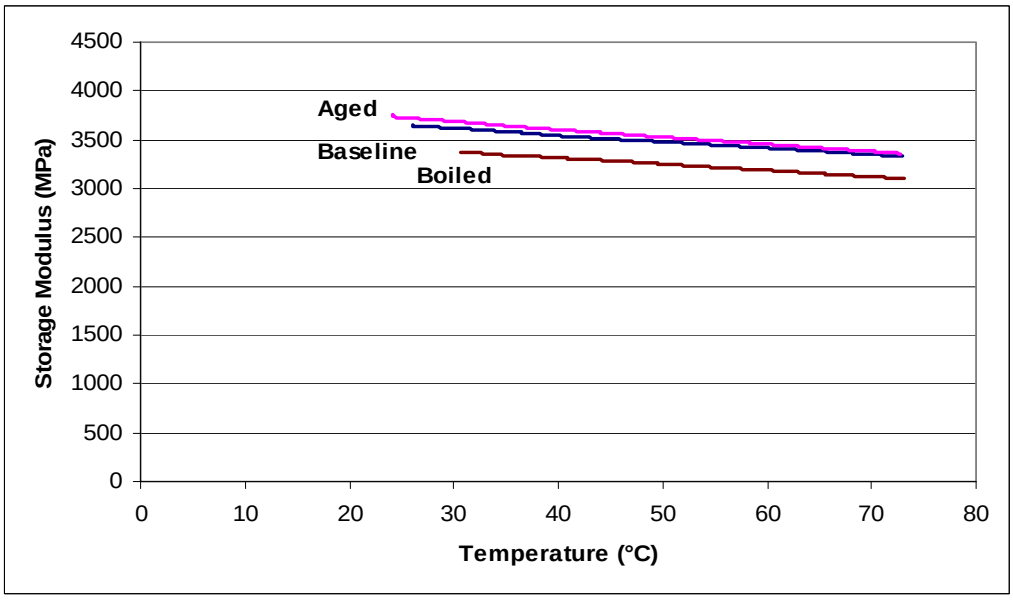

Figure 4. Dynamic Mechanical Analysis of LaRC SI 5%

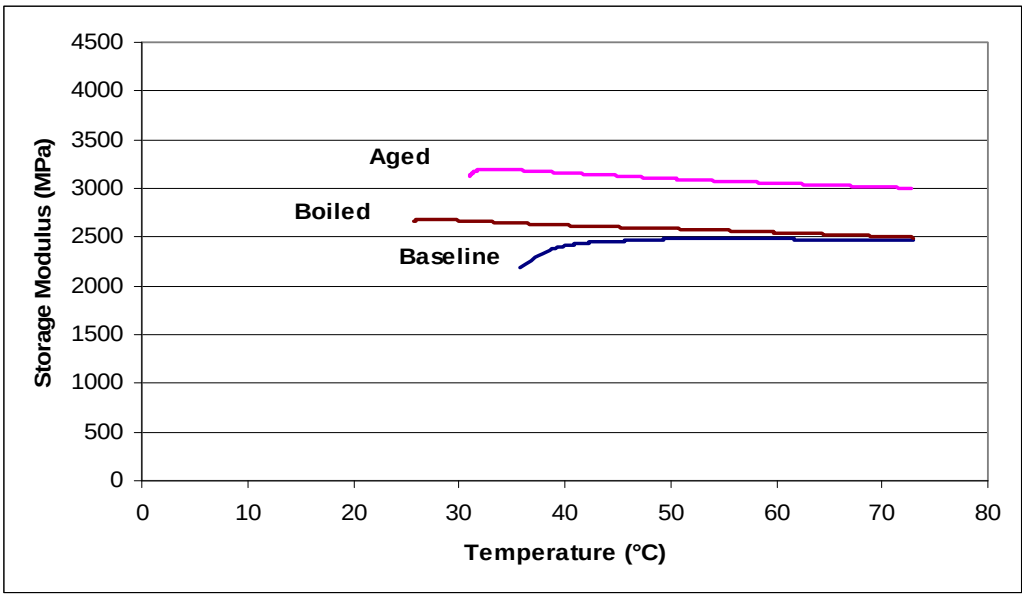

Figure 5. Dynamic Mechanical Analysis of K3B

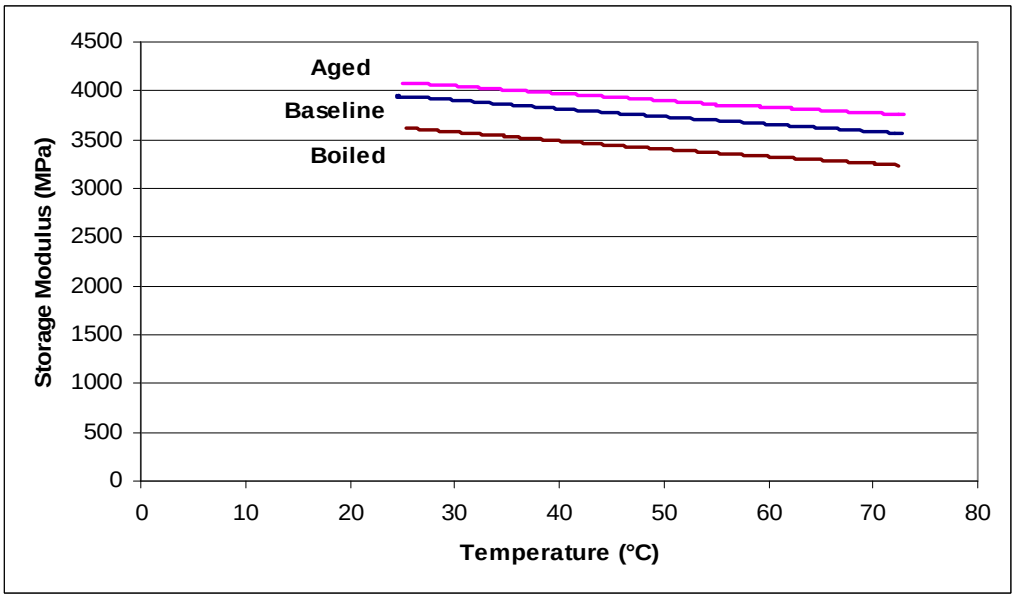

Figure 6. Dynamic Mechanical Analysis of 5260

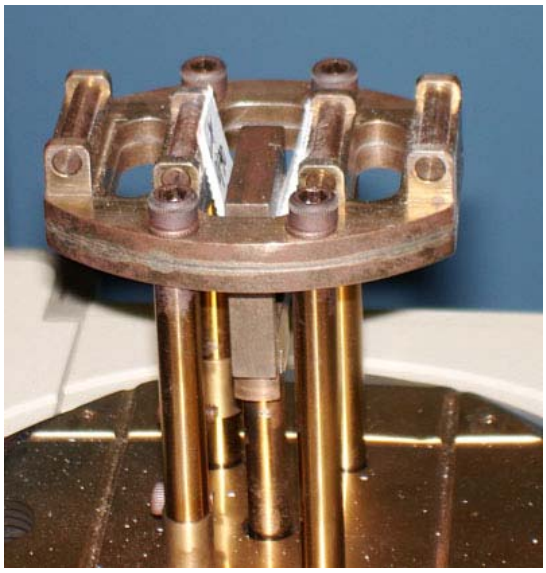

Figure 7. Failed 0.5-in Wide Bend Test Specimen in Dynamic Mechanical Analyzer

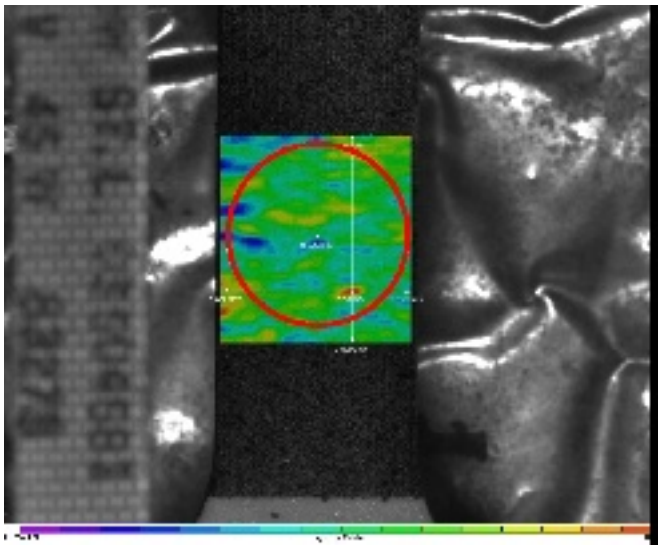

Figure 8. Area Strain Analysis of Captured Visual Image of Closed-Cell Foam

1 = RT Tension to failure
2 = Cool to -325F then Tension to failure
3 = RT Tension to 0.8 Pcr, cool down to -325F, unload to 0.125Pcr, Tension to failure
4 = RT Tension to 0.2Pcr, cool down to -325F, Tension to failure

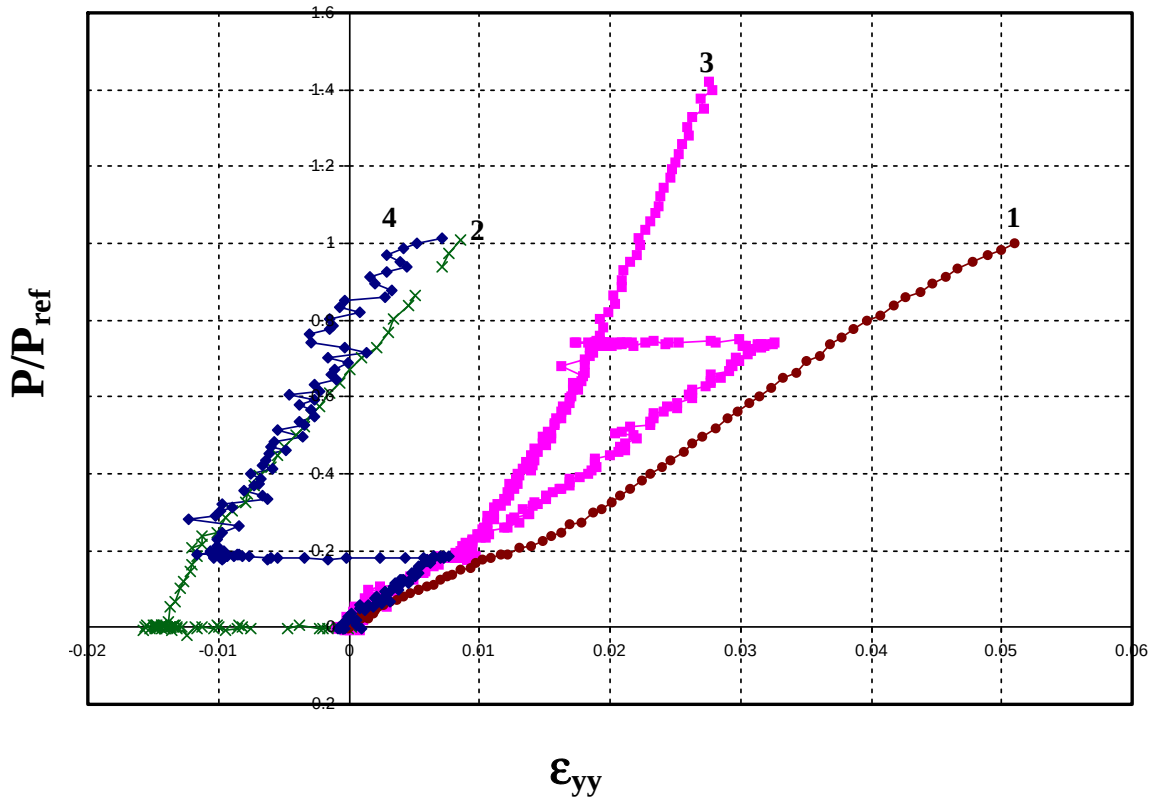

Figure 9. Example plot of Load vs. Strain of Rigid Foam in Various Load Scenarios

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14. ABSTRACT

Various solid polymers, polymer-based composites, and closed-cell polymer foam are being characterized to determine their mechanical properties, using low-load test methods. The residual mechanical properties of these materials after environmental exposure or extreme usage conditions determines their value in aerospace structural applications. In this experimental study, four separate polymers were evaluated to measure their individual mechanical responses after thermal aging and moisture exposure by dynamic mechanical analysis. A ceramic gap filler, used in the gaps between the tiles on the Space Shuttle, was also tested, using dynamic mechanical analysis to determine material property limits during flight. Closed-cell polymer foam, used for the Space Shuttle External Tank insulation, was tested under low load levels to evaluate how the foam's mechanical properties are affected by various loading and unloading scenarios.

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