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Author(s): Thompson, Darla G.

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memorandum

Weapons Experiments Division
High Explosives Science & Technology: WX-7

To/MS: Daniel J. Trujillo, W-15, dxt@lanl.gov
From/MS: Darla G. Thompson, WX-7, dkgraff@lanl.gov
Phone/Fax: 7-0840/Fax 7-0500
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SUBJECT: PBX 9502 CREEP DATA, COMPRESSION AND TENSION

This is a summary of the constant-load, constant-temperature mechanical creep data that has been collected on PBX 9502 in tension and compression over the last 5+ years. This work was primarily funded by the Enhanced Surveillance Campaign (C-8).

This document is intended to accompany the specific data files to aid with data interpretation. This is especially true for compression, where a careful zeroing process must be performed on the Heidenhain strain channel in order for the data to be meaningful. In addition, a preliminary analysis is presented in which the secondary creep curves are characterized and plotted, highlighting empirical relationships that 1) clarify regions of parameter space most interesting to explore next and 2) lay the foundation for the parameter space and relationships that will need to be captured in a PBX 9502 creep model.

This report and these data will be provided to Nathan Miller, W-13, for his analysis towards creep model development.

DT:mq

Cy:
Thomas Zocco, W-DO, zocco@lanl.gov
Sheldon Larson, WX-7. larson@lanl.gov
Nathan Miller, W-13, nathanm@lanl.gov
Miles Buechler, W-13, buechler@lanl.gov
Bob Stevens, W-13, stevens@lanl.gov
Geoff Brown, WX-7, geoffb@lanl.gov
Racci Deluca, WX-7, rdeluca@lanl.gov
Richard Browning, W-13, rvb@lanl.gov
D J Luscher, T-3, djl@lanl.gov
Matt Lewis, W-13, mlewis@lanl.gov
Eric Mas, W-13, mas@lanl.gov
DCM File, MS P942

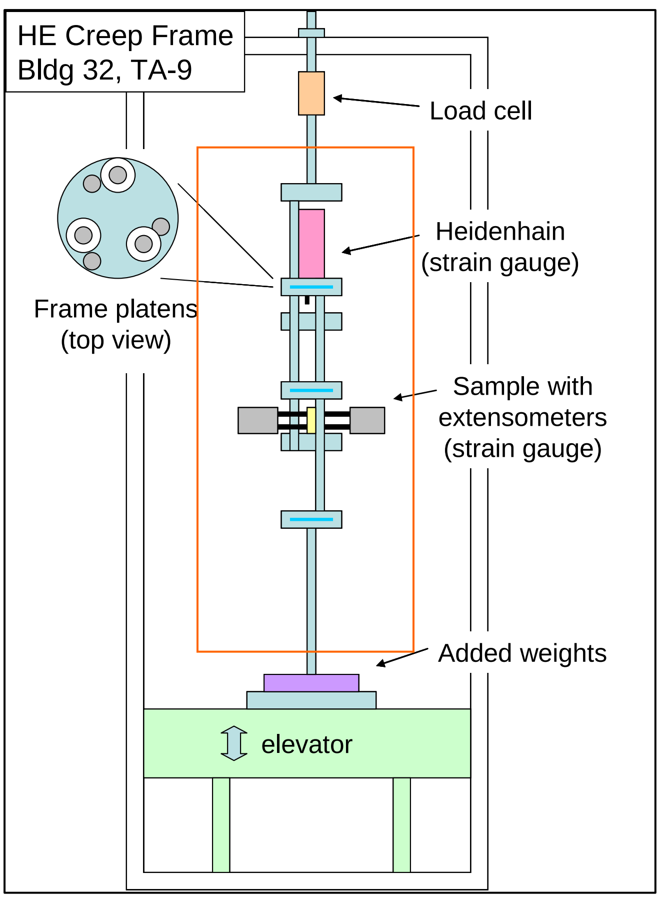
PBX 9502 Creep Data, Compression and Tension
File description, experimental information, and analysis; funded by C-8
Darla Graff Thompson, February 2014
Racci DeLuca and Geoff W. Brown
WX-7, LANL, dkgraff@lanl.gov

I - PBX 9502 (Virgin) Compressive Creep Data from Long-Term Creep Frame

The Virgin PBX 9502 lot used in this study was 890-019. Specimens were machined from a stockpile-return charge, isostatically pressed. Specimen geometry was 0.375-inch diameter by 1.125-inch long, l/d=3.

The long-term creep frame was designed by Richard Browning (ESA, LANL, now retired) and assembled in 2003/2004. The frame was moved several times to different buildings at TA-9 for various reasons, always in search of a better facility due to power or temperature issues. The frame was finally set up in Bldg 32 of TA-9 and data was acceptable. The PBX 9502 compressive creep data given here were collected in 2010 through 2013.

A rough diagram of the load frame is shown below, Figure 1. An orange line indicates the thermal box used to control the temperature. This thermal box was constructed of 2” Styrofoam and had enough room between the walls and the load train



for 1 to 2 feet of wound copper tubing that entered and exited through a port in the back of the box to a Julabo temperature-controlled water bath. The tubing also ported in and out of the two platens directly above and below the specimen (see diagram on left, Figure 1) to help maintain constant temperature at this location. A small muffin fan is mounted in the top of the thermal box to circulate air and to help maintain uniform temperature, minimize gradients.

The concept of the load fixture is that it comes as two, interleaved halves. The first half is comprised of three plates, between each pair of plates are three rods that connect them firmly. The second half is comprised of three other plates, also with plate pairs connected by three rods. The central plates have holes cut (top view of plate on left side of drawing) that allow the two plate assemblies to be interleaved, sliding through each other as the specimen is compressed. Plates and rods are made of Invar which has a very low CTE. Kirk Weisbrod (AET, LANL) made a copy of this design and put some kind of bushings or bearings at the interface between the plates and rods; we learned in a round robin study with him (9501 compression) that this did result in a slight decrease of load that was applied to his specimen. In our system, there is a gap between the rods and the holes in the plates. If the specimen fails non-uniformly then the rods could rub on the plates. We believe this doesn't happen, but our load measurements wouldn't likely detect it if it did.

Data Analysis

Here is an example file of compressive creep on stand-alone creep frame in Bldg 32 at TA-9, file 040412a read into Excel:

DATE	TIME	Seconds	T1	T2	T3	T4	T5	T6	T7	25 lb cell	100 lb cell	mini 100 lb cell	Heidenhain	Extens 7	Extens 8	10 V Supply	5 V Supply	Batt reference
4/4/2012	########	1	21.10813	50.5721	49.87058	46.30699	44.54132	44.83349	47.17183	-1.2029E+41	-14.2500864	-143.7322107	-0.0000012	0.0023287	0.0014924	-10.015391	4.9718603	3.107405
4/4/2012	########	2	21.1048	50.56872	49.87058	46.30699	44.53456	44.83686	47.17521	-1.2029E+41	-14.2415748	-29.7705134	-0.0000014	0.0023338	0.0014915	-10.015378	4.9718472	3.107405
4/4/2012	########	4	21.09811	50.56195	49.86718	46.30699	44.53456	44.8301	47.16846	-1.2029E+41	-14.2132954	-353.0158755	-0.0000018	0.0023321	0.0014907	-10.015417	4.9718735	3.107392
4/4/2012	########	5	21.09811	50.57887	49.87058	46.31375	44.53793	44.83686	47.17183	-1.2029E+41	-14.1719421	-250.4980016	-0.000002	0.0023338	0.0014933	-10.015417	4.9718735	3.107392
4/4/2012	########	6	21.09475	50.56195	49.87058	46.32052	44.53456	44.83349	47.16507	-1.2029E+41	-14.2041387	-4.0883022	-0.000002	0.0023346	0.0014924	-10.015391	4.9718735	3.107392
4/4/2012	########	7	21.09475	50.57547	49.86718	46.32728	44.53117	44.83349	47.16846	-1.2029E+41	-14.2914399	-1.0274996	-0.0000018	0.0023279	0.0014942	-10.015391	4.9718603	3.107392
4/4/2012	########	8	21.08808	50.56195	49.86718	46.32728	44.53456	44.83349	47.16169	-1.2029E+41	-14.2835262	-113.4131029	-0.0000018	0.0023321	0.0014933	-10.015417	4.9718472	3.107392
4/4/2012	########	9	21.08808	50.57547	49.86718	46.34079	44.53456	44.83686	47.16846	-1.2029E+41	-14.2540087	-48.6261588	-0.0000014	0.0023338	0.0014951	-10.015404	4.9718472	3.107392
4/4/2012	########	11	21.08139	50.58565	49.85703	46.36109	44.54132	44.83686	47.16507	-1.2029E+41	-14.2257662	-15.7068059	-0.000002	0.0023346	0.0014951	-10.015417	4.9718209	3.107379

The last three columns (10 V and 5 V Supply and Batt reference) were just to keep an eye on the power supplies for the data channels, in case there were unexplained problems. I have not used these in any of my analysis. Also, I have ignored the first two columns of date and time, using use third column (seconds) for time correlation of data.

Next, seven thermocouples (TCs) were installed at various points inside the thermal box and recorded during the tests. TC1 is outside the box. TC2 and TC3 were mounted on the upper and lower specimen platens and are specifically relevant to the specimen temperature during the test. Some tests controlled temperatures better than others (see Figure 5) and this should be taken into account when data are analyzed (some ran hot, some had unplanned variability during the test).

The primary means of measuring creep strain in the specimen is the Heidenhain strain gauge which is a bit like an LVDT with a single arm extended downward from the mounting platen to a mobile platen (the mobile platen moves relative to the mounting platen as the specimen creeps, see diagram). The Heidenhain was selected for this work because of its high precision and long-term stability. Before the elevator is lowered (placing the load on the specimen), the Heidenhain arm is extended until it pushes on the mobile platen with a very small force controlled via feedback. As the elevator is lowered and the load is applied, the Heidenhain captures the motion. There is nothing in the Heidenhain displacement that indicates the exact zero-point of the specimen, when the specimen is contacted. This is the reason for the two, oppositely-mounted knife-edge extensometers. As strain measuring devices, these do not work well over the long haul; they drift (likely creep of their own weight) and there are many examples in this dataset where the rubber bands used for mounting them break and extensometers literally fall off the specimen. Analysis of the loading portion of the curves (first 50 to 100 data points) is performed as follows, using extensometers to zero the Heidenhain strain data:

The Extens7 and Extens8 data columns are averaged and an amount added or subtracted to make the initial values equal to 0. These columns have units of displacement in INCHES and so next we need to calculate STRAIN using the extensometer gauge length of 0.5 inches.

AveExtensometerStrain = (AveExt7&8 - zeroing) / 0.5

Next we convert the output of the Heidenhain, also in INCHES of displacement, into strain. This signal spans the full length of the specimen (1.125 inches) and also comes out as negative, so we have:

AveHeidenhainStrain = Heiden/1.125*-1

Now, plot both of these strain curves versus time, and zoom in so you are seeing the first HOUR of applied load. Add or subtract from the AveHeidenhainStrain data until it overlaps best with the AveExtensometerStrain. The AveHeidenhainStrain data is now zeroed. Typically, the two strain measurements will overlay quite well initially (first hour), but eventually they deviate and the Heidenhain data is the one to keep.

It is important here to comment about the load channels. There is a data column entitled “25 lb cell” and this is garbage, as is the data column “mini 100 lb cell” (the former was to be in the load train between the frame and the load, the latter was to be sandwiched between the loading platens, in contact with the specimen--- neither of these was used at all). This leaves the one meaningful load cell, column “100 lb cell,” which is mounted at the top of the load train. If you look at the early loading times, you see that the loading of the load cell has slightly different timing than the strain registered on the extensometers. Using the extensometer data to zero the Heidenhain is the most accurate process, and in our 2008 round robin creep testing of PBX 9501, the three different researchers agreed with this procedure (Cunningham from LLNL and Weisbrod from AET, LANL and myself).

Table 1:

Compressive Creep PBX 9502 Virgin (FY12 and FY13)

Load	5 MPa (725 psi)	4 MPa (580 psi)	3 MPa (435 psi)
Weights	291 N (65 lbs)	220 N (49.5 lbs)	149 N (33.5 lbs)
50°C	040412a(10) 060712a(8)	101410a 030311a(6)	080812/082912 022113a
40°C	030112a(4) 051412a(5)	020711a 030211a** 050211a/051711a 110310a	101712 121812

The load calculations, Table 1, take into account the weight of lower half of frame + hanger + weights. Also in Table 1, in the case of double filenames – data collection was stopped and started again immediately, without unloading/reloading the specimen.

The graph of zero-corrected Heidenhain data is shown in Figure 2. We have at least two tests in each test condition, giving us some confidence in our reproducibility. There is a reasonable trend in the strain response with temperature, and a reasonable trend with applied load (given as an engineering stress).

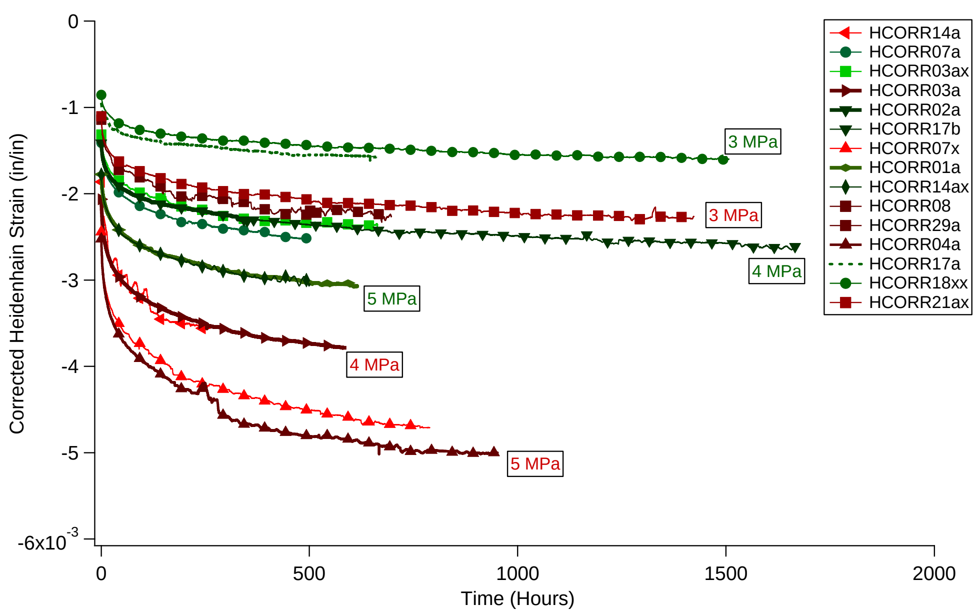


Figure 2: Zero-corrected Heidenhain strain data (see legend for specimen ID, numbers in legend correspond to the third and fourth digits in the filename from Table 1).

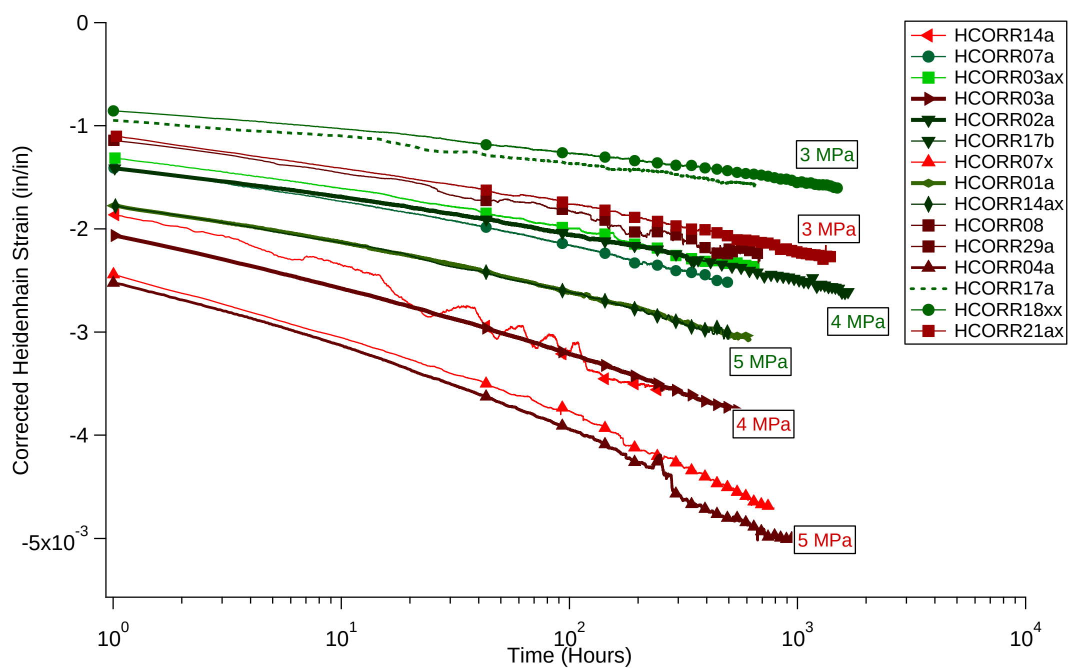


Figure 3: Same as Figure 2 but plotted on a log-scale Time axis.

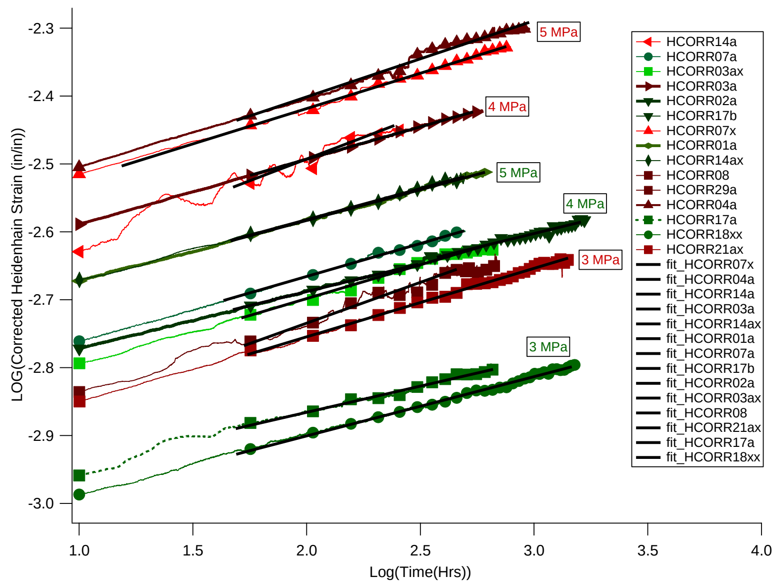


Figure 4: Same data as in Figures 2 and 3 but now on a log-log plot with "best-fit" least-squares lines through each curve.

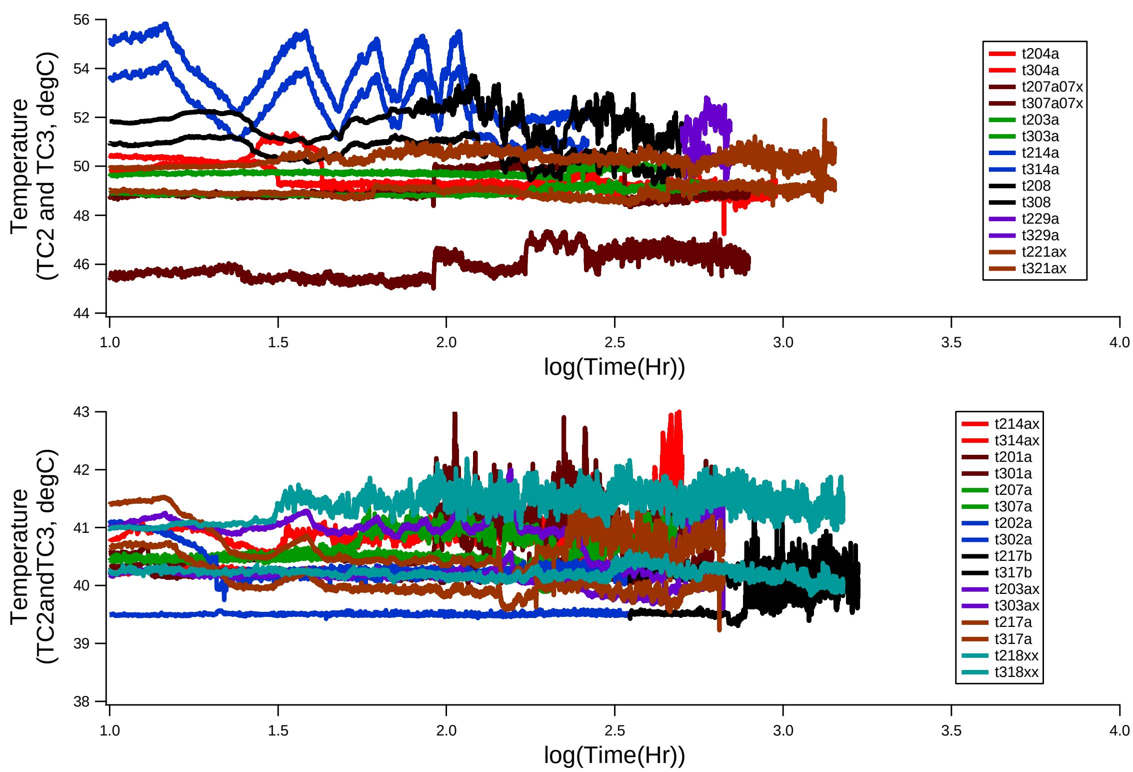


Figure 5: TC2 and TC3 outputs for all tests at 50°C (top) and 40°C (bottom); the effect of poor temperature control can often be seen by comparing TC output with corresponding strain data in Figure 4.

Table 2: Linear fitting results of log-log plots, Figure 4 (slope units strain(%)/time(sec))

Temp (°C)	Filename	Stress (MPa)	slope	y-intercept	Stress ratio	Density (g/cm^3)
50	040412a(04a)	5	0.1120	-0.2317	0.360	1.8958
	060712a(07x)	5	0.1040	-0.2602	0.360	1.8966
	030311a(03a)	4	0.0950	-0.3452	0.290	1.8960
	101410a(14a)	4	0.1280	-0.2938	0.290	1.8948
	080812/082912(08)	3	0.1203	-0.5472	0.210	1.8939
	022113a(21ax)	3	0.1009	-0.5978	0.210	1.8958
40	051412a(14ax)	5	0.0916	-0.4393	0.290	1.8950
	030112a(01a)	5	0.0907	-0.4426	0.290	1.8952
	020711a(07a)	4	0.0968	-0.4558	0.235	1.8970
	050211a(02a)/(17b)	4	0.0820	-0.5606	0.235	1.8933
	110310a(03ax)	4	0.1000	-0.5437	0.235	1.8963
	101712(17a)	3	0.0772	-0.7459	0.177	1.8958
	121812(18xx)	3	0.0873	-0.7648	0.177	1.8967

II - PBX 9502 (Virgin and Recycled) Tensile Creep Data from Instron 5567

For these tests, both Virgin and Recycled PBX 9502 processing methods were included. Recycled specimens came from lot 891-006, isostatically pressed. Virgin specimens came from lot 891-008. Both materials were isostatically pressed at LANL as the hemispherical mold type A42. Specimens are 3-inch long dogbones with a gauge length of 1.5 inches; the diameter of the gauge section is 0.5 inches.

All tensile creep tests were run on the Instron 5567 (TA-9, Bldg 33). This instrument is equipped with a Bemco environmental chamber for temperature control and dehumidification. The liquid N_2 Dewar size was the main limitation on our creep test duration. Strain was measured using two oppositely-mounted knife-edge extensometers with a gauge length of 1.0 inch. The primary reason we could not run tensile creep tests on our creep frame is that the Instron has a “break detection” feedback system that detects when the specimen load shows a sudden and significant drop, indicating failure of the specimen; the system then immediately stops any further crosshead motion so that the extensometers will not be pulled beyond their safe operational displacement.

Table 3 is the test matrix for our tensile creep. Filenames are given along with test conditions such as temperature and stress. Other relevant test results are given such as time to failure and stain at failure (or maximum strain in the tests where specimens did not fail). For these tests, log(strain) vs. log(time) plots of some of the tests were made, see Figure 6. For these tests, we are primarily interested in quantifying the secondary “linear” part of the creep curve, and to this end, least-squares fits were made. Slopes and y-intercept values are listed in Table 3 (note that not all of the data sets are plotted or were fit). There is a fair amount of scatter in the data--- more than we expect to see in uniaxial quasi-static tension or compression. Creep data is known to be sensitive to flaws or microstructural differences [3]. Within the experimental scatter, if one focuses on the averaged numbers, there are clear trends that appear as a function of creep stress and temperature. In Figure 7 we plot creep stress and tensile strain at failure (or maximum strain for those specimens which did not fail) versus log failure time. Note that tensile strain at failure is pretty much the same for all temperatures and stresses, between 0.15 and 0.2 %. Tensile failure strain in quasi-static tests is also quite independent of temperature and strain rate, with values between 0.22 and 0.26% [1].

Table 3: Recycled and Virgin PBX 9502 Tensile Creep on the Instron 5567

Temp (°C)	Filename	Stress (MPa)	Stress Ratio	Time to Failure (hrs)	Max Strain (%)	Slope	Intercept	Density (g/cm^3)
50	021710a/19a	2.00	0.38	137*	0.159	0.1152	-1.4591	1.8928
	053111a			309*	0.191	0.1082	-1.3607	1.8913
	082008a	2.63	0.50	1.0*	0.206			1.8911
	081908a/b			1.18	0.152			1.8911
	081108b			10.0*	0.192			1.8919
	081908c			19.3	0.215	0.1291	-1.3052	1.8926
	022310b			7.1	0.192			1.8921
	081208a/b	2.98	0.56	0.1	0.131	0.1029	-1.148	1.8912
	081808c			0.22*	0.203	0.203	-1.283	1.8909
	041811a			1.64	0.184	0.128	-1.252	1.8932
	042711a			1.19	0.194			1.8964
	081108a	3.42	0.65	0.25*	0.191			1.8916
	022310a			0.20	0.188			1.8929
	022410a			0.16	0.177	0.1150	-1.092	1.8919
	041111a			0.18	0.202	0.1633	-1.151	1.8929
40	102110a	2.63	0.38	168	0.156			1.8937
	020811a			216*	0.160	0.0928	-1.339	1.8927
	013111a			76*	0.147			1.8926
	042711b			23	0.133			1.8964
	042811a			428*	0.172	0.0916	-1.330	1.8906
	110110a	2.98	0.43	103	0.187			1.8922
	111510a			350*	0.208	0.1001	-1.296	1.8939
	041811b			105	0.163	0.095	-1.335	1.8938
	110810a	3.42	0.49	30.5	0.208	0.1051	-1.224	1.8930
	120210c			8.8	0.193			1.8959
	040711a			22.6	0.224			1.8932
30	120710a	3.42	0.38	341*	0.142	0.0760	-1.320	1.8931
	010411a			264	0.179			1.8938
	120610a	5.00	0.56	1.49	0.170			1.8931
	012711a			3.3	0.181	0.0872	-1.105	1.8929

* Did not fail, test stopped manually.

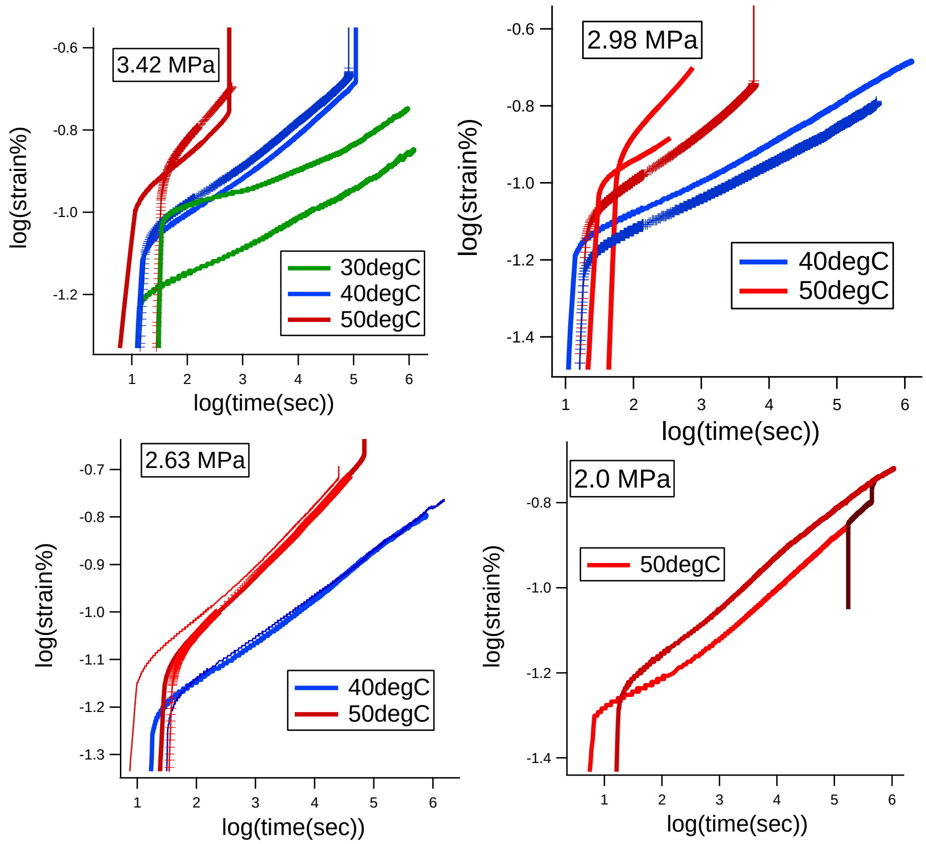


Figure 6: Example PBX 9502 tensile creep curves from Table 3 showing the effects of temperature and creep stress.

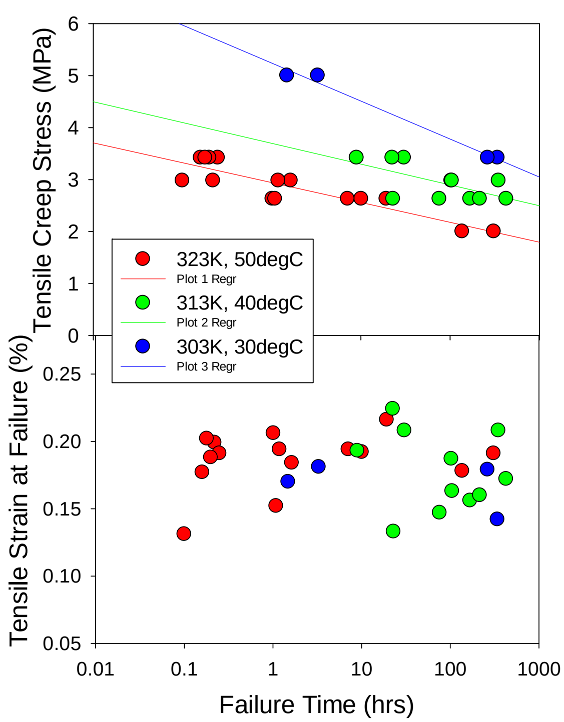


Figure 7: Plots of Creep Stress and Tensile Strain at Failure (or Maximum Strain for those specimens which did not fail) versus Failure time on a log scale. Note that Tensile Strain at Failure is quite invariant at all stresses and temperatures.

Figures 8 and 9 show slope and y-intercept values from the secondary creep curve for both tension and compression. (Note that to put tensile and compressive log-log plot parameters on the same graph, the log-log plots must be made on axes with the same strain and time units, which we have done.) Figure 8 shows these fitting parameters as a function of temperature, while Figure 9 shows them as a function of stress. See legends for symbol identification.

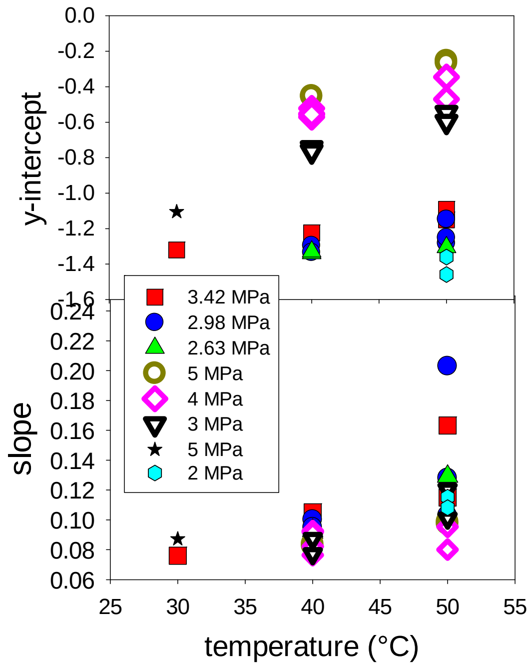


Figure 8: Fitting parameters (slope and y-intercept) plotted versus test temperature; open symbols are from compression data, solid symbols are tension. See legend for stresses.

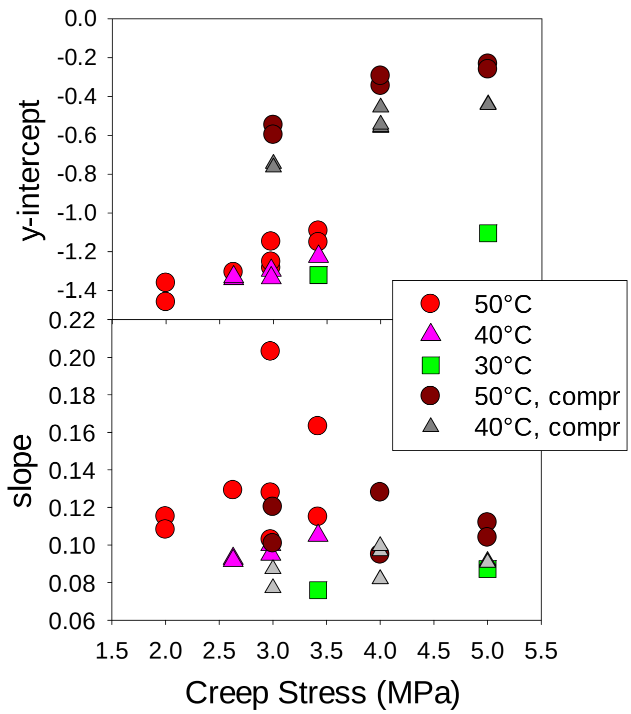


Figure 9: Fitting parameters (slope and y-intercept) plotted versus creep stress; see legend for symbol identification and temperatures ("compr" indicates compression data).

The purpose of Figures 7, 8 and 9 is to try to establish trends as a function of test parameters. To the extent this can be done reliably with the data we have, we can use it to 1) establish an empirical model and 2) interpolate and possibly extrapolate creep response to conditions somewhat different than those we have actually measured. Inspection of Figures 7 thru 9 show that the trends are reasonable and consistent and should be useful in building a model. However, see below, we propose that Figures 10 and 11 are the best for showing consistency and establishing data trends.

For this next step of analysis, instead of simply considering the creep stress of any given test, we consider the stress ratio [2]. Stress ratio is defined as being the ratio of the creep stress (i.e. the stress applied during the creep test) to the ultimate stress of the material at these loading conditions (specifically at this temperature and this strain rate of loading). Using the results of time-temperature superposition [1] we calculate the stress ratio for each of the conditions, and these values are included in Table 2 and 3. In Figure 10, we plot the linear fitting parameters once again but this time versus stress ratio. As stated earlier, there is some variation in the fitting parameters, particularly the slope values at 50°C in both tension and compression, however, if one eyeballs an average response at each set of unique conditions, there are some very useful observations that can be made. Looking at the y-intercept versus stress ratio plot we see that all the compression data clusters together, all the tensile data clusters together; the y-intercept value relates to the strain reached during the loading process… and it is exactly what we expect (based on quasi-static data) that at the same stress ratio, compressive strain will be larger than tensile strain (ultimate compressive strength is larger by a factor of 3 than ultimate tensile strength). Interestingly, the slope values for tension and compression all appear to fall on a common trend line showing an increasing slope with increase in stress ratio. These observations point us in useful directions as to what conditions should be tested next, to determine if these trends hold true over a wider range of parameters.

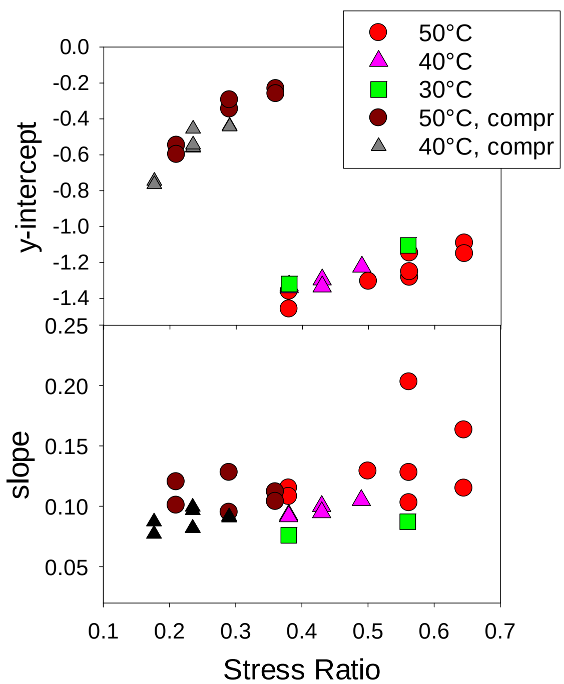


Figure 10: Fitting parameters (slope and y-intercept) plotted versus stress ratio; see legend for symbol identification and temperatures ("compr" indicates compression data), the other data are tensile.

Figure 11 is a repeat plot of Figure 7, except instead of plotting tensile creep stress versus failure time in the upper plot, we have plotted tensile stress ratio versus failure time. Similar to what was shown in Figure 10, by plotting creep stress information as stress ratio, the data appear to fall on the same trend line, with higher stress ratios giving rise to shorter failure times (as one would expect). By "normalizing" creep stress (i.e. dividing by the ultimate stress at that condition), we have reduced an aspect of the creep response (failure time) at different temperatures to a common curve.

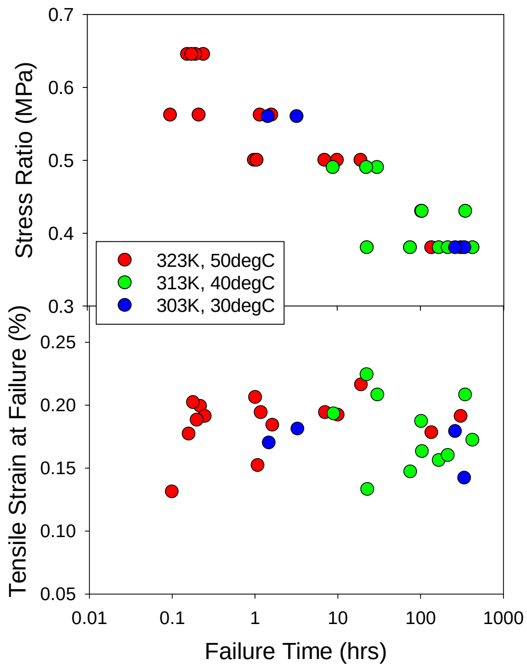


Figure 11: Repeat plot of Figure 7, however this time showing tensile stress ratio plotted versus failure time in the upper graph.

Summary

Creep tests on PBXs are inherently difficult. Strains are small and temperature control must be rigorous over an extended period. Small microstructural differences (PBX heterogeneities) are known to affect creep data and cause variation in the measured results [3]. Nevertheless, with the modest set of compressive and tensile creep data reported here, we believe we have the basis for understanding creep response in this material and for outlining a creep model with appropriate parameter dependencies. These PBX 9502 data include both Recycled and Virgin processing methods; careful analysis of the data may show that the creep response of the two processing types is statistically different, however, we have chosen to put both types on the same plots with the assumption that processing type is not a strong factor in determining creep response. We are aware of some PBX 9502 creep data collected by Gagliardi, et al. [4-6], and ideally at some point we would be in the position to directly compare our results with theirs. In addition, the creep failure parameters (strain rate, stress and strain at failure) observed in our work could be expected to fall on the same master curve as all our quasi-static test data, using the principles of time-temperature superposition [1]. This effort is underway.

By way of summary, the interesting points to be made from this paper are as follows:

- Tensile failure occurs at approximately constant strain regardless of temperature or stress;

- Tensile time to failure appears to correlate inversely with stress ratio, independent of temperature.
- We must test in compression at higher stress ratios if we are to observe failure strain and time to failure for these tests.
- At a given temperature, the slope of secondary creep appears to depend on stress ratio only, with an identical dependence for tension and compression (Figure 10); however, more tests need to be conducted in overlapping regions of stress ratio to confirm that tension and compression slopes are the same;
- The y-intercept values of secondary creep correlate linearly with the ultimate strength of the material, i.e. compressive intercept values are 3 to 4 times larger than tension (compressive strength is 3 to 4 times higher than tensile strength [1]); in addition, for tensile data alone, y-intercept values are slightly larger for cold temperatures than warm (cold temperatures have higher ultimate strength); again, more tests could be performed to confirm this observation.
- Stress-ratio appears to be a very valuable parameter for analyzing PBX creep data, it appears to unify the response at different temperatures, for example.

Acknowledgements: Richard Browning designed the long-term compressive creep frame many years ago. Thanks to ESC (C-8) for the ongoing funding for this work and analysis.

<u>References:</u>

[1] Thompson, Darla Graff; DeLuca, Racci; Brown, Geoff W., "Time-Temperature Analysis, Tension and Compression in PBXs," 2012, J. Energetic Matls., 30(4), 299-323.

[2] Leppard, Claire, Atomic Weapon Establishment, unpublished results, ca. 2003.

[3] "Creep: Time-Dependent Deformation, Chapter 18" from Advanced Mechanics of Materials, 6th ed., Arthur P. Boresi, Richard J. Shmidt, John Wiley and Sons Inc., 2003.

[4] Gagliardi, F., Cunningham, B., Creep Testing Plastic-Bonded Explosives in Uni-axial Compression, proceedings of the XI International Congress and Exposition on Experimental Mechanics, June 2008, Orlando, Florida.

[5] Cunningham, B., Gagliardi, F., Confined Creep Testing of Plastic-Bonded Explosives, presented at the Annual Society of Experimental Mechanics (SEM) Conference, Albuquerque, New Mexico, June, 2-4, 2009.

[6] Cunningham, B., Gagliardi, F., PBX 9502 and LX-17 Creep Data, ADaPT/ HEWO Report, LLNL, February 9, 2009.

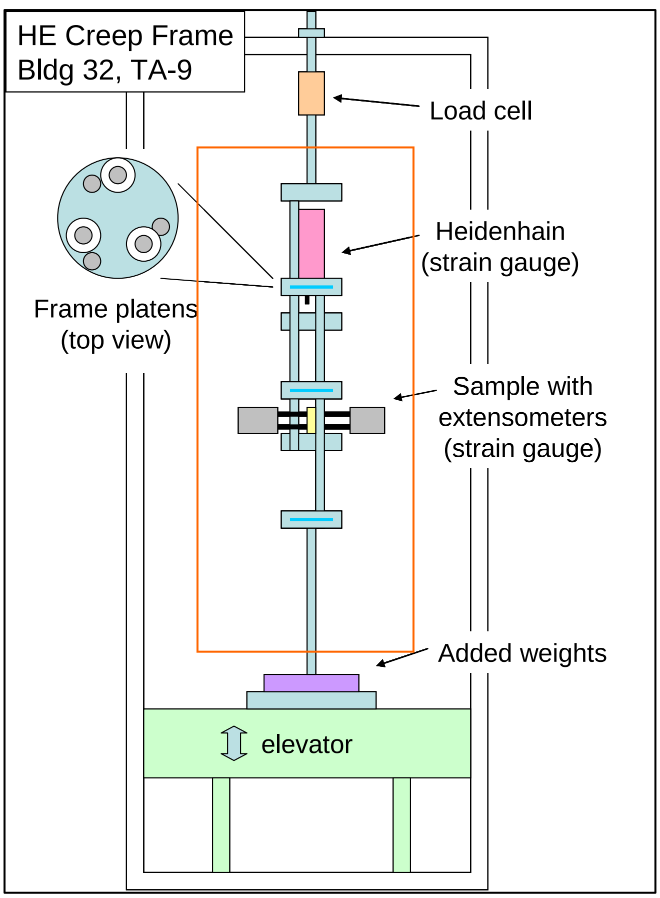
PBX 9502 Creep Data, Compression and Tension
File description, experimental information, and analysis; funded by C-8
Darla Graff Thompson, February 2014
Racci DeLuca and Geoff W. Brown
WX-7, LANL, dkgraff@lanl.gov

I - PBX 9502 (Virgin) Compressive Creep Data from Long-Term Creep Frame

The Virgin PBX 9502 lot used in this study was HOL83H890-019. Specimens were machined from a stockpile-return charge, isostatically pressed. Specimen geometry was 0.375-inch diameter by 1.125-inch long, l/d=3.

The long-term creep frame was designed by Richard Browning (ESA, LANL, now retired) and assembled in 2003/2004. The frame was moved several times to different buildings at TA-9 for various reasons, always in search of a better facility due to power or temperature issues. The frame was finally set up in Bldg 32 of TA-9 and data was acceptable. The PBX 9502 compressive creep data given here were collected in 2010 through 2013.

A rough diagram of the load frame is shown below, Figure 1. An orange line indicates the thermal box used to control the temperature. This thermal box was constructed of 2” Styrofoam and had enough room between the walls and the load train for 1 to 2 feet of wound copper tubing that entered and exited through a port in the back of the box to a Julabo temperature-controlled water bath. The tubing also ported in and out of the two platens directly above and below the specimen (see diagram on left, Figure 1) to help maintain constant temperature at this location. A small muffin fan is mounted in the top of the thermal box to circulate air and to help maintain uniform temperature, minimize gradients.



The concept of the load fixture is that it comes as two, interleaved halves. The first half is comprised of three plates, between each pair of plates are three rods that connect them firmly. The second half is comprised of three other plates, also with plate pairs connected by three rods. The central plates have holes cut (top view of plate on left side of drawing) that allow the two plate assemblies to be interleaved, sliding through each other as the specimen is compressed. Plates and rods are made of Invar which has a very low CTE. Kirk Weisbrod (AET, LANL) made a copy of this design and put some kind of bushings or bearings at the interface between the plates and rods; we learned in a round robin study with him (9501 compression) that this did result in a slight decrease of load that was applied to his specimen. In our system, there is a gap between the rods and the holes in the plates. If the specimen fails non-uniformly then the rods could rub on the plates. We believe this doesn't happen, but our load measurements wouldn't likely detect it if it did.

Data Analysis

Here is an example file of compressive creep on stand-alone creep frame in Bldg 32 at TA-9, file 040412a read into Excel:

DATE	TIME	Seconds	T1	T2	T3	T4	T5	T6	T7	25 lb cell	100 lb cell	mini 100 lb cell	Heidenhain	Extens 7	Extens 8	10 V Supply	5 V Supply	Batt reference
4/4/2012	########	1	21.10813	50.5721	49.87058	46.30699	44.54132	44.83349	47.17183	-1.2029E+41	-14.2500864	-143.7322107	-0.0000012	0.0023287	0.0014924	-10.015391	4.9718603	3.107405
4/4/2012	########	2	21.1048	50.56872	49.87058	46.30699	44.53456	44.83686	47.17521	-1.2029E+41	-14.2415748	-29.7705134	-0.0000014	0.0023338	0.0014915	-10.015378	4.9718472	3.107405
4/4/2012	########	4	21.09811	50.56195	49.86718	46.30699	44.53456	44.8301	47.16846	-1.2029E+41	-14.2132954	-353.0158755	-0.0000018	0.0023321	0.0014907	-10.015417	4.9718735	3.107392
4/4/2012	########	5	21.09811	50.57887	49.87058	46.31375	44.53793	44.83686	47.17183	-1.2029E+41	-14.1719421	-250.4980016	-0.000002	0.0023338	0.0014933	-10.015417	4.9718735	3.107392
4/4/2012	########	6	21.09475	50.56195	49.87058	46.32052	44.53456	44.83349	47.16507	-1.2029E+41	-14.2041387	-4.0883022	-0.000002	0.0023346	0.0014924	-10.015391	4.9718735	3.107392
4/4/2012	########	7	21.09475	50.57547	49.86718	46.32728	44.53117	44.83349	47.16846	-1.2029E+41	-14.2914399	-1.0274996	-0.0000018	0.0023279	0.0014942	-10.015391	4.9718603	3.107392
4/4/2012	########	8	21.08808	50.56195	49.86718	46.32728	44.53456	44.83349	47.16169	-1.2029E+41	-14.2835262	-113.4131029	-0.0000018	0.0023321	0.0014933	-10.015417	4.9718472	3.107392
4/4/2012	########	9	21.08808	50.57547	49.86718	46.34079	44.53456	44.83686	47.16846	-1.2029E+41	-14.2540087	-48.6261588	-0.0000014	0.0023338	0.0014951	-10.015404	4.9718472	3.107392
4/4/2012	########	11	21.08139	50.58565	49.85703	46.36109	44.54132	44.83686	47.16507	-1.2029E+41	-14.2257662	-15.7068059	-0.000002	0.0023346	0.0014951	-10.015417	4.9718209	3.107379

The last three columns (10 V and 5 V Supply and Batt reference) were just to keep an eye on the power supplies for the data channels, in case there were unexplained problems. I have not used these in any of my analysis. Also, I have ignored the first two columns of date and time, using use third column (seconds) for time correlation of data.

Next, seven thermocouples (TCs) were installed at various points inside the thermal box and recorded during the tests. TC1 is outside the box. TC2 and TC3 were mounted on the upper and lower specimen platens and are specifically relevant to the specimen temperature during the test. Some tests controlled temperatures better than others (see Figure 5) and this should be taken into account when data are analyzed (some ran hot, some had unplanned variability during the test).

The primary means of measuring creep strain in the specimen is the Heidenhain strain gauge which is a bit like an LVDT with a single arm extended downward from the mounting platen to a mobile platen (the mobile platen moves relative to the mounting platen as the specimen creeps, see diagram). The Heidenhain was selected for this work because of its high precision and long-term stability. Before the elevator is lowered (placing the load on the specimen), the Heidenhain arm is extended until it pushes on the mobile platen with a very small force controlled via feedback. As the elevator is lowered and the load is applied, the Heidenhain captures the motion. There is nothing in the Heidenhain displacement that indicates the exact zero-point of the specimen, when the specimen is contacted. This is the reason for the two, oppositely-mounted knife-edge extensometers. As strain measuring devices, these do not work well over the long haul; they drift (likely creep of their own weight) and there are many examples in this dataset where the rubber bands used for mounting them break and extensometers literally fall off the specimen. Analysis of the loading portion of the curves (first 50 to 100 data points) is performed as follows, using extensometers to zero the Heidenhain strain data:

The Extens7 and Extens8 data columns are averaged and an amount added or subtracted to make the initial values equal to 0. These columns have units of displacement in INCHES and so next we need to calculate STRAIN using the extensometer gauge length of 0.5 inches.

AveExtensometerStrain = (AveExt7&8 - zeroing) / 0.5

Next we convert the output of the Heidenhain, also in INCHES of displacement, into strain. This signal spans the full length of the specimen (1.125 inches) and also comes out as negative, so we have:

AveHeidenhainStrain = Heiden/1.125*-1

Now, plot both of these strain curves versus time, and zoom in so you are seeing the first HOUR of applied load. Add or subtract from the AveHeidenhainStrain data until it overlaps best with the AveExtensometerStrain. The AveHeidenhainStrain data is now zeroed. Typically, the two strain measurements will overlay quite well initially (first hour), but eventually they deviate and the Heidenhain data is the one to keep.

It is important here to comment about the load channels. There is a data column entitled “25 lb cell” and this is garbage, as is the data column “mini 100 lb cell” (the former was to be in the load train between the frame and the load, the latter was to be sandwiched between the loading platens, in contact with the specimen--- neither of these was used at all). This leaves the one meaningful load cell, column “100 lb cell,” which is mounted at the top of the load train. If you look at the early loading times, you see that the loading of the load cell has slightly different timing than the strain registered on the extensometers. Using the extensometer data to zero the Heidenhain is the most accurate process, and in our 2008 round robin creep testing of PBX 9501, the three different researchers agreed with this procedure (Cunningham from LLNL and Weisbrod from AET, LANL and myself).

Table 1:

Compressive Creep PBX 9502 Virgin (FY12 and FY13)

Load	5 MPa (725 psi)	4 MPa (580 psi)	3 MPa (435 psi)
Weights	291 N (65 lbs)	220 N (49.5 lbs)	149 N (33.5 lbs)
50°C	040412a(10) 060712a(8)	101410a 030311a(6)	080812/082912 022113a
40°C	030112a(4) 051412a(5)	020711a 030211a** 050211a/051711a 110310a	101712 121812

The load calculations, Table 1, take into account the weight of lower half of frame + hanger + weights. Also in Table 1, in the case of double filenames – data collection was stopped and started again immediately, without unloading/reloading the specimen.

The graph of zero-corrected Heidenhain data is shown in Figure 2. We have at least two tests in each test condition, giving us some confidence in our reproducibility. There is a reasonable trend in the strain response with temperature, and a reasonable trend with applied load (given as an engineering stress).

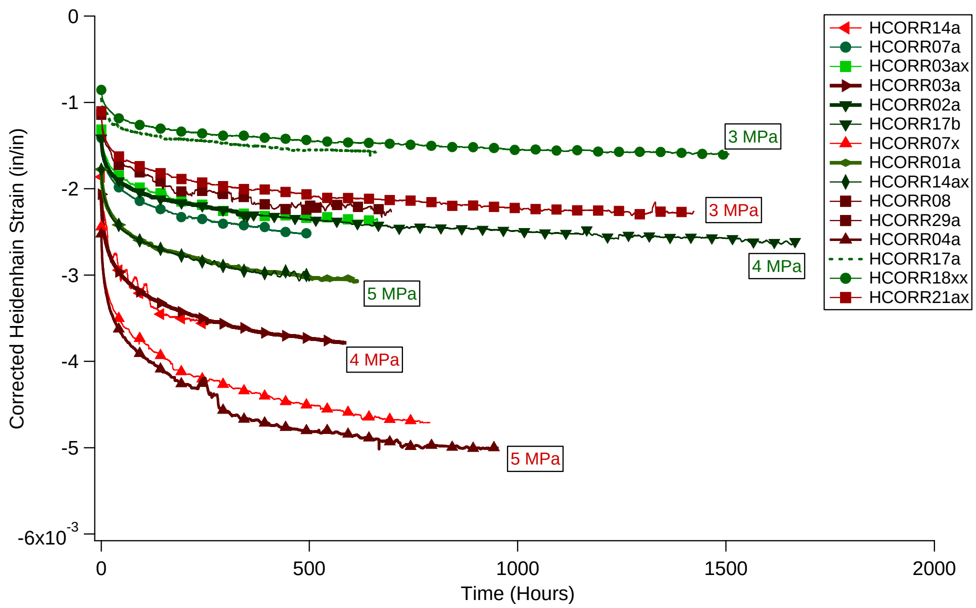


Figure 2: Zero-corrected Heidenhain strain data (see legend for specimen ID, numbers in legend correspond to the third and fourth digits in the filename from Table 1).

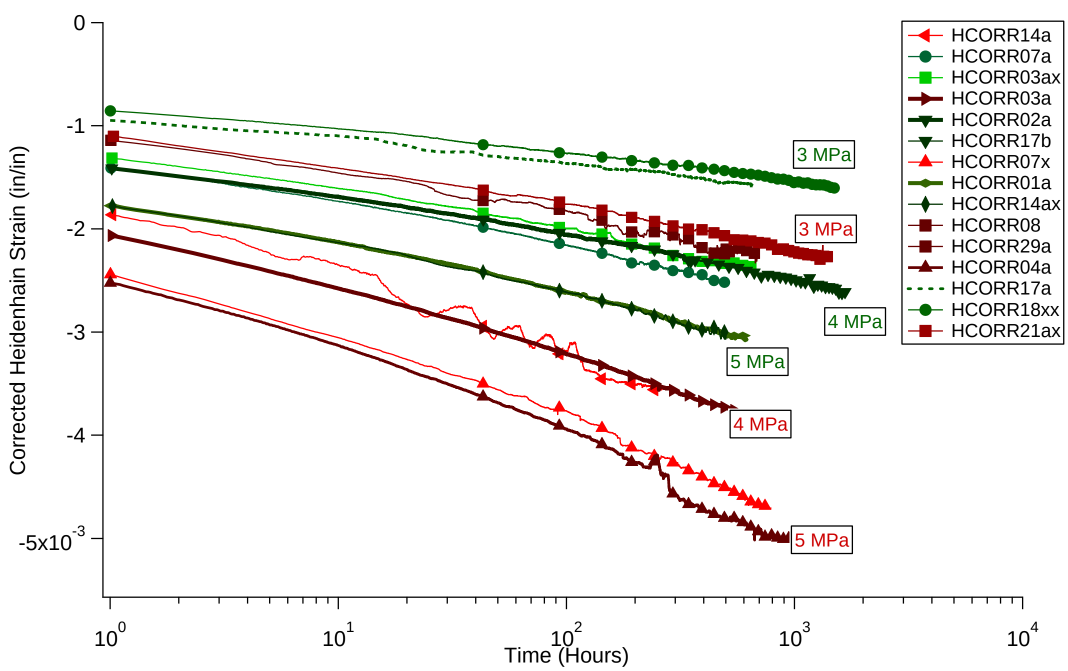


Figure 3: Same as Figure 2 but plotted on a log-scale Time axis.

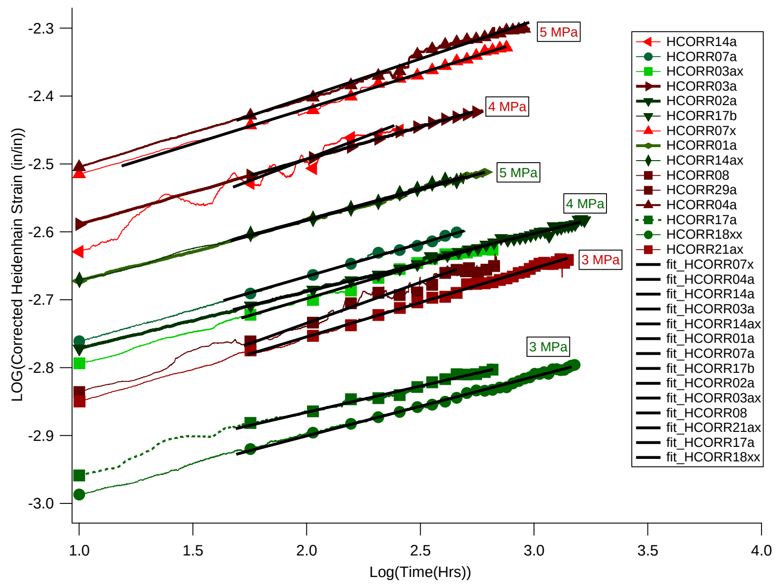


Figure 4: Same data as in Figures 2 and 3 but now on a log-log plot with "best-fit" least-squares lines through each curve.

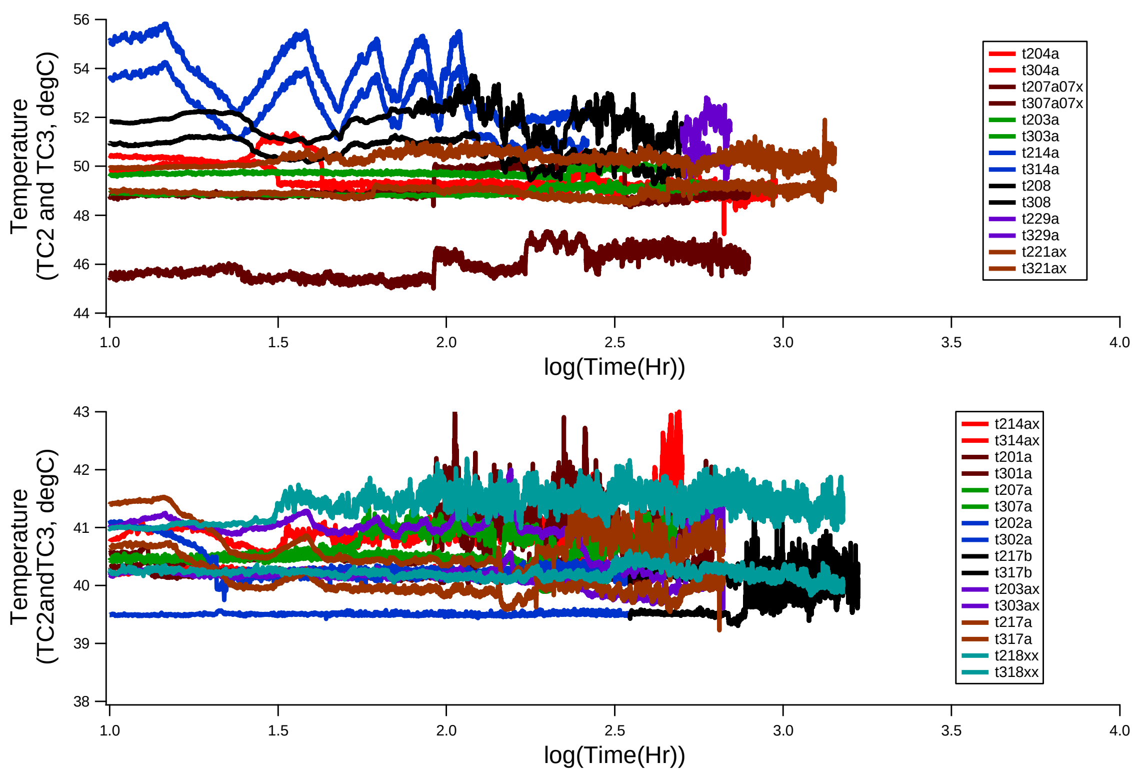


Figure 5: TC2 and TC3 outputs for all tests at 50°C (top) and 40°C (bottom); the effect of poor temperature control can often be seen by comparing TC output with corresponding strain data in Figure 4.

Table 2: Linear fitting results of log-log plots, Figure 4 (slope units strain(%)/time(sec))

Temp (°C)	Filename	Stress (MPa)	slope	y-intercept	Stress ratio	Density (g/cm^3)
50	040412a(04a)	5	0.1120	-0.2317	0.360	1.8958
	060712a(07x)	5	0.1040	-0.2602	0.360	1.8966
	030311a(03a)	4	0.0950	-0.3452	0.290	1.8960
	101410a(14a)	4	0.1280	-0.2938	0.290	1.8948
	080812/082912(08)	3	0.1203	-0.5472	0.210	1.8939
	022113a(21ax)	3	0.1009	-0.5978	0.210	1.8958
40	051412a(14ax)	5	0.0916	-0.4393	0.290	1.8950
	030112a(01a)	5	0.0907	-0.4426	0.290	1.8952
	020711a(07a)	4	0.0968	-0.4558	0.235	1.8970
	050211a(02a)/(17b)	4	0.0820	-0.5606	0.235	1.8933
	110310a(03ax)	4	0.1000	-0.5437	0.235	1.8963
	101712(17a)	3	0.0772	-0.7459	0.177	1.8958
	121812(18xx)	3	0.0873	-0.7648	0.177	1.8967

II - PBX 9502 (Virgin and Recycled) Tensile Creep Data from Instron 5567

For these tests, both Virgin and Recycled PBX 9502 processing methods were included. Recycled specimens came from lot HOL88A891-006, isostatically pressed. Virgin specimens came from lot HOL88H891-008. Both materials were isostatically pressed at LANL as the hemispherical mold type A42. Specimens are 3-inch long dogbones with a gauge length of 1.5 inches; the diameter of the gauge section is 0.5 inches.

All tensile creep tests were run on the Instron 5567 (TA-9, Bldg 33). This instrument is equipped with a Bemco environmental chamber for temperature control and dehumidification. The liquid N_2 Dewar size was the main limitation on our creep test duration. Strain was measured using two oppositely-mounted knife-edge extensometers with a gauge length of 1.0 inch. The primary reason we could not run tensile creep tests on our creep frame is that the Instron has a “break detection” feedback system that detects when the specimen load shows a sudden and significant drop, indicating failure of the specimen; the system then immediately stops any further crosshead motion so that the extensometers will not be pulled beyond their safe operational displacement.

Table 3 is the test matrix for our tensile creep. Filenames are given along with test conditions such as temperature and stress. Other relevant test results are given such as time to failure and stain at failure (or maximum strain in the tests where specimens did not fail). For these tests, log(strain) vs. log(time) plots of some of the tests were made, see Figure 6. For these tests, we are primarily interested in quantifying the secondary “linear” part of the creep curve, and to this end, least-squares fits were made. Slopes and y-intercept values are listed in Table 3 (note that not all of the data sets are plotted or were fit). There is a fair amount of scatter in the data--- more than we expect to see in uniaxial quasi-static tension or compression. Creep data is known to be sensitive to flaws or microstructural differences [3]. Within the experimental scatter, if one focuses on the averaged numbers, there are clear trends that appear as a function of creep stress and temperature. In Figure 7 we plot creep stress and tensile strain at failure (or maximum strain for those specimens which did not fail) versus log failure time. Note that tensile strain at failure is pretty much the same for all temperatures and stresses, between 0.15 and 0.2 %. Tensile failure strain in quasi-static tests is also quite independent of temperature and strain rate, with values between 0.22 and 0.26% [1].

Table 3: Recycled and Virgin PBX 9502 Tensile Creep on the Instron 5567

Temp (°C)	Filename	Stress (MPa)	Stress Ratio	Time to Failure (hrs)	Max Strain (%)	Slope	Intercept	Density (g/cm^3)
50	021710a/19a	2.00	0.38	137*	0.159	0.1152	-1.4591	1.8928
	053111a			309*	0.191	0.1082	-1.3607	1.8913
	082008a	2.63	0.50	1.0*	0.206			1.8911
	081908a/b			1.18	0.152			1.8911
	081108b			10.0*	0.192			1.8919
	081908c			19.3	0.215	0.1291	-1.3052	1.8926
	022310b			7.1	0.192			1.8921
	081208a/b	2.98	0.56	0.1	0.131	0.1029	-1.148	1.8912
	081808c			0.22*	0.203	0.203	-1.283	1.8909
	041811a			1.64	0.184	0.128	-1.252	1.8932
	042711a			1.19	0.194			1.8964
	081108a	3.42	0.65	0.25*	0.191			1.8916
	022310a			0.20	0.188			1.8929
	022410a			0.16	0.177	0.1150	-1.092	1.8919
	041111a			0.18	0.202	0.1633	-1.151	1.8929
40	102110a	2.63	0.38	168	0.156			1.8937
	020811a			216*	0.160	0.0928	-1.339	1.8927
	013111a			76*	0.147			1.8926
	042711b			23	0.133			1.8964
	042811a			428*	0.172	0.0916	-1.330	1.8906
	110110a	2.98	0.43	103	0.187			1.8922
	111510a			350*	0.208	0.1001	-1.296	1.8939
	041811b			105	0.163	0.095	-1.335	1.8938
	110810a	3.42	0.49	30.5	0.208	0.1051	-1.224	1.8930
	120210c			8.8	0.193			1.8959
	040711a			22.6	0.224			1.8932
30	120710a	3.42	0.38	341*	0.142	0.0760	-1.320	1.8931
	010411a			264	0.179			1.8938
	120610a	5.00	0.56	1.49	0.170			1.8931
	012711a			3.3	0.181	0.0872	-1.105	1.8929

* Did not fail, test stopped manually.

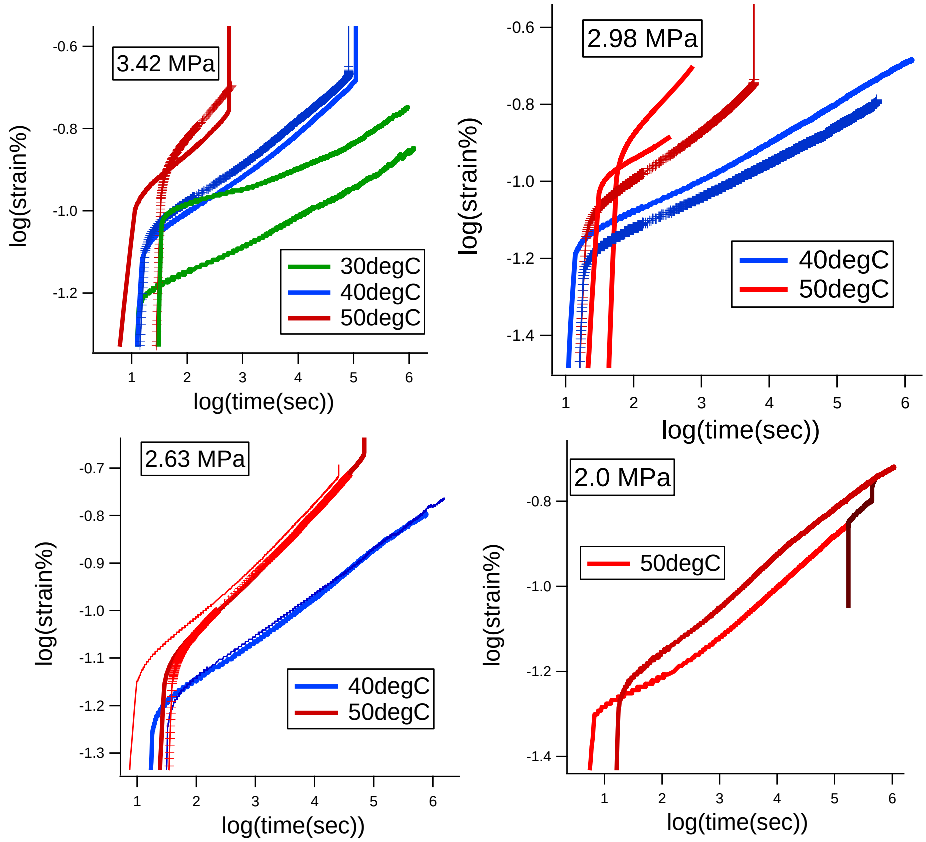


Figure 6: Example PBX 9502 tensile creep curves from Table 3 showing the effects of temperature and creep stress.

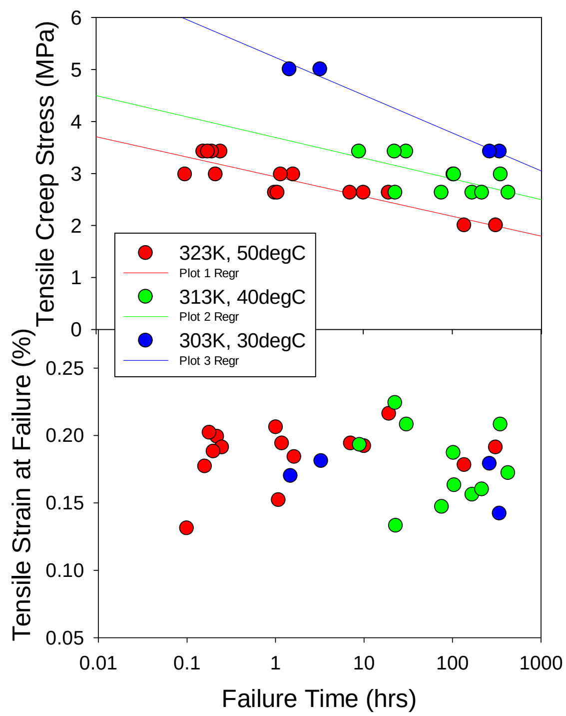


Figure 7: Plots of Creep Stress and Tensile Strain at Failure (or Maximum Strain for those specimens which did not fail) versus Failure time on a log scale. Note that Tensile Strain at Failure is quite invariant at all stresses and temperatures.

Figures 8 and 9 show slope and y-intercept values from the secondary creep curve for both tension and compression. (Note that to put tensile and compressive log-log plot parameters on the same graph, the log-log plots must be made on axes with the same strain and time units, which we have done.) Figure 8 shows these fitting parameters as a function of temperature, while Figure 9 shows them as a function of stress. See legends for symbol identification.

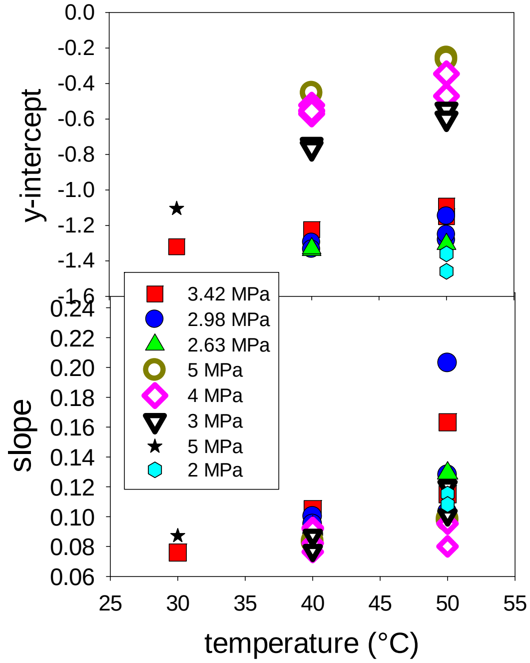


Figure 8: Fitting parameters (slope and y-intercept) plotted versus test temperature; open symbols are from compression data, solid symbols are tension. See legend for stresses.

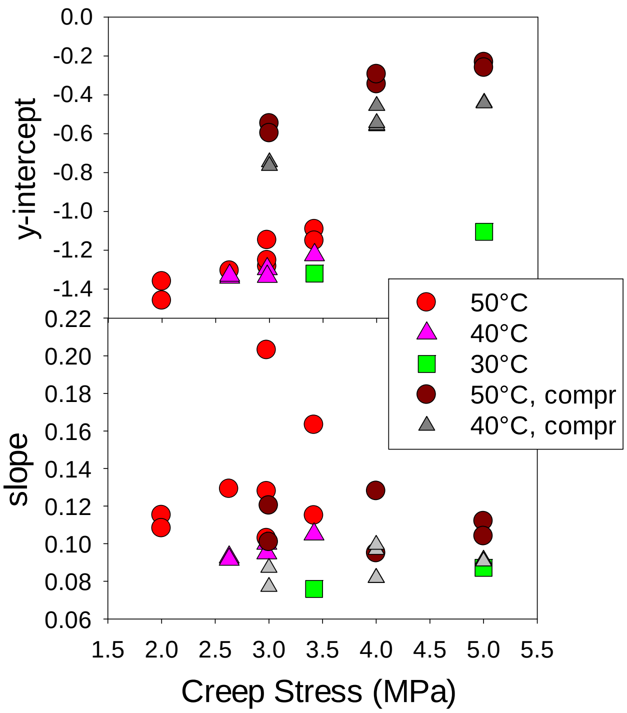


Figure 9: Fitting parameters (slope and y-intercept) plotted versus creep stress; see legend for symbol identification and temperatures ("compr" indicates compression data).

The purpose of Figures 7, 8 and 9 is to try to establish trends as a function of test parameters. To the extent this can be done reliably with the data we have, we can use it to 1) establish an empirical model and 2) interpolate and possibly extrapolate creep response to conditions somewhat different than those we have actually measured. Inspection of Figures 7 thru 9 show that the trends are reasonable and consistent and should be useful in building a model. However, see below, we propose that Figures 10 and 11 are the best for showing consistency and establishing data trends.

For this next step of analysis, instead of simply considering the creep stress of any given test, we consider the stress ratio [2]. Stress ratio is defined as being the ratio of the creep stress (i.e. the stress applied during the creep test) to the ultimate stress of the material at these loading conditions (specifically at this temperature and this strain rate of loading). Using the results of time-temperature superposition [1] we calculate the stress ratio for each of the conditions, and these values are included in Table 2 and 3. In Figure 10, we plot the linear fitting parameters once again but this time versus stress ratio. As stated earlier, there is some variation in the fitting parameters, particularly the slope values at 50°C in both tension and compression, however, if one eyeballs an average response at each set of unique conditions, there are some very useful observations that can be made. Looking at the y-intercept versus stress ratio plot we see that all the compression data clusters together, all the tensile data clusters together; the y-intercept value relates to the strain reached during the loading process… and it is exactly what we expect (based on quasi-static data) that at the same stress ratio, compressive strain will be larger than tensile strain (ultimate compressive strength is larger by a factor of 3 than ultimate tensile strength). Interestingly, the slope values for tension and compression all appear to fall on a common trend line showing an increasing slope with increase in stress ratio. These observations point us in useful directions as to what conditions should be tested next, to determine if these trends hold true over a wider range of parameters.

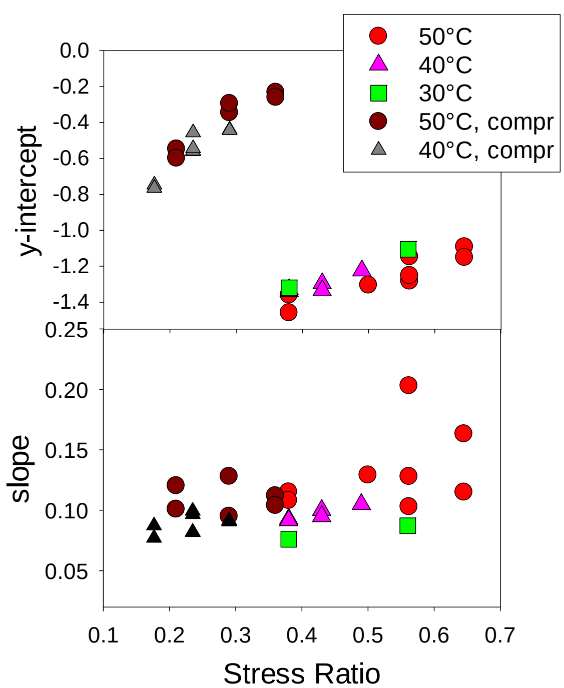


Figure 10: Fitting parameters (slope and y-intercept) plotted versus stress ratio; see legend for symbol identification and temperatures ("compr" indicates compression data), the other data are tensile.

Figure 11 is a repeat plot of Figure 7, except instead of plotting tensile creep stress versus failure time in the upper plot, we have plotted tensile stress ratio versus failure time. Similar to what was shown in Figure 10, by plotting creep stress information as stress ratio, the data appear to fall on the same trend line, with higher stress ratios giving rise to shorter failure times (as one would expect). By "normalizing" creep stress (i.e. dividing by the ultimate stress at that condition), we have reduced an aspect of the creep response (failure time) at different temperatures to a common curve.

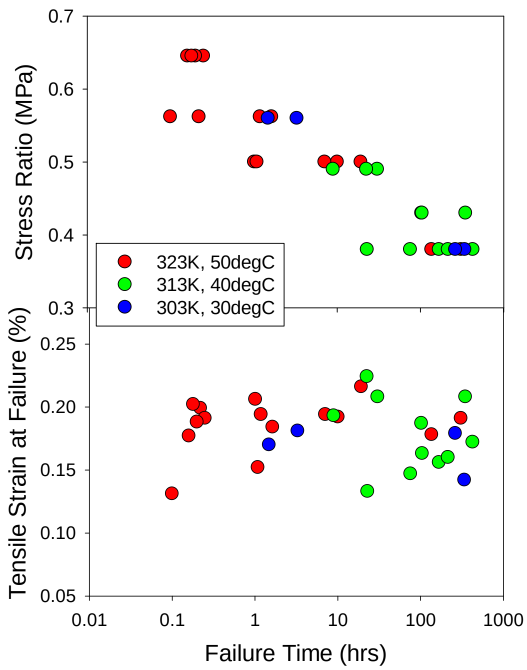


Figure 11: Repeat plot of Figure 7, however this time showing tensile stress ratio plotted versus failure time in the upper graph.

Summary

Creep tests on PBXs are inherently difficult. Strains are small and temperature control must be rigorous over an extended period. Small microstructural differences (PBX heterogeneities) are known to affect creep data and cause variation in the measured results [3]. Nevertheless, with the modest set of compressive and tensile creep data reported here, we believe we have the basis for understanding creep response in this material and for outlining a creep model with appropriate parameter dependencies. These PBX 9502 data include both Recycled and Virgin processing methods; careful analysis of the data may show that the creep response of the two processing types is statistically different, however, we have chosen to put both types on the same plots with the assumption that processing type is not a strong factor in determining creep response. We are aware of some PBX 9502 creep data collected by Gagliardi, et al. [4-6], and ideally at some point we would be in the position to directly compare our results with theirs. In addition, the creep failure parameters (strain rate, stress and strain at failure) observed in our work could be expected to fall on the same master curve as all our quasi-static test data, using the principles of time-temperature superposition [1]. This effort is underway.

By way of summary, the interesting points to be made from this paper are as follows:

- Tensile failure occurs at approximately constant strain regardless of temperature or stress;
- Tensile time to failure appears to correlate inversely with stress ratio, independent of temperature.
- We must test in compression at higher stress ratios if we are to observe failure strain and time to failure for these tests.
- At a given temperature, the slope of secondary creep appears to depend on stress ratio only, with an identical dependence for tension and compression (Figure 10); however, more tests need to be conducted in overlapping regions of stress ratio to confirm that tension and compression slopes are the same;
- The y-intercept values of secondary creep correlate linearly with the ultimate strength of the material, i.e. compressive intercept values are 3 to 4 times larger than tension (compressive strength is 3 to 4 times higher than tensile strength [1]); in addition, for tensile data alone, y-intercept values are slightly larger for cold temperatures than warm (cold temperatures have higher ultimate strength); again, more tests could be performed to confirm this observation.
- Stress-ratio appears to be a very valuable parameter for analyzing PBX creep data, it appears to unify the response at different temperatures, for example.

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