SANDIA REPORT
SAND2010-6014
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Printed September 2010

Hydrostatic Compaction of Microtherm®HT

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Prepared by
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Abstract

Two samples of jacketed Microtherm[1]®HT were hydrostatically pressurized to maximum pressures of 29,000 psi to evaluate both pressure-volume response and change in bulk modulus as a function of density. During testing, each of the two samples exhibited large irreversible compactive volumetric strains with only small increases in pressure; however at volumetric strains of approximately 50%, the Microtherm®HT stiffened noticeably at ever increasing rates. At the maximum pressure of 29,000 psi, the volumetric strains for both samples were approximately 70%. Bulk modulus, as determined from hydrostatic unload/reload loops, increased by more than two-orders of magnitude (from about 4500 psi to over 500,000 psi) from an initial material density of ~0.3 g/cc to a final density of ~1.1 g/cc. An empirical fit to the density vs. bulk modulus data is $K = 492769\rho^{4.6548}$, where K is the bulk modulus in psi, and ρ is the material density in g/cm^3. The porosity decreased from 88% to ~20% indicating that much higher pressures would be required to compact the material fully.

[1] Microtherm is a registered trademark of Microtherm International Ltd

Acknowledgments

The authors would like to thank Tom Pfeifle and Ronald Lipinski for their critical review of this report. In addition, the authors wish to acknowledge support from DOE/NE-43.

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Introduction

A sheet of Microtherm®HT material, composed of SiO_2 and a binder having an initial porosity and density of 88% and ~0.3 g/cc respectively (under ambient pressure and temperature conditions), was supplied by Sandia's Advanced Nuclear Fuel Cycle Technologies Department (sponsor) for use in the experimental study. The sponsor's desire was to compress the material hydrostatically to full compaction, i.e. to near zero porosity, and to evaluate changes in elastic bulk modulus as a function of density. An experimental method previously developed for foam deformation (Broome and Bauer, 2008) was considered applicable for this study; however before the method could be used, a test system comprising a reaction load frame, pressure vessel and instrumentation needed to be re-assembled and re-calibrated.

System calibrations and two tests were successfully completed under the current study. In each test, Microtherm®HT samples were loaded hydrostatically to a maximum pressure of 29,000 psi. This pressure was not sufficient to fully compact the material but did produce volumetric strains of ~70%, a final density of ~1.1 g/cc, and a final porosity of ~20%. Unload/reload loops were performed during hydrostatic pressurization to acquire data used to determine elastic bulk modulus changes with density increases.

Test Setup, Methods, and Calibrations

Setup/Methods

Samples were prepared from a portion of the Microtherm®HT sheet using a circular die to cut right circular cylinders nominally 1 inch tall by 1.4 inches in diameter. Each die-cut sample was weighed and its dimensions were measured accurately both before and after testing. From these measurements, pre and post test volumes and densities were determined (Table 1). An image of die-cut test sample, MT-H-30-1, is shown in Figure 1.

Each sample was jacketed in two layers of latex prophylactics (Figure 2) to protect the test material from the hydraulic fluid (silicon oil) used during hydrostatic pressurization and the jacketed sample was subsequently placed in a steel pressure vessel (Figure 3) that had been fitted with metal "stuffers." The stuffers served to mostly fill the free volume inside the pressure vessel with nearly rigid material that would otherwise be occupied by the more compressible hydraulic fluid.[2] The pressure vessel was then filled with hydraulic fluid, sealed on top using a steel plate equipped with a central loading piston, and placed in the reaction frame (Figure 4).

[2] Minimizing the volume of compressible fluid in the pressure vessel is advantageous when performing system calibrations needed to infer accurate sample volume changes from total volume changes (system + sample) during testing.

Table 1 Pre and post test physical properties for Microtherm®HT samples

MT-H-30-01				
Initial physical properties				
Length (in)	Diameter (in)	Weight (g)	Volume (cc)	Density (g/cc)
1.001	1.366	7.79	24.040	0.324
Final physical properties				
0.52	1.045	7.81	7.308	1.069
MT-H-30-02				
Initial physical properties				
Length (in)	Diameter (in)	Weight (g)	Volume (cc)	Density (g/cc)
0.990	1.374	8.12	24.055	0.338
Final physical properties				
0.53	1.021	8.11	7.111	1.141

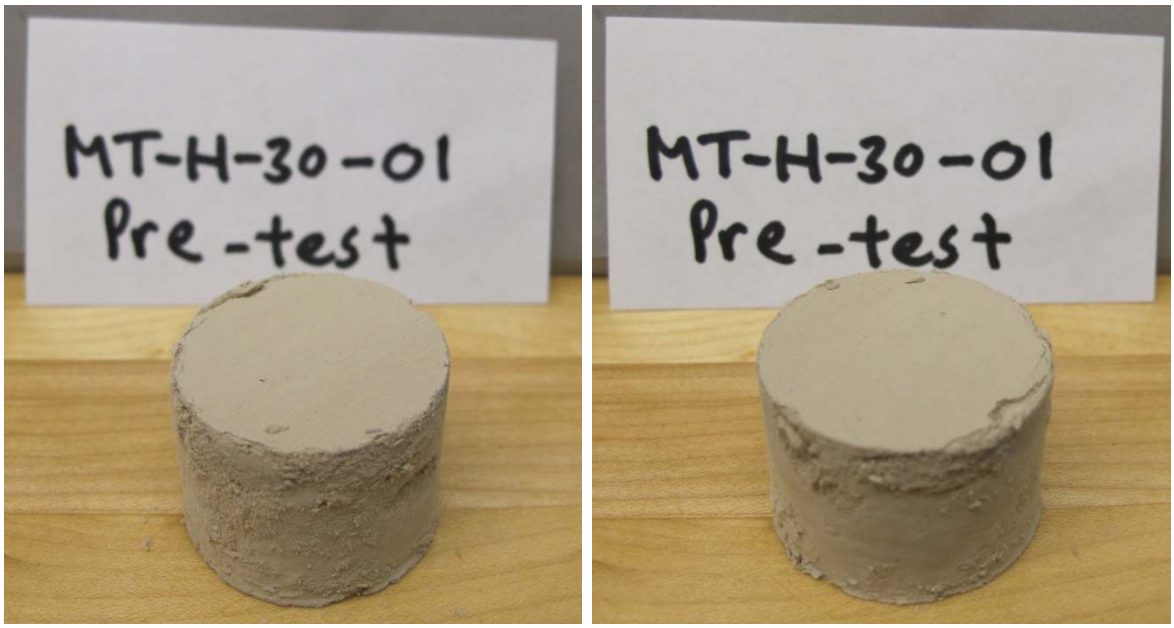

Figure 1 Pre-test images of die-cut sample MT-H-30-01 prior to jacketing.

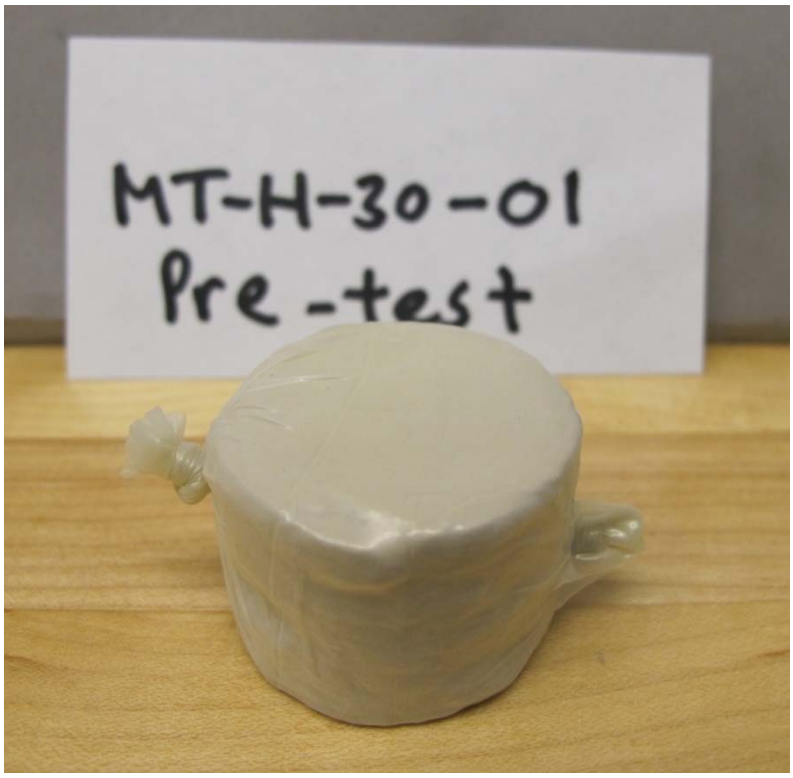

Figure 2 Pre-test image of sample MT-H-30-01 secured within impermeable jacket.

Figure 3 Pre-test image of sample MT-H-30-01 secured within impermeable jacket and inside pressure vessel.

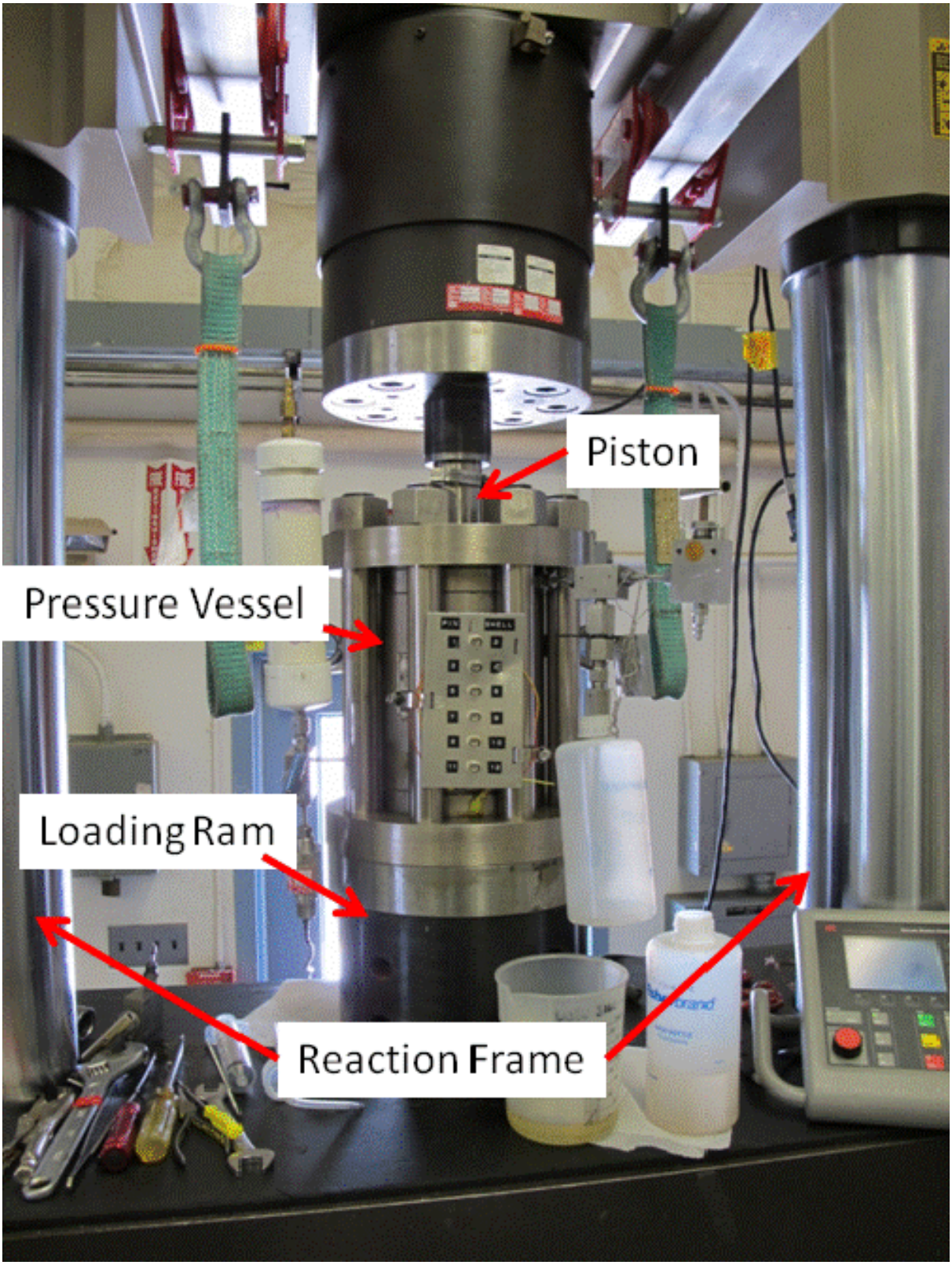

Figure 4 Test assembly showing pressure vessel mounted in the reaction frame and locations of loading ram and piston.

The test is initiated by advancing the loading ram upward to force the top piston down into the pressure vessel. This process is performed slowly to force any entrained air out of the vessel hydraulic ports. The vessel ports are then sealed and further advancement of the ram increases the hydrostatic pressure inside the vessel that then compacts the jacketed sample. At pre-specified pressures throughout the hydrostatic loading, the ram is retracted and then advanced to acquire data from unload/reload pressure loops that are used to determine elastic bulk modulus. Once the maximum pressure is reached, it is decreased to ambient and the test is completed. A pressurization rate of 30 psi/sec was used in the tests both for increasing and decreasing pressures. This test method was successfully followed for both samples MT-H-30-01 and MT-H-30-02.

Volume changes of the samples are determined from the volume changes calculated from the measured displacements of the loading piston (into or out of the vessel) and the piston cross-sectional area and then corrected for test system compliance based on calibration (see discussion below). Engineering volumetric strain is then calculated as the ratio of the sample volume change to the initial sample volume ($\Delta V_s/V_i$). The bulk modulus is determined post-test using the data acquired during the unload loops of the pressure-volumetric strain plots.

Volume Calibration

Pressure changes produced by advancing/retracting the top piston into and out of the pressure vessel result in changes in volumes of the tested sample (ΔV_S), the hydraulic confining fluid (ΔV_{hf}), and the pressure vessel itself (ΔV_V). However because the vessel is sealed such that hydraulic fluid can neither enter nor exit the system, the total volume change, ΔV_T, is zero. Mathematically, then the volume changes can be expressed as:

$$\Delta V_T = \Delta V_s + \Delta V_{ps} + \Delta V_{hf} + \Delta V_V = 0 \qquad \textbf{Equation 1}$$

where ΔV_{ps} is the volume displaced by the piston as it advances/retracts. The last two terms in Eq. 1 represent non-sample deformation (i.e., system deformation) and can be combined into a new term, ΔV_{sys}. With substitution of this term into Eq. 1 followed by some re-arrangement of terms, the sample volume change can be expressed as:

$$\Delta V_S = -\left(\Delta V_{ps} + \Delta V_{sys}\right) \qquad \textbf{Equation 2}$$

In Eq. 2, ΔV_{ps} is determined directly during testing from the piston displacement and its cross-sectional area, but ΔV_{sys} must be determined through independent calibration.

The system volume calibration is performed following the identical test procedure used for the current study, however the Microtherm®HT test sample is replaced by a double-jacketed steel slug (assumed rigid) of the same dimensions as the test samples (Figure 5). During calibration, the steel slug is hydrostatically pressurized following the same load path as that used in the actual tests, including unload/reload loops performed at identical pressure magnitudes as in the actual tests.

The pressure-volume response recorded from a calibration run on the steel slug is shown in Figure 6 (blue curve). Because the steel slug is rigid (no volume change) compared to the compliance of the test system, the volume change of the non-sample test system is identical to the volume change represented by the piston displacement (Eq. 2 with $\Delta V_s = 0$). The pressure-volume response for a test on foam (Broome and Bauer, 2008) is also shown in Figure 6 (pink curve) and includes both the foam sample and the non-sample system response). It is evident from a comparison of the two curves that the pressure-volume response of the foam itself is only a small portion of the overall pressure-volume response and can be isolated by subtracting the calibrated system response (blue curve) from the overall response (pink curve).

The test data acquired for the Microtherm®HT material is interpreted using an identical procedure as that described above. That is, 1) the apparent pressure-volume change response is measured from a hydrostatic pressurization test up to 29,000 psi including unload/reload loops, 2) the non-sample calibrated system pressure-volume change response is subtracted from the measured response to isolate the sample pressure-volume change response, 3) the engineering volumetric strain is calculated as the ratio of the volume change and the initial sample volume, and finally 4) the pressure-volumetric strain response is plotted for each test.

For both the foam testing completed previously and that conducted in this study, the system deformation is much greater than that of the material which leads to potentially larger errors than if the system deformation is relatively small. Using larger Microtherm®HT samples could have alleviated some of this error; however given the imposed time constraint, a test system needing minimal assembly and calibration was selected that necessitated using samples of a relatively small size.

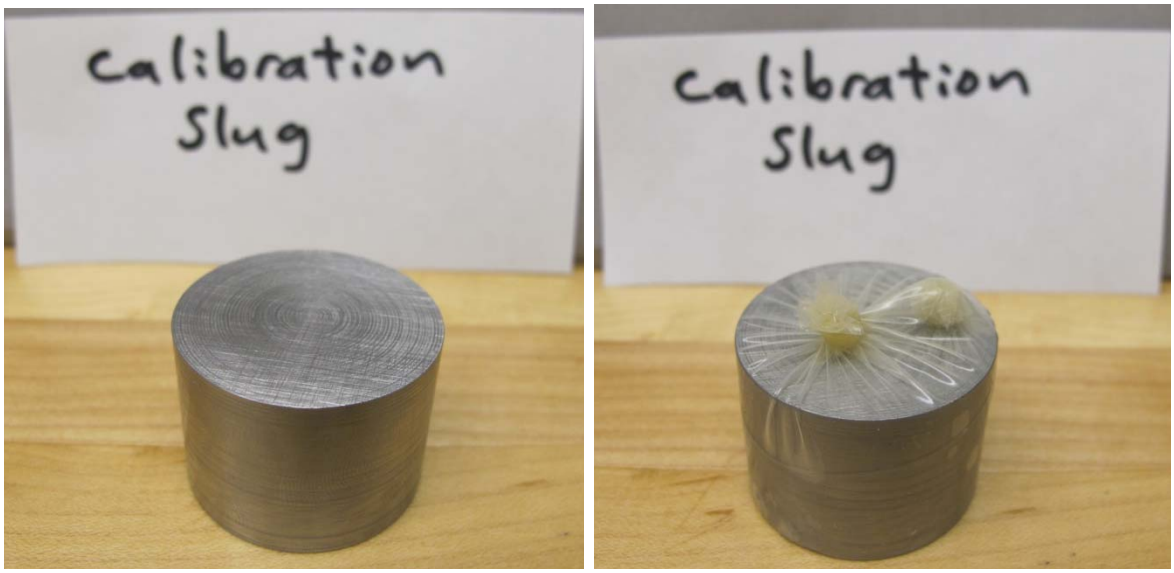

Figure 5 Calibration slug of steel.

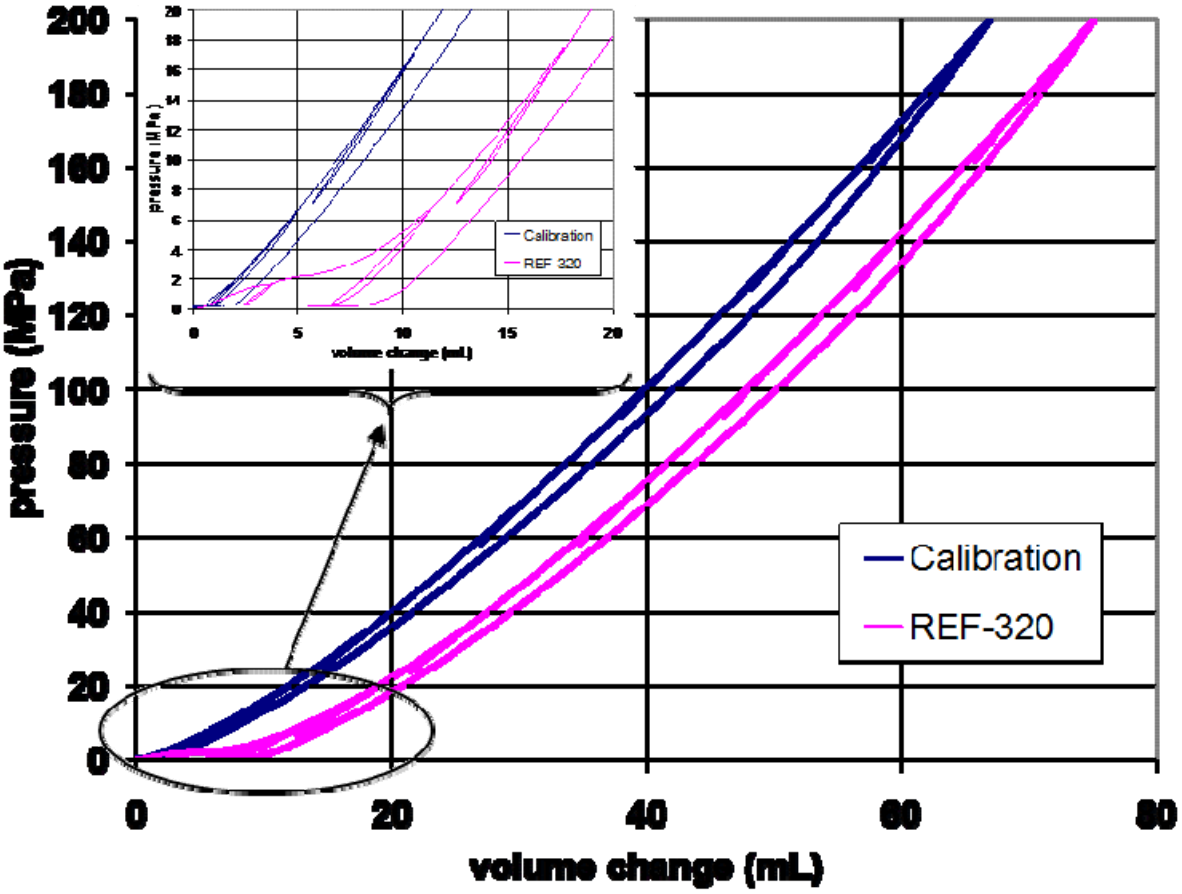

Figure 6 Calibration (blue) and test sample (pink) response for a foam (after Broome and Bauer 2008); Note: 1 MPa = 145 psi.

Experimental Results

Images of deformed samples are shown in Figure 7 (jacketed) and Figure 8 (unjacketed), and exhibit the relatively uniform nature of the compacted deformation. Experimental results for the two tests (pressure versus volumetric strain) are shown in Figures 9 and 10. It is observed that the Microtherm®HT samples begin to compact immediately with application of pressure and that the pressure-volumetric strain response is similar for both tests. The load-unload loops conducted during the course of the test visually demonstrate the stiffening of the Microtherm®HT as it compacts.

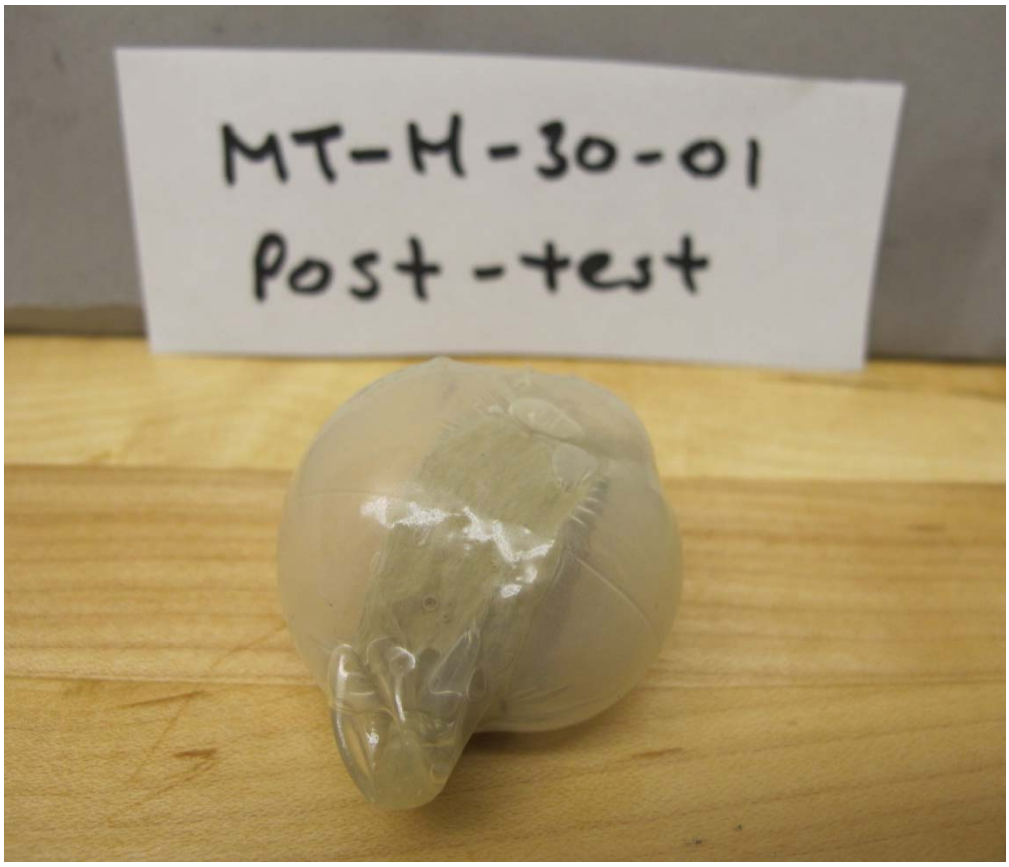

Figure 7 Post-test image of sample MT-H-30-01 in impermeable jacket.

Figure 8 Post-test images of sample MT-H-30-01 removed from jacketing material.

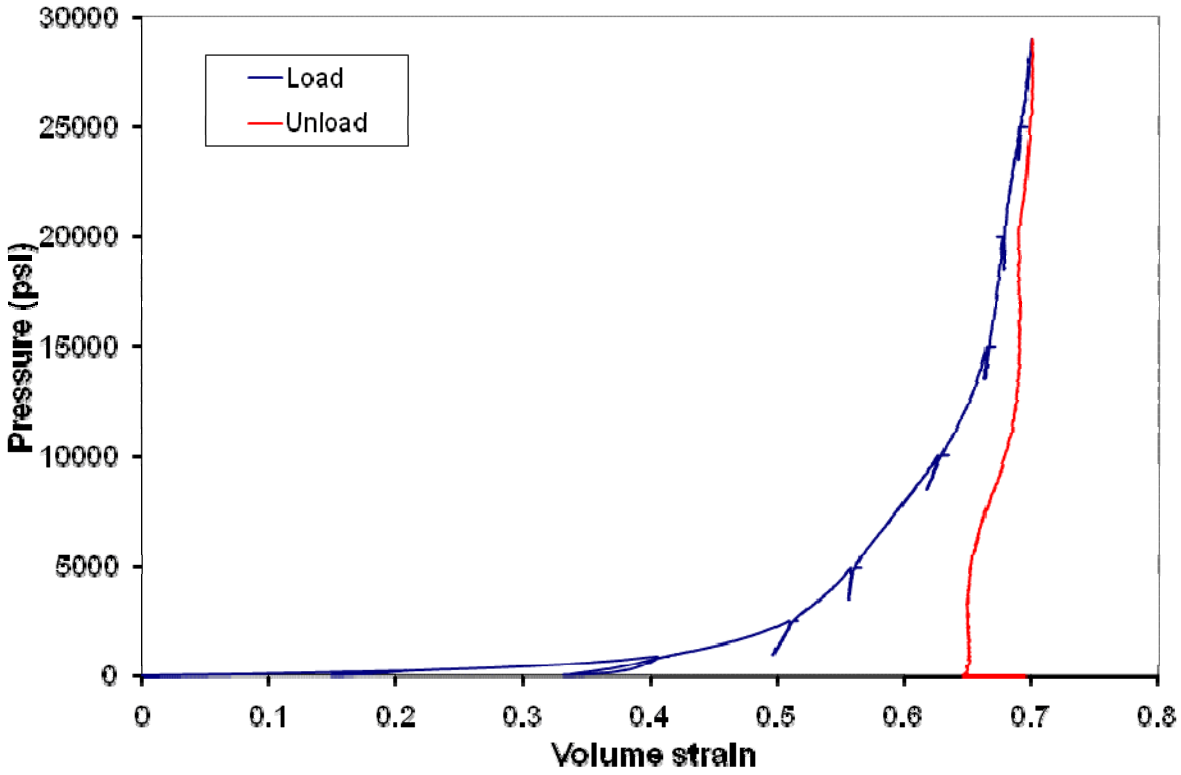

Figure 9 Pressure versus volumetric strain, sample MT-H-30-01.

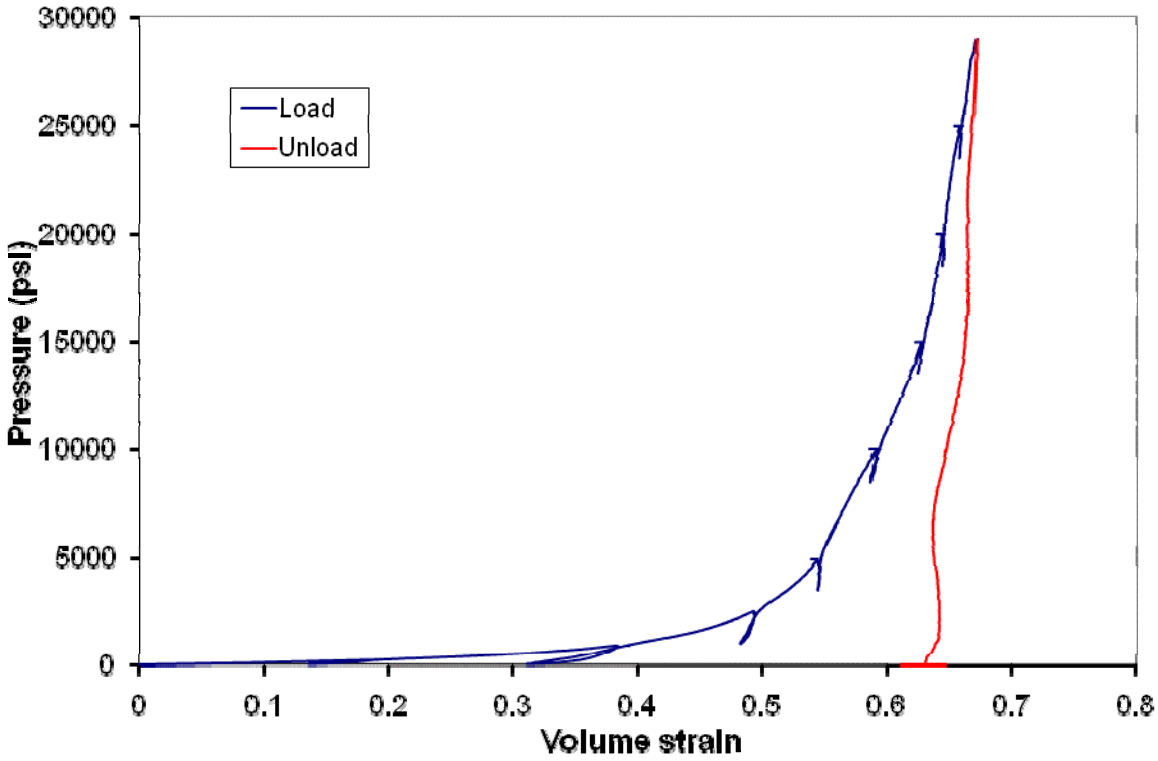

Figure 10 Pressure versus volumetric strain, sample MT-H-30-02.

Test results, including volumetric strain (ε_V), porosity (Φ), bulk modulus (K), and sample density at specified test pressures are presented in Table 2. Porosity is calculated from the initial porosity and volumetric strain (i.e., $\Phi = \Phi_i - \varepsilon_V$). The bulk modulus is calculated from a linear fit to the pressure-volumetric strain data acquired during the unload portion of each unload/reload loop. Table 2 also shows the coefficients of determination (R^2) for these linear fits. Sample density is calculated as the ratio of the sample mass to its current volume. Density, K, and ε_V generally increase with increasing pressure, while porosity decreases with pressure but does not reach zero indicating that higher pressures would be needed to compact the samples fully. Higher pressures could have been applied if a different pressure vessel had been used but set-up and calibration would have required much longer times which could not be accommodated by the current schedule.

The calculated results are presented graphically in Figures 11 through 14. Figures 11 and 12 plot bulk modulus versus pressure and volumetric strain, respectively, and show bulk modulus increases both with increasing pressure and with volumetric strain. Figure 13 plots bulk modulus versus density for all data from both tests, while Figure 14 plots the same data together with a nonlinear empirical fit that relates bulk modulus and density.

Table 2 Test results for Microtherm®HT samples

MT-H-30-01						
Pressure* (psi)	ε_V	Φ	K (psi)	K (MPa)	R^2	Density (g/cc)
0 (pre-test)	0	0.88				0.324**
246	0.206	0.674	4093	28	0.992	0.408
929	0.404	0.476	28809	199	0.995	0.544
2532	0.509	0.371	104541	721	0.988	0.660
4928	0.558	0.322	410075	2827	0.854	0.733
10025	0.628	0.252	152676	1053	0.969	0.871
14970	0.666	0.214	578541	3989	0.658	0.970
19978	0.679	0.201	462133	3186	0.153	1.009
24966	0.692	0.188	457541	3155	0.671	1.052
28964	0.700	0.180	207454	1430	0.101	1.080
0 (post-test)						1.069**
MT-H-30-02						
Pressure* (psi)	ε_V	Φ	K (psi)	K (MPa)	R^2	Density (g/cc)
0 (pre-test)	0	0.88				0.338**
248	0.189	0.691	4759	33	0.981	0.416
926	0.382	0.498	25307	174	0.993	0.546
2541	0.493	0.387	144600	997	0.965	0.666
4944	0.544	0.336	286875	1978	0.466	0.740
10041	0.592	0.288	210754	1453	0.941	0.827
14990	0.629	0.251	331538	2286	0.819	0.910
19986	0.646	0.234	382071	2634	0.209	0.954
24982	0.660	0.220	529110	3648	0.733	0.993
28980	0.672	0.208	503766	3473	0.271	1.029
0 (post-test)						1.141**

* Highest pressure before unloading of sample for bulk modulus determination

** Determined from pre- and post-test sample measurements assuming right-circular cylinder geometry

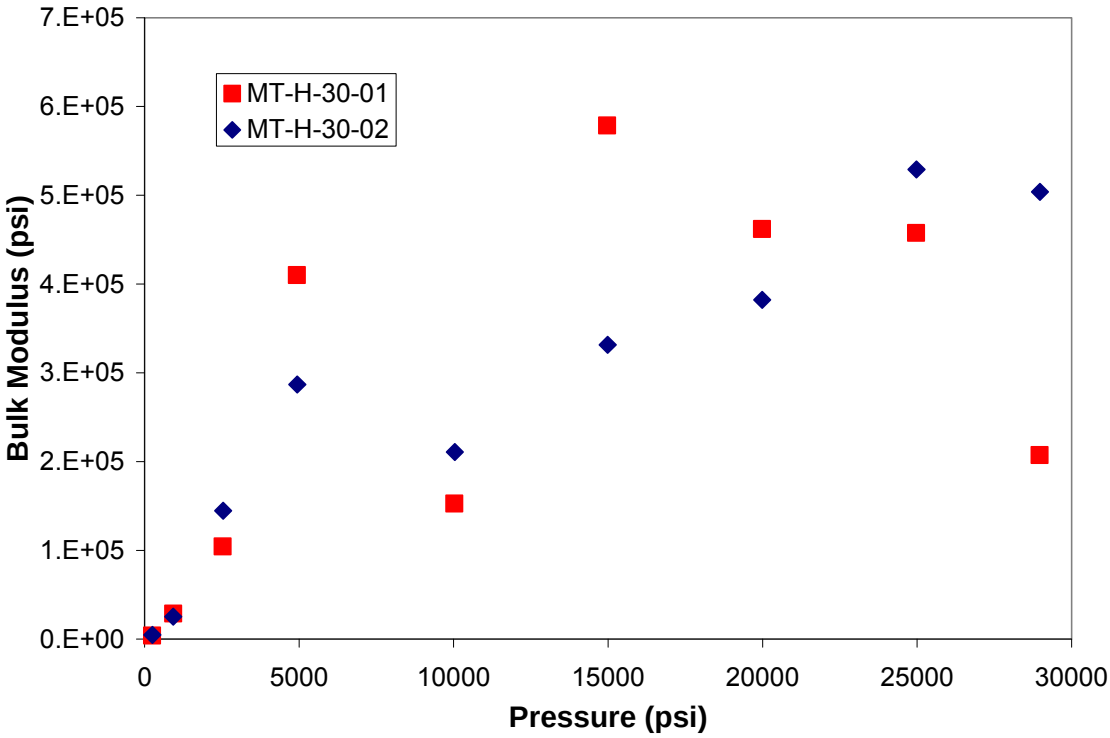

Figure 11 Bulk modulus versus pressure for both Microtherm®HT tests.

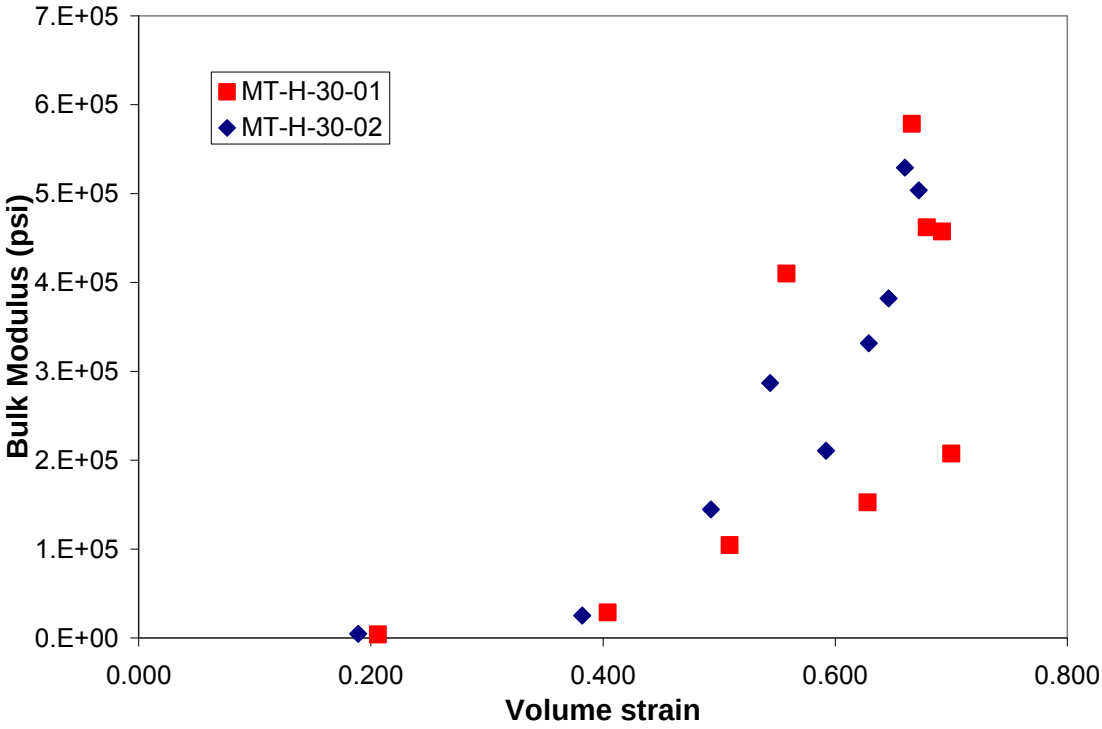

Figure 12 Bulk Modulus versus volumetric strain for both Microtherm®HT tests.

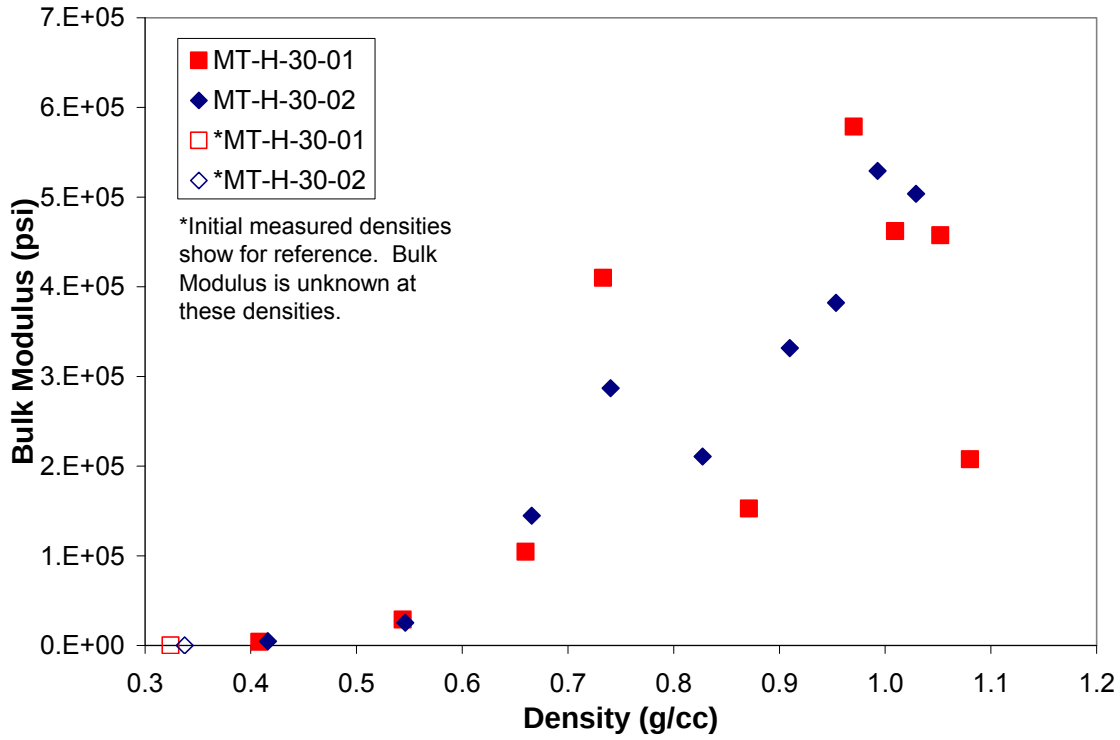

Figure 13 Bulk Modulus versus density for both Microtherm®HT tests.

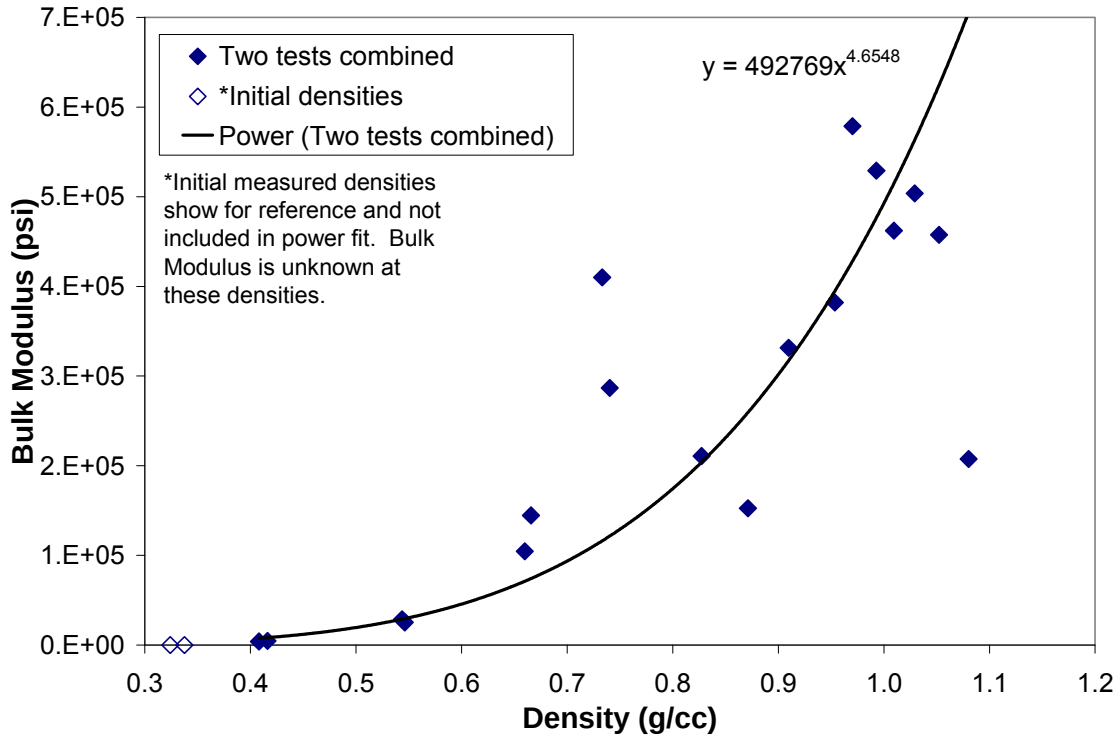

Figure 14. Bulk Modulus versus density for both Microtherm®HT tests together with curve fit.

Summary

Two samples of jacketed Microtherm®HT having an initial porosity of 88% were hydrostatically pressurized to maximum pressures of 29,000 psi to evaluate both pressure-volume response and change in bulk modulus as a function of density. During testing, each of the two samples exhibited large irreversible compactive volumetric strains with only small increases in pressure; however at volumetric strains of approximately 50%, the Microtherm®HT stiffened noticeably at ever increasing rates. At the maximum pressure of 29,000 psi, the volumetric strains for both samples were approximately 70%. Bulk modulus, as determined from hydrostatic unload/reload loops, increased by more than two-orders of magnitude (from about 4500 psi to over 500,000 psi) from an initial material density of ~0.3 g/cc to a final density of ~1.1 g/cc. The final porosity achieved at the maximum pressure of 29,000 psi was only ~20% indicating that much higher pressures would be needed to compact the material fully.

References

Broome, S.T., and Bauer, S.J., unpublished (2008).

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