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PBX 9502 TENSILE ANALYSIS

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INTRODUCTION

Tensile testing of PBX 9502 specimens is required for both qualification and surveillance testing at Pantex (PX). Individual and average ultimate stress and strain values are reported for surveillance with the averages of the qualification data for the same PBX 9502 lots. Qualification and surveillance data are compared for potential age-related trends. In May 1997, the averages of the surveillance stress and strain values for two Cycle 15 units were compared to the qualification stress-strain averages. The comparisons showed higher stress and lower strain values and suggested an age-related mechanical property change. With the recent creation of the HE Core Surveillance Database [1], individual specimen surveillance values can be easily compared to the corresponding individual qualification values to evaluate for trends. This paper describes the past tensile testing methods, changes in equipment and test procedures resulting in a broad scatter observed in stress-strain values. Using the available HE surveillance database, the surveillance measurements are shown to fall within the range of qualification stress and strain values recorded previously for all PBX 9502 lots.

BACKGROUND

Historically PBX 9502 tensile testing at PX has been conducted using three different materials-testing workstations and two different gripping methods. Also, strain measurements have been obtained with different extensometer methods. The consequence of these changes is that data have been recorded using 4 different test methods described as:

(1) Old Instron with glued end caps,
(2) Old Instron with current collar clamp method,
(3) Tinius-Olsen with current collar clamp method, and
(4) Instron Frame Model 1331 upgraded to an 8562 model with current clamp method.

Test method 1 required that the glued end have a consistent and evenly bonded surface to establish uniaxial tension across the gauge length of the specimens. If the glued end began to fail, uniaxial conditions were not achieved and the data was invalid. The current gripping method (used for test methods 2, 3, and 4) uses aluminum collars to surround the conical dogbone ends. No lubrication is used with the method. These are mounted into conical clamps, which are attached to the crosshead and the stationary platform of the test workstation. Specimens are manually mounted so that the grips are initially somewhat loose and no additional tension is applied to the specimen. The specimen is "seated" with a preload up to 1 lb, which is released back to a lower value ranging from 0.004 to 0.55 lb. A foam insert is used under the bottom clamp to provide support to the clamp once the tensile specimen has been broken.

Calibration occurs on a yearly basis. Throughout the history of PBX 9502 tensile testing, no specification was established for a calibration standard to be used in the transition from method-to-method to evaluate for the possibility of subtle systematic test changes or for normalization of the test data on a day-to-day basis. Neither can the methods be resurrected because the older test workstations have been removed from the facility. Hence, inherent differences in the data from method-to-method cannot be quantified.

Past core surveillance specifications required tensile tests to be performed using crosshead speed control at room temperatures, ~23±2°C, without humidity control or monitoring. Six to 8 cylindrical-dogbone, tensile specimens were machined from the forward charge, dependent on charge dimensions. Machining for tensile specimens required core cuts penetrating through the curved portion of the charge. The cores are removed and cut on a

lathe to the final dogbone dimensions with an internal gauge diameter of 0.5-in. and gauge length of 1.5-in.
PBX 9502 is nominally composed of 95 wt% triamino-trinitrobenzene (TATB) and 5 wt% Kel-F 800™ binder. TATB (T_{melt} ~448°C) is a triclinic crystal with a micaceous, plate-like morphology that is susceptible to preferential orientation (texturing) during processing. Specifically, the plate normals are aligned perpendicular to the direction of an applied shear stress or parallel to compressive loading. The dimensions of the TATB crystals are on the order of 10-50 microns in diameter and sub-micron to 10 microns in thickness. The glass-transition temperature (T_g) of PBX 9502 is slightly less than the T_g of Kel-F 800™ indicating little interaction between the binder and the TATB. Kel-F 800™ is an amorphous, 3:1 copolymer of chlorotrifluoroethylene and vinylidene fluoride with a T_g of ~28°C, a melting temperature of ~105°C, and an ambient specific gravity of 2.0 g/cm³. [2]

The longitudinal and transverse loading orientations (due to preferred orientation of TATB) and virgin versus recycled TATB have significant effects on the properties of PBX 9502. [3-7] Significant mechanical properties differences are also evident with slight density variations in specimens and when testing at temperatures close to the glass-transition temperature range. The glass-transition temperature range is also dependent on strain rate, relative humidity, temperature gradient in the specimen, and polymer molecular weight properties. Polymeric aging factors, e.g. increased crystallinity, could also potentially influence mechanical property behavior with time.

Twenty-two PBX 9502 lots have been used in stockpile systems. Lot numbers with 890 and 891 correspond to the virgin and recycled lots, respectively. The 891 recycled lots were formulated with 50% new material, and 50% of PBX 9502 machine cuttings. Lots 00E135 and 00E136 are also referred to as recycled lots, but these were formulated with 50% reclaimed TATB mixed with 50% molding powder from one of three virgin lots, HOL81D890-008, HOL81D890-009, and HOL81H890-012. [8]

From May 1979 to February 1990 17 virgin lots and 10 recycled PBX 9502 lots were tensile tested for qualification with 3 of the test methods described previously. These data are shown graphically in Figures 1 to 3. The data plots show that the number of lots and, types of lots tested coupled with different tests periods and lack of normalization have contributed to a wide range of stress-strain values measured during lot qualification. Strain % and stress data ranged from 0.158 to 0.394%, and from 964.50 to 1647.63 psi respectively. It is also evident by the data in Figure 3 that in general, the recycled lots, both 891- and 00E13# types, tend to produce higher tensile strengths than the virgin materials.

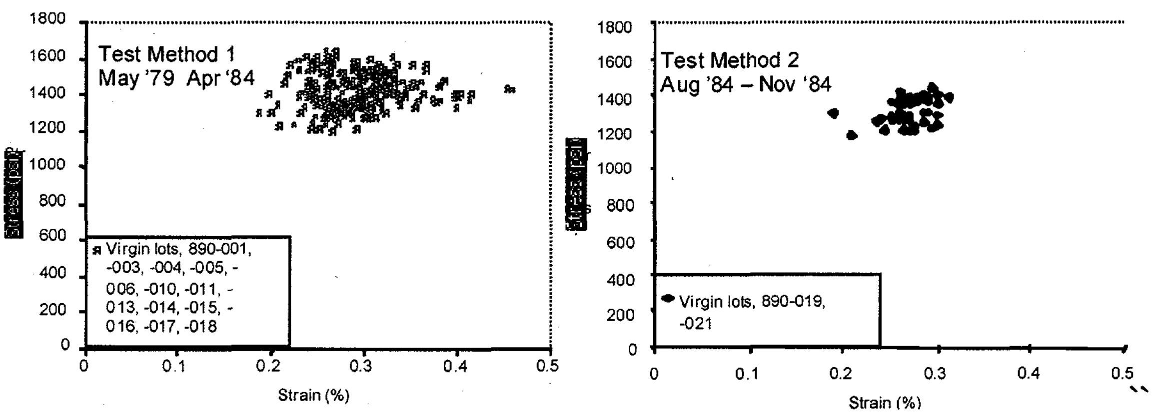
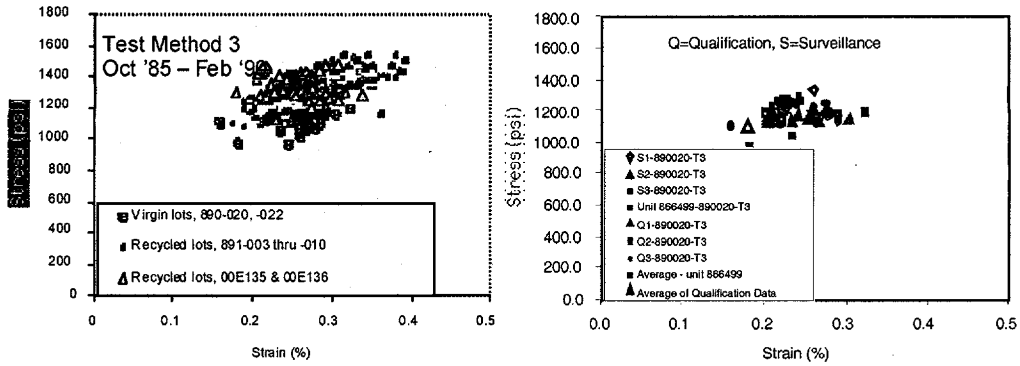


Figure 1 and 2: PBX 9502 ultimate tensile stress-strain lot qualification data obtained on individual specimens with Test Method 1 and 2 respectively. The lot numbers are provided in the legend of the graph.



Figures 3 and 4: [#3] PBX 9502 ultimate tensile stress-strain lot qualification data obtained on individual specimens with Test Method 3. The recycled lots tend to have higher ultimate strengths than the virgin lots. Lot HOL83J890-020 was qualified with Test Method 3. [#4] PBX 9502, lot HOL83J890-020 tensile qualification (Q) and surveillance (S) data for different units obtained with Test Method 3.

ANALYSES AND DISCUSSION

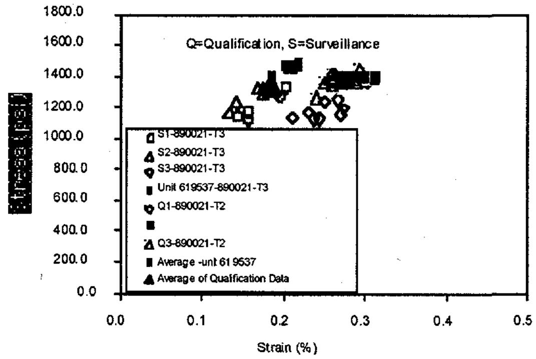
The two PBX 9502 lots, HOL83M890-021 and HOL83J890-020, from the Cycle 15 units were qualification tested with Test Methods 2 and 3, respectively. To date only Test Method 3 has been used for core surveillance tensile testing of these two units.

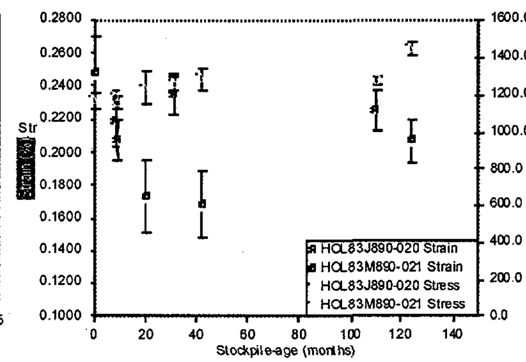
Additional tensile data from surveillance units manufactured with these same 2 lots of PBX 9502 are plotted in figures 4 and 5. All of these data were obtained with Test Method 3. Besides lot-to-lot differences these specimens vary both in density and stockpile age as well as stress and strain. A review of the data shows

- scatter in the stress and strain values in both the qualification (Q) and surveillance (S) data for both PBX 9502 lots HOL83J890-020 and HOL83M890-021, and
- the individual specimen data from unit 866499 does not exceed the stress or strain ranges measured for the qualification data, but the averages give the appearance of a difference, and some of the individual specimen data from unit 619537 appear to have higher tensile strength or lower tensile strain than the qualification data but they were tested with different methods.

All of the PBX 9502 qualification data were used to examine the distribution of stress and strain data for the different test methods. Because the measurement distributions exhibit different amounts of scatter, it is important to consider which test method was used when comparing data. Preliminary statistical modeling of stress and strain as individual univariate responses was performed to assess potential differences between qualification and surveillance data, while accounting for multiple measurements from the same pressing. Further analysis would be required to properly model stress and strain as a bivariate response.

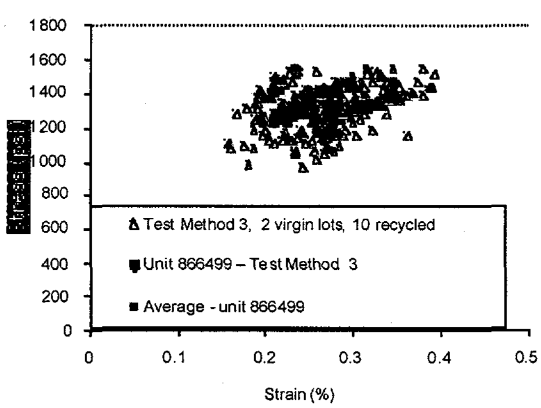
Figure 6 is a graphical comparison of the stress and strain averages of the all of the surveillance units to-date manufactured from the same two PBX 9502 lots as a function of stockpile aged. Error bars represent the calculated standard deviation values. A comparison of the stress behavior (shown in green symbols) shows a possible trend to slightly higher stress with stockpile age if the last average of 1448.9±40.6 psi at 124 months is considered significant. Notably this stress value does not exceed the highest stress, 1674.63 psi, measured previously for PBX 9502 during qualification testing. A more rigorous analysis of the data as a function of time would include an error assessment, which incorporates error in the individual data points as well as error in the estimated regression line. Since the observed data appears to be within the range of qualification values, and since increased stress is not considered to have a significant impact at this time, such an analysis has not been pursued. Possible trends in strain behavior are considered potentially more significant due to the reliability requirement of the charges to maintain size and shape during use. It is immediately apparent from a review of the strain data (shown in blue symbols) that there is no significant trend in the strain behavior of either PBX 9502 lot, or as a collection.

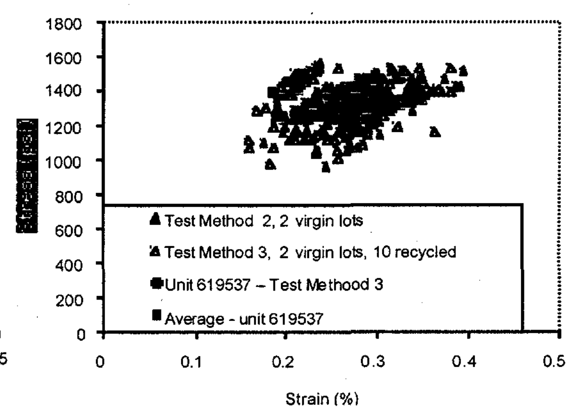




Figures 5 and 6: [#5] PBX 9502, lot HOL83M890-021 tensile qualification (Q) and surveillance (S) data for different units obtained with Test Methods 2 and 3 respectively. [#6] Average tensile stress and strain values for the two different lots of PBX 9502. Error bars represent the standard deviation measured for the total number of specimens tested. The strain axis is shown to the left, and stress on the right.

Additional comparisons of the surveillance data from units 866499 and 619537 with the qualification data for PBX 9502 lots tested with the same methods are shown in Figures 7 and 8. These graphs show that the individual specimen and average data did not exceed the tensile stress, or drop below the tensile strain previously measured for PBX 9502 lots during qualification testing. Thus there is no apparent trend related to stockpile age in the tensile stress-strain behavior of the specimens taken from these two units.





Figures 7 and 8: [#7] PBX 9502, lot HOL83J890-020 tensile stress-strain core surveillance data (individual and average) for unit S/N 866499 compared to lot qualification data obtained with Test Method 3. [#8] PBX 9502 tensile stress-strain core surveillance data (individual and average) for unit S/N 619537 compared to lot qualification data obtained with Test Methods 2 and 3.

New changes for future core surveillance testing specifications are under consideration to minimize scatter in the data as follows:

- include a calibration standard for normalization from method to method
- lower the test temperature to -20°C to avoid mechanical properties measurements in the quasi-static glass-transition temperature range,
- monitor and record humidity for each test, and
- determine if different sampling methods may identify potential trends.

CONCLUSIONS

With the recent creation of the PX HE Core Surveillance Database, individual specimen surveillance values can be easily compared to the corresponding individual qualification values to evaluate for trends. A review of the data shows a broad scatter in measured stress-strain values. Using the available HE surveillance database, it is clear that the surveillance measurements from the two Cycle 15 charges fall within the range of qualification stress and strain values recorded previously for PBX 9502 lots and that no apparent stockpile-age related trends are evident in the tensile stress-strain data. As a result of this investigation, some changes are being made to the core surveillance specifications to minimize the effects on tensile data scatter due to temperature and humidity differences and method to method changes.

These data analyses do point out the need for a comprehensive understanding of the effect of a number of variables, i.e. formulation and pressing method, density, stockpile age, lot-to-lot variations, temperature, and humidity on the mechanical property behavior of HE composite materials. Too often data have been compared without the relevant details made available to determine if the test conditions were nominally the same or different. These results also point out the critical need to establish useful stress-strain limits for qualification and surveillance testing of HEs.

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