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F. J. Gagliardi, B. J. Cunningham

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Franco J. Gagliardi, gagliardi7@llnl.gov
Bruce J. Cunningham, cunningham1@llnl.gov

Lawrence Livermore National Laboratory
Energetic Materials Center
P.O. Box 808, L-282, Livermore, CA 94550, USA

ABSTRACT: High fidelity measurements of time-dependent strain in the plastic-bonded explosives LX-17-1 and PBX 9502 have been performed under constant, uni-axial, compressive load using a custom designed apparatus. The apparatus uses a combination of extensometers and linear variable differential transformers coupled with a data acquisition system, thermal controls, and gravitational loading. The materials being tested consist of a crystalline explosive material mixed with a polymeric binder. The behavior of each material is related to the type of explosive and to the percentage and type of binder. For any given plastic-bonded explosive, the creep behavior is also dependent on the stress level and test temperature. Experiments were conducted using a 3x3 stress-temperature matrix with a temperature range of 24˚C to 70˚C and with stresses ranging from 250-psi to 780-psi. Analysis of the data has shown that logarithmic curve fits provide an accurate means of quantification and facilitate a long-term predictive capability. This paper will discuss the design of the apparatus, experimental results, and analyses.

INTRODUCTION

Plastic-bonded explosives (PBXs) are typically bi-phase composite materials consisting of a mixture of explosive crystals and a polymeric binder. The ratio of the explosive component to the binder varies from one material to the next, but, typically, the explosive component comprises 80-95% of the mass of the composite [1]. One benefit of the presence of binder is the reduction in the composite's sensitivity. This occurs because the binder coats the explosives crystals and acts as a barrier between them, reducing heat generation created by friction during deformation [2]. Another benefit is that PBX composites have superior mechanical strength compared to pure explosive parts, allowing precision parts to be machined from the compacted material [3]. Precision geometries are important features in systems where detonation propagation needs to be highly predictable. Stability of the machined geometry is related to the mechanical properties of the PBX.

Many types of testing are used to characterize the mechanical properties of various PBXs, including quasi-static uni-axial compressive, and uni-axial tensile tests. Uni-axial tension and compression tests are used as a means of characterization for nuclear stockpile certification, material model development, and new lot qualification [4]. These tests provide the much-needed information for the development of accurate material models. The development of such models allows us to examine the material response of various charge configurations to diverse load and thermal conditions.

The mechanical properties of PBXs are highly dependent on both temperature and strain rate [5]. Thus, multiple tests covering a broad range of temperatures and strain rates are needed for any material before we may feel confident that we can predict its behavior. In many of our PBX applications, creep behavior is of particular interest because it can lead to part fracture. Creep is very low-rate, time-dependent strain that can occur because of the mechanical configuration of a system, the thermal environment to which a system is exposed, or a combination of the two. One of our objectives at LLNL has been to develop a robust predictive PBX material model that can be validated with materials testing and that includes an accurate means of accounting for the creep behavior of the material. In generating data for creep model development we must accurately measure loads, stresses, temperatures, and strains over extended periods of time [6].

From the standpoint of an explosive reaction, a subset of PBX materials exhibits extraordinarily insensitive behavior to diverse stimuli and has consequently achieved the designation of insensitive high explosives (IHEs). The particular explosive molecule that currently stands alone in the IHE class is triamino-trinitrobenzene (TATB). Several PBX materials have been formulated with TATB as their main constituent and because of their extreme insensitivity they are used in a number of very important applications. LX-17-1 is the LLNL name given to the composite of 92.5% TATB and 7.5% Kel-F 800™ by weight. PBX 9502 is the LANL name given to the material, whose constituents are 95% TATB and 5% Kel-F 800™ by weight [7]. The creep behavior of these two IHE materials is the focus of this paper.

CREEP AND DESIGN OF THE CREEP APPARATUS

Recent work done at LLNL has focused on enhancing our understanding of the creep behavior of LX-17-1 and PBX 9502 when subjected to a constant load over extended periods of time. A typical uni-axial compressive creep test of PBX material consists of four phases (see Fig. 1). The first phase is the initial load-up, which happens rapidly as the load is initially applied to the sample. The second phase is the creep period during which strain accumulates over time, usually at a diminishing rate. Third, after a prescribed time period, the load is removed and the sample immediately responds by recovering elastically. Then in the last phase, the sample continues to gradually recover some additional deformation during what we refer to as the recovery period.

Many factors were considered when designing our creep apparatus in an effort to ensure that the accuracy of the measurements would be sufficient. Some of the considerations included sample size, test temperature range, test temperature stability, sample preparation, the potential effects of sample history, load and stress magnitudes, loading methods, test duration, gauging, and repeatability (see Fig. 2). While each of these variables is important on an individual basis, many of them overlap, in a practical sense.

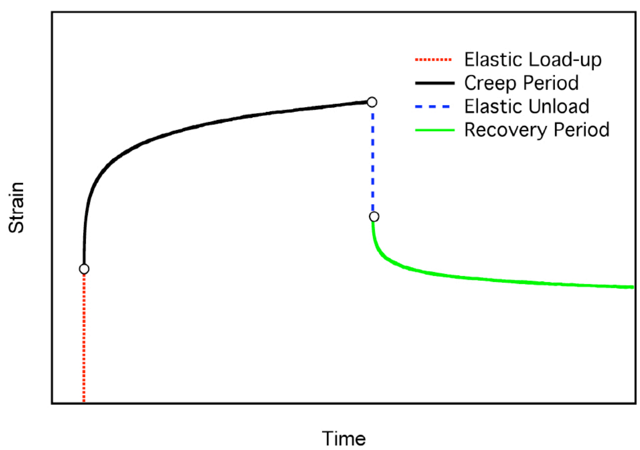

FIGURE 1: A typical uniaxial compressive creep test consists of four phases. First, the specimen exhibits largely elastic behavior in the initial moments during load application. The next portion of the curve is referred to as the creep period. During this time the rate of strain accumulation typically decreases as time progresses. Next comes the elastic unload, during which some of the deformation is rapidly recovered when the load is removed. The final stage occurs gradually over time, and is labeled the recovery period.

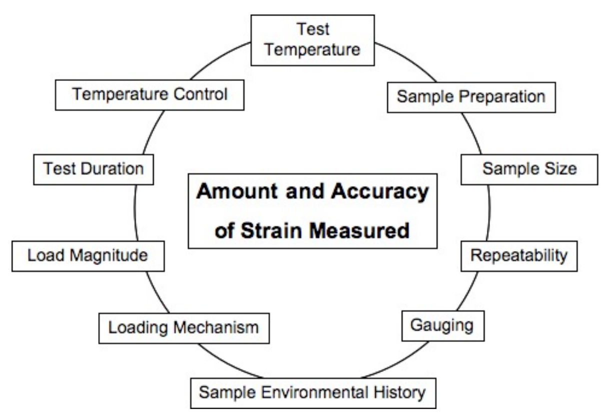

FIGURE 2: Diagram of many of the considerations that were made when designing and planning for creep experimentation. The temperature of the sample affects the material properties of the PBX binder, the sample size affects the amount of load needed to achieve a certain stress, the sample history can affect the part integrity and geometry, the loading needs to be constant, the gauges must be stable and have fine resolution, and the tests must be repeatable for system validation and so that material comparisons can be made.

EXPERIMENTAL METHOD

The typical specimen used in our compression tests has a 0.500-inch diameter and is 1.000-inch long. Compression samples of LX-17-1 and PBX 9502 are shown in Fig. 3. A PBX specimen of the given dimensions weighs approximately 6-grams. The availability and the compact size of these standard parts made them good candidates for use in the compressive creep tests.

The binder in LX-17-1 and PBX 9502 is Kel-F 800™, which has a glass transition temperature (T_g) around 30˚C. Additionally, Kel-F 800™ has a melt temperature (T_m) that occurs around 95˚C [8]. Between the T_g and the T_m the binder gets progressively more compliant. To best understand the material behavior over the full range of practical temperatures, data must be taken at multiple temperatures throughout the range.

Specimen temperature changes during a test will dynamically alter the creep behavior of the material, as is illustrated in Fig. 4 [9]. Also, temperature changes can cause the displacement gauges to output data that does not reflect the actual changes that are occurring in the sample, due to non-canceling thermal expansion behavior of various parts of the fixture. With this in mind, the maintenance of a stable test temperature is essential. To achieve temperature stability, a standard 60-100-watt light bulb is electronically connected to an Omega PID closed-loop temperature controller that is programmed to control to a set temperature. The chamber is insulated using fiberglass insulation and for low temperature tests, the creep chambers are equipped with copper coils connected to a liquid circulator that utilize a chilled mixture of water and ethylene glycol. During the test, a thermocouple is attached to the PBX sample using Teflon® tape, and the specimen temperature is monitored and recorded remotely on a custom designed LabView-based data acquisition system. Prior to initiating the test (i.e. loading the specimen) the temperature and gauges are carefully monitored to ensure the system is at thermal equilibrium. This system is capable of controlling the test temperature within a few tenths of degree Centigrade.

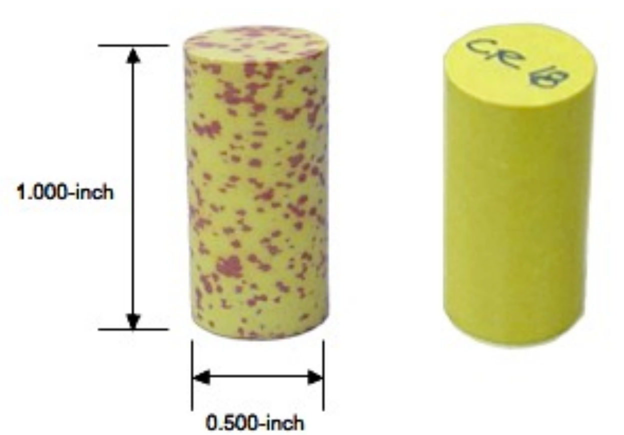

FIGURE 3: LX-17-1 (on left) and PBX 9502 (on right) compression samples. The samples used for creep testing at LLNL are typically machined from iso-statically or hydrostatically pressed billets or hemispheres into the desired size of 0.500-inch in diameter and 1.000-inch long cylinders. Before and after testing the parts undergo immersion density measurements, as well as geometric measurements using a micrometer.

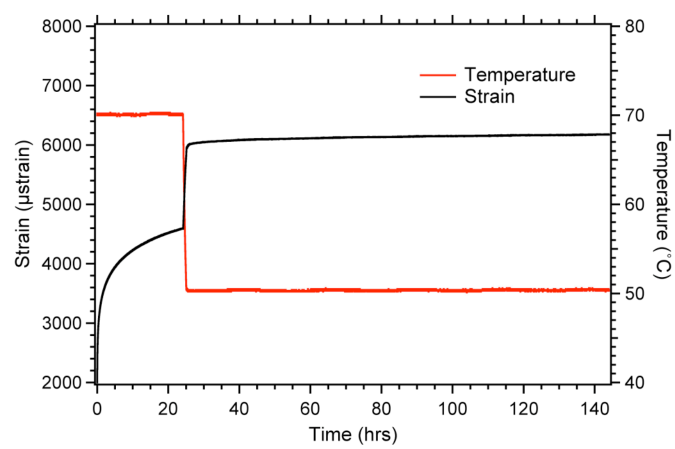

FIGURE 4: PBX 9502 creep test results displaying changes in material creep response to temperature change (from 70˚C to 50˚C). The amount of strain offset during the temperature change is a related to the coefficient of thermal expansion of the sample.

PBX samples are made by iso-statically or hydrostatically compacting heated molding powder into rough cylindrical billets or rough hemispheres [10]. The large compactions are then x-rayed to ensure they are free from any inclusions or other internal defects. Suitable billets or hemispheres are then sliced, or cored, and the sub-portions are further machined into the final desired dimensions. The parts are machined to a high degree of precision, ±0.001-inch in length, diameter, parallelism, perpendicularity and flatness [11]. Using this technique, multiple samples with precision geometry and near uniform densities can be obtained. Alternatively, individual samples can be die pressed using a heated steel die and plunger set. These parts may be pressed to size, or subsequently they may be machined down to an appropriate geometry [10].

Neither LX-17-1 nor PBX 9502 is a perfectly elastic material. In addition, these materials are known to exhibit permanent property changes when exposed to fluctuating temperatures [12]. Thus, their environments, prior to testing, must be controlled. A sample that has experienced material changes due to load or thermal conditions will not compare well with a pristine part, so efforts must be made to keep the parts as pristine as possible if uncertainties associated with a specimen's history are to be avoided.

Some PBX materials, other than LX-17-1 and PBX 9502, have constituents that are sensitive to humidity and therefore must be tested in humidity-controlled environments. For the LX-17-1 and PBX 9502 tests hygrometric controls are unnecessary.

Adjusting the amount of force exerted on the load train controls the load magnitude, which is directly related to the stress in a given sample. Standard exercise weights are used in various quantities to achieve the desired sample stress. Prior to starting a test with a newly selected weight set, a load cell is placed in the fixture where the sample would normally reside. The load is then applied and monitored to determine whether the desired magnitude has been achieved. Depending on the results, weights will be added or removed until the target load is achieved. The cross-sectional area of the sample is directly related to the stress seen in the material, so an accurate measurement of sample geometry is important in determining the average stress value.

To maximize load stability, gravitational loading is used. While pneumatic and hydraulic loading mechanisms were possible alternatives, we felt that neither method provided the stability that can be achieved using a simple weight system. An electronic screw lift, which sits below a steel platform, can be lowered or raised, allowing us to load or unload the system remotely.

Creep is a function of time and thus, the test duration will determine the total amount of strain accumulated in the sample during the test. Also, because PBXs will fail at sufficiently high uni-axial strains, creep can ultimately result is material collapse [13]. In real applications, the material may be subject to loads (either mechanically, or thermally induced) over a very long period of time, so a broad understanding of the temporal effects is important. A long-term predictive modeling capability is one of the overarching goals associated with our effort and so tests must be of sufficient duration to reliably establish long-term trends.

The rate of displacement change that we measure during a typical creep test may be on the order of 10^{-6}-inch per hour (approximately 10^{-10} μstrain per second) or less, and this requires displacement gauges that have a resolution that can accommodate such small measurements. Additionally, for our purposes, the significant temperature range extends from -54˚C to approximately 70˚C. Our gauges must be functional and accurate over the entire temperature range. Tests may be run continuously for multiple months, so long-term gauge stability is another necessity.

Our creep fixture allows for both contact and non-contact displacement measuring devices to be used simultaneously. A contact pair of extensometers built by the John A Shepic Company of Lakewood, Colorado has a 0.5-inch gauge length and these gauges are attached to the sample using rubber bands or springs. The extensometers have many attractive features, such as high strain resolution and compact size, but they have proven to have stability problems over long periods of time.

Non-contact linear variable differential transformers (LVDTs) are mounted to the upper loading plates and are used to measure the relative motion between the two hexagonal plates in the fixture. The LVDTs used in the fixture are Schaevitz MHR 100 series, which have a range of ±2.54-mm and a resolution of 100-nm. Due to the fact that a sample may have imperfectly parallel ends, three LVDTs spaced 120˚ around center are used to compensate for uneven loading. During data analysis the average of the three gauges is taken and used to calculate inferred strain in the sample. Also, because of imperfect contact between the sample ends and the fixture plates, a certain amount of "false strain" may be seen in the LVDT measurements during the initial load and unload phases of the experiment. "False strain" is corrected for during the data analyses by adjusting the LVDTs strain at load-up to match the load-up strain measured by the extensometers.

Combining all of the considerations, from temperature control, to loading mechanism, to gauging requirements, a system has been assembled and a process determined that is designed to mitigate as many sources of uncertainty as possible. Fig. 5 shows a schematic of the system overlaid with a photograph of the inside of the chamber. Fig. 6 shows a solid model drawing of the fixture connected to the load train.

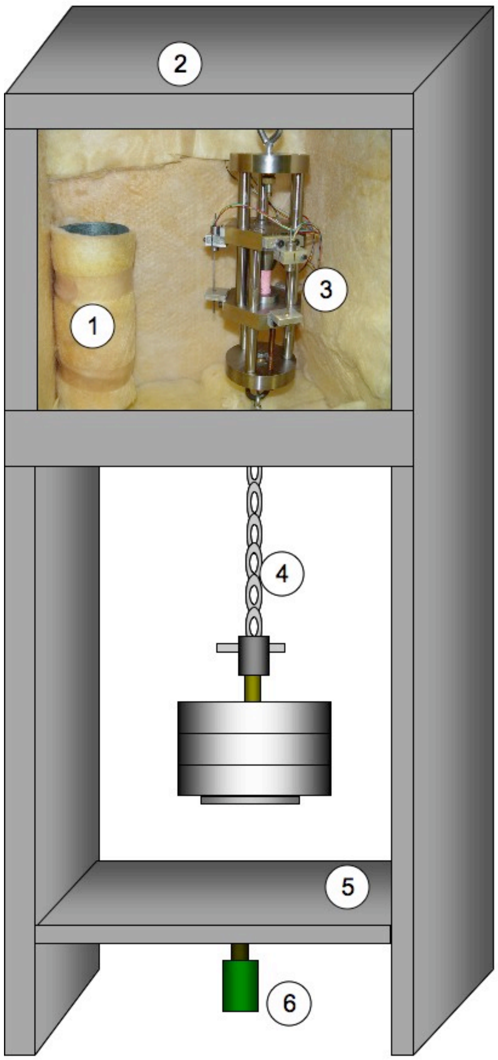

FIGURE 5: Schematic of the creep frame, with photo inset, showing 1) an insulated covering that surrounds the light bulb heat source, 2) a steel frame that is layered with fiberglass insulation on the inside (fiberglass front covering is not shown), 3) the creep fixture with an inert sample in place, 4) the load train, 5) an adjustable platform used for loading the sample and 6) an electronic screw-lift that provides the work needed to move the load train.

FIGURE 6: Solid model drawing of the creep system highlighting the linked connections between the fixture and the load train. Stress is applied to the sample gravitationally using standard exercise weights. Quick links and eyebolts are used to connect a chain to a tube and platform that supports the weights.

Fig. 7 shows an enlarged view of the fixture highlighting the main features including the LVDT mounts, the sample centering pins, the connector rods, the eyebolts and brass spheres that provide pivotal motion and reduce bending moments. The contact extensometers are not shown on the sample. The connector rods and the cylindrical and hexagonal plates are all made from stainless steel. The top cylindrical plate and the bottom hexagonal plate are connected to each other and are constrained in the vertical direction during the test. The top hexagonal plate and the lower cylindrical plate are connected to each other and to the load train. When the load is applied, the force is transmitted through the lower cylindrical plate and into the connector rods, which create a downward motion on the top hexagonal plate. This motion creates closure between the two hexagonal plates and transforms the tensile force from the gravitational loading into a compressive force in the sample.

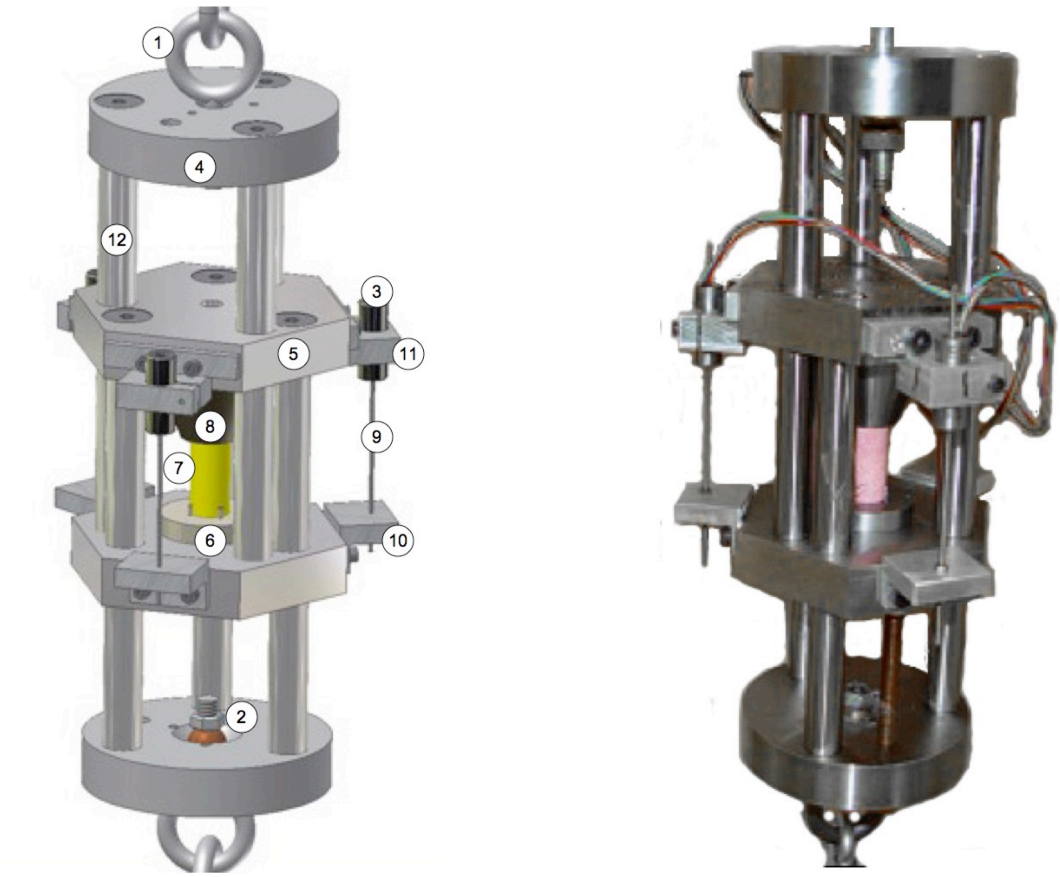

FIGURE 7: Schematic diagram on left and photograph on right of the creep fixture. 1) Eyebolts serve to connect the fixture to the loading chain as well as to the top of the chamber. 2) Brass spheres are used to create a pivotal linkage between the eyebolts and the outer cylindrical plates of the fixture. A nut serves to hold the spherical bearing on the eyebolt. 3) Three LVDTs are used as the long-term displacement gauges (spaced 120˚ degrees apart around center to compensate for uneven loading). 4) Cylindrical plates act as the outer support for the fixture. 5) Two hexagonal plates transmit the compressive load, as the outer plates are pulled apart. 6) The lower adaptor plate has two pins spaced 90˚ apart that center the sample. 7) The sample (yellow) is 0.5-inch in diameter and 1.0-inch long. 8) The conical adaptor piece is in contact with the top of the sample and transmits the compressive load from the upper hexagonal plate into the sample. 9) Three LVDT core mount rods, made of 0.0625-inch all-thread, hold the LVDT cores in place during the test. 10) Platforms attached to the lower hexagonal plate hold each of the three LVDT core mount rods. 11) The upper LVDT mount platform holds the actual LVDT body. 12) Six 0.5-inch rods connect the outer cylindrical plates to the inner hexagonal plates creating the pull-push motion used to apply the compressive load to the sample.

Test repeatability and experiment-to-experiment consistency are necessary in order to produce comparable, reliable data. To ensure our system is both repeatable and reliable, many validation tests have been run on inert test specimens, as well as on PBX samples. Fig. 8 is a plot overlay of the average extensometer and average LVDT readings from a PBX 9502 test. After the “false strain” has been corrected by shifting the LVDT data, the two sets of measurements overlay well. Fig 9. shows another validation test in which two different samples of PBX 9502 were run using the same load and thermal conditions. The data for the tests compare remarkably well.

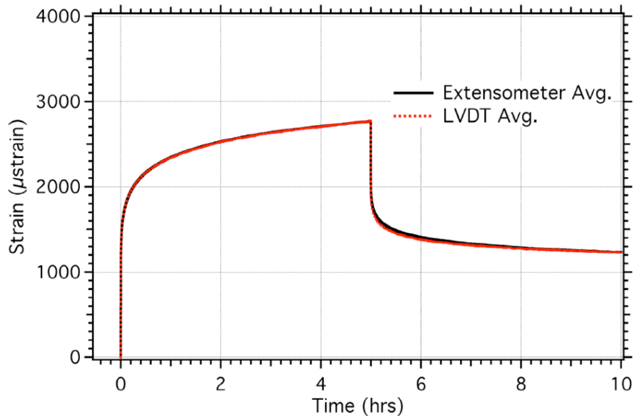

FIGURE 8: Plot from a PBX 9502 test showing agreement between the strain recorded by the contact extensometers and the strain inferred from the LVDTs. The data for the LVDTs has been shifted vertically to compensate for the “false strain” typically seen during the load application.

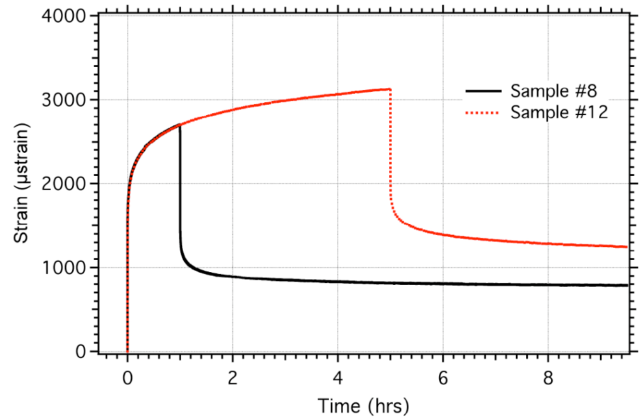

FIGURE 9: A comparison of PBX 9502 creep samples #8 and #12 showing the overlay of the strain change over time, displaying the reproducibility of the test technique.

EXPERIMENTAL PROCEDURE

To characterize the behavior of LX-17-1 and PBX 9502 a three-by-three stress/temperature matrix was chosen for use with both materials. Nominal stresses were ~270-psi, ~500-psi and ~780-psi. These stress levels were chosen because they are consistent to stress levels to which the material may be subjected in real-world applications. The temperatures chosen for this evaluation were 24˚C, 50˚C, and 70˚C. These temperatures were chosen because they stretch across the above-ambient range of likely temperatures seen during the life of the material once it has been fielded. A total of nine samples of LX-17-1, lot 851-010, and nine samples of virgin PBX 9502, lot 890-005, were used to perform these tests.

The PBX 9502 matrix was run once validation tests showed that the LVDTs and extensometers could be used in tandem to produce reliable and repeatable strain measurements. The test duration for the nine tests was chosen to be four weeks of creep and eight weeks of recovery.

The LX-17-1 test matrix was run, following the PBX 9502 test series. Analyses of the PBX 9502 data indicated that shorter tests, on the order of one week, would be sufficient to characterize the data. Test durations of one week under load followed by a three-day recovery period was chosen for the LX-17 experiments. Loads and test temperatures were approximately the same as those used in the PBX 9502 test series.

Each test periodically recorded time, LVDT displacements, extensometer strains, the specimen temperature, and the room temperature outside the chamber at a rate of 100-Hz (during load-up) to 0.0167-Hz for the extensometers and at 0.1-Hz for the thermal and LVDT measurements. The room temperature data was used to investigate the possible correlations between room temperature fluctuations and strain and temperature fluctuations in the sample.

RESULTS AND ANALYSES

The data for each test was analyzed and the “false strain” in the LVDTs was removed using the average of the extensometer readings as the source for the actual load-up strain. The data were compared and certain behavioral characteristic trends were noted. One such trend was the strong creep rate dependence on temperature. Another was the relatively greater strain seen in the LX-17-1 elevated temperature tests compared to that seen in comparable elevated temperature PBX 9502 tests. The 70˚C, 780-psi test resulted in failure of LX-17-1 at approximately 135-hrs, while the sample of PBX 9502 did not show any signs of impending failure at the same stress and temperature. Fig. 10 shows the strain versus time data for the PBX 9502 matrix and Fig. 11 shows the data for the LX-17-1 matrix.

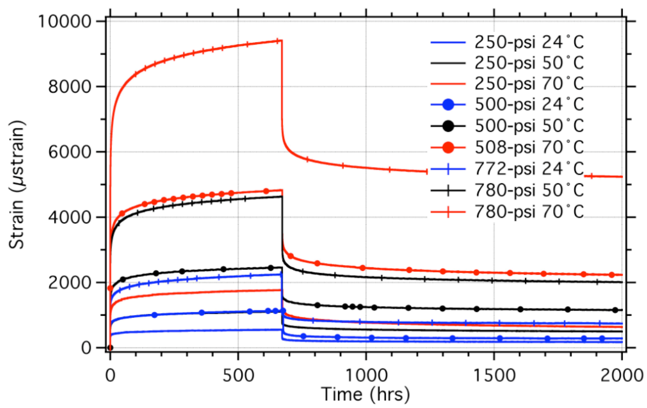

FIGURE 10: PBX 9502 creep test matrix results showing strain versus time. Creep rate dependence on temperature and stress are apparent in the plots. The data for the 250-psi, 50˚C creep period and the 500-psi, 24˚C creep period overlay on the plot.

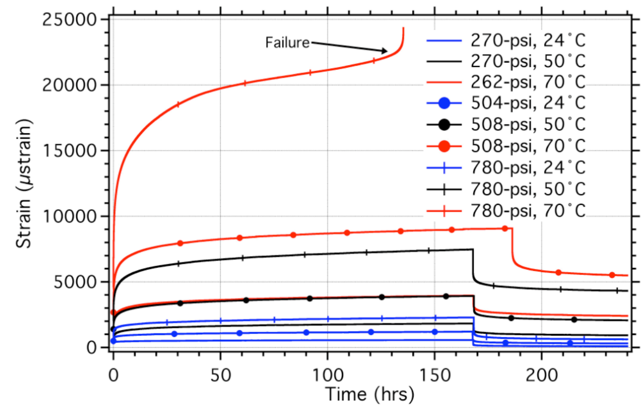

FIGURE 11: LX-17-1 creep test matrix results showing strain versus time. The sample tested at 70˚C, 780-psi failed in ~135-hrs.

The creep and recovery portions of the strain versus time curves show logarithmic behavior and can be fit with the equation:

$$\varepsilon(t) = C_1 + C_2 Log(t) \quad (1)$$

Each curve was analyzed and the coefficient C_2, referred to henceforth as the creep slope, was used as a metric to compare the creep rate, test to test. The creep slope for each test was tabulated and the data for the two test matrices were plotted on isothermal curves shown in Fig. 12 and Fig. 13. The isothermal curves were then fit with second order polynomial fits, shown on the plots (solid lines for PBX 9502, and dotted lines for LX-17-1).

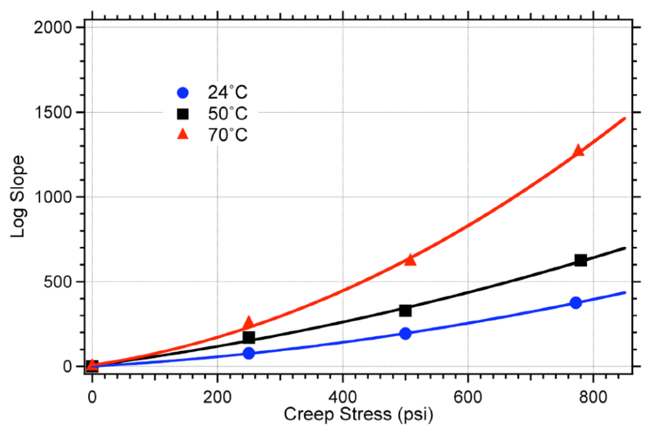

FIGURE 12: Log slope versus creep stress for PBX 9502 test matrix. The solid line curves shown connecting the data points are second order polynomial fits.

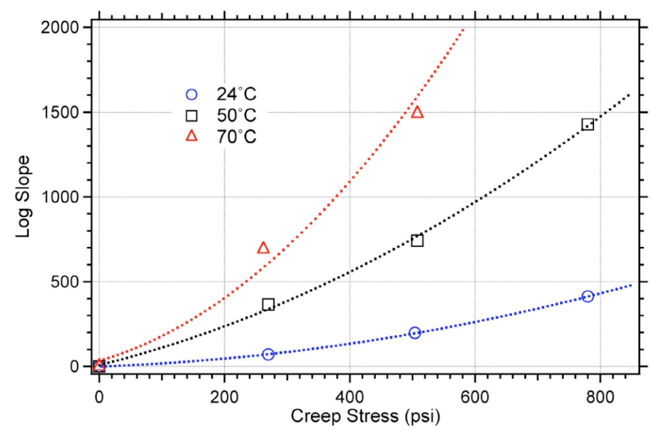

FIGURE 13: Log slope versus creep stress for LX-17-1 test matrix. The dotted line curves shown connecting the data points are second order polynomial fits. The 70˚C, 780-psi data point is not shown because that specimen failed.

DISCUSSION

LX-17-1 contains 1.5 times the amount of binder that PBX-9502 does and exhibits greater creep strain accumulation at temperatures above the binder T_g (~30˚C). This data shows that when the materials were tested below the T_g, the creep slopes for LX-17-1 and PBX 9502 tended to overlay. However, as the test temperature increased, the creep slopes at comparable stresses began to diverge as a function of temperature, with LX-17-1 showing notably higher strain rates than PBX 9502. This difference was highlighted at the highest stress level when the LX-17-1 failed.

Experimentation at LLNL and at LANL has shown that while log fits work remarkably well for TATB-based PBXs, the same does not hold true for all PBX materials [14]. For example, PBX 9501 (95% HMX, 2.5% Estane, 2.5% BDNPA-F) experiences creep accumulation that is not logarithmic [7]. The reasons for the difference could be associated with the explosive crystal difference, the different binder, the presence of a plasticizer or any combination of reasons. Future investigation is needed to determine an appropriate means of comparison between dissimilar PBXs.

SUMMARY

The creep behaviors of PBX 9502 and LX-17-1 have been measured at three load levels and at three test temperatures using a custom designed creep system. Strain measurements were taken with two gauge types, each used to best utilize its measurement capabilities. Contact extensometers ensured that strains were recorded accurately during the periods of rapid strain change that occur during the load-up and unload phases of the experiment. The LVDTs, being relatively more reliable over long periods of time, were used to ensure the data was accurate for tests lasting several months. Comparative testing has shown that after removing the "false strain" from the load-up portion of the test, the two gauge sets correlated well. Furthermore, a comparison of redundant tests using multiple samples of similar material has validated the system.

Logarithmic curves have been found to accurately fit the PBX 9502 and LX-17-1 creep data. The factor described as the *creep slope* has been found to be an effective means of comparing the creep rates of the same or different materials, assuming their creep behavior is logarithmic, under various thermal and stress conditions. The resulting data from the two three-by-three test matrices has produced three isothermal creep slope versus creep stress curves that can serve as a tool for material comparison and model development.

AKNOWLEDGEMENTS

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