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Compacting Plastic-Bonded Explosive Molding Powders to Dense Solids

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Bart Olinger

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Compacting Plastic-Bonded Explosive Molding Powders to Dense Solids

by

Bart Olinger

Abstract

Dense solid high explosives are made by compacting plastic-bonded explosive molding powders with high pressures and temperatures for extended periods of time. The density is influenced by manufacturing processes of the powders, compaction temperature, the magnitude of compaction pressure, pressure duration, and number of repeated applications of pressure. The internal density variation of compacted explosives depends on method of compaction and the material being compacted.

Introduction

This is a compilation of studies done to understand the effects various compaction variables have on the density of the plastic-bonded explosives (PBX). The explosives were compacted to high density to maximize the explosive energy in a given volume, to give the explosives strength to prevent breakup under severe shock-and-vibration conditions, to give the explosive predictable, reproducible detonation characteristics, and to make the explosives machinable using water as a coolant and lubricant.

PBX 9501 Molding Powder

The kinetic energy source for the plastic-bonded explosive PBX 9501 is HMX with an energy release of 1.48 kcal/g and a density of 1.905 g/cm^3. Formulation of molding powder begins with the suspension of HMX particles of two size distributions, 3 parts by weight coarse (class 1) and 1 part fine (class 2), in agitated water. A solution of the plastic binder constituents, Estane™ and nitroplasticizer dissolved in methyl ethyl ketone, is added. (Nitroplasticizer is mixed into Estane™ to increase the binder's strain-to-failure ratio, which subsequently decreases the explosive's sensitivity.) After further heating and agitation, the solvent is drawn off, causing the binder mix to precipitate onto the HMX particles that then agglomerate into granules, the molding powder. The molding powder is made in 500-pound batches. Many batches are blended together into a lot so that any part of it should be like another. The weight percentages in the granules are 95% HMX, 2.5% Estane™, and 2.5% nitroplasticizer. The calculated density of the HMX and Estane™-nitroplasticizer mixture without voids and contaminants is 1.859 g/cm^3, the PBX 9501 theoretical density.

PBX 9502 Molding Powder

The kinetic energy source for the plastic-bonded explosive PBX 502 is TATB with an energy release of 1.20 cal/g and a density of 1.937 /cm^3. Formulation of this molding powder begins with the suspension of TATB particles in agitated water. The plastic binder, Kel-F-800™, dissolved in ethyl acetate, is added. After further heating and agitation, water is added, causing the binder to begin to precipitate onto the TATB particles. Then the solvent is drawn off, increasing the precipitation and causing agglomeration into granules, the molding powder. The eight percentages in the granules are 95% TATB and 5% Kel-F-800™. The calculated density of the TATB and Kel-F™ without voids and contaminants is 1.942 /cm^3, the PBX 502 theoretical density. The PBX 502 used by Los Alamos National Laboratory is a 50:50 combination of reconstituted PBX 502 machining scrap and new material.

Physical Properties of Molding Powders

The weights and densities of PBX 501 and PBX 502 molding powder granules were measured and listed in Table 1. These values will vary from sample to sample, but they give an indication of the differences between the two molding powders.

Table 1. Molding Powder Properties of PBX 9501 and PBX 9502

Explosive	PBX 9501	PBX 9502
Lot	RON00H144 - 001	HOL88A891 - 006
Theoretical Density, g/cm^3	1.859	1.942
Required Pressing Density, g/cm^3	1.830 minimum	1.890 ± 0.005
Powder Granule Density, g/cm^3	1.668	1.401
Granule Packing Density, g/cm^3	0.952	0.943
Granules per gram	143	1,050
Average and deviation, g/100 granules	0.68 ± 0.12	0.094 ± 0.020
Granule Packing Efficiency	57.1%	67.3%
“Spherical” Granule Diameter, mm	2.00	1.09

Molding Powder Preparation for Compaction

The molding powder is prepared for compaction by being heated and evacuated. Heating makes the binder materials more fluid and susceptible to welding granules together under pressure. Temperature is limited by the decomposition of the binder components. Evacuation removes gases that remain inside and around the particles, eliminating gas pressure resistance to compaction.

Density Determination

Densities of the compacted explosives were calculated from weights before and during immersion in water with density known from its temperature. The compacted explosives were given sufficient time to come into thermal equilibrium with the water. The balances used had a precision of 1 part in 10,000. Corrections were made based on simultaneous measurements of the density of similar masses of standard single-crystal silicon. All densities were corrected to 20°C.

Specimen Material and Size

The first density measurements for PBX 9501 were made with 1-5/8-in.-diameter and 2-3/16-in.-long cylinders weighing 136 g. Some of these measurements were repeated with a second press that compacted 4-in.-diameter and 4-in.-long cylinders weighing 1,800 g, to investigate the dependency of the properties of the compacted plastic-bonded explosives on size and the press used. These measurements with the larger press were, in turn, repeated using PBX 9502.

Compaction Methods

Three types of presses were used to compact molding powder. The first is a steel-die press. One or two pistons slide into a thick-walled cylindrical sleeve, trapping and pressing the molding powder packed between them. Evacuation and heating is done in the press. Forms, called mandrels, can be stacked in the sleeve with the explosives to give the product specific shapes that minimize machining. The second press type is called isostatic or wet-bag. It uses a hydrostatic medium, usually water, to compact submerged molding powder enclosed in a thin rubber membrane. Mandrels are used; they are part of a pedestal to which the membrane is attached. The powder is evacuated before being placed in the press, and is heated before and during pressing. The third type is a hydrostatic or dry-bag press that also uses a hydrostatic medium, usually oil. The powder is hand-packed with tampers into a thick rubber-like mold. The press lid seals the mold opening and then evacuation occurs, followed by the application of pressure. The powder is heated only before packing. Mandrels are embedded in the molding powder. For the comprehensive compaction studies presented here, the steel-die presses were used because the sample sizes were much smaller than the hydrostatic pressed materials.

Internal Density Distribution

The densities throughout compacted cylinders are not uniform. The density distributions were determined for cylinders of PBX 9501 and 9502 that had aspect ratios similar to those used in the compaction studies to be discussed. Diameter-wide slabs were cut from along the axes of compacted cylinders of PBX 9501 and 9502. The slabs were marked with a grid, each element of the grid is labeled, and then all elements were cut free. The density of each element was then measured. A composite of the measured densities portrayed the density distribution in these cylinders, as shown in Figs. 1 and 2.

Both cylinders were compacted at 20,000 psi. The PBX 9502 molding powder was heated and pressed at 110°C for 5 min and the PBX 9501 at 90°C for 10 min. The relative motion of the piston into the cylinder was from the bottom upward. The direction determines the density distribution in the cylinders. The pressed PBX 9501 cylinder has a lower density region that forms directly above the center of the moving bottom piston face. Below the center of the stationary top is a higher density region. The density difference between the two is only 5 mg/cm^3.

The same low- to high-density distribution pattern along the axis was found in PBX 9502. But superimposed was a much greater density anomaly, a high-density region adjacent to the walls of the cylinder at the moving piston end. The densities along the cylinder wall range 40 mg/cm^3 from bottom to top, 8 times greater than for PBX 9501. The cause of this large density gradient has not been determined. The sliding friction with the steel cylinder walls may cause the tabular structure of the TATB crystals to reorient, to stack up, and subsequently reduce porosity.

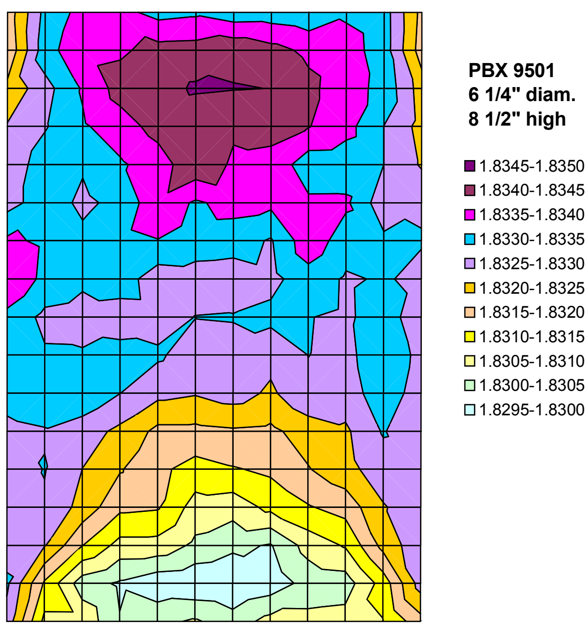

Figure 1. Density distribution in a cross-section slice of a 6-1/4-in.-diameter, 8-1/2-in.-high compacted cylinder of PBX 9501. The densities range from 1.8295 to 1.8350 g/cm^3, or 0.3% variation.

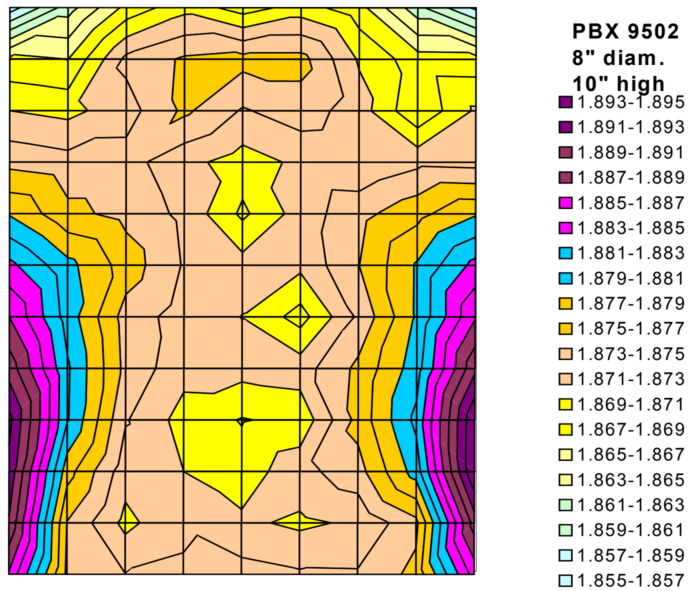

Figure 2. Density distribution in a cross-section of an 8-in.-diameter, 10-in.-high compacted PBX 9502 cylinder. The densities ranged from 1.855 to 1.895 g/cm^3, or 2.1% variation.

Characteristics of Dwell

The effect of time held at pressure (the dwell) was studied for PBX 9501, Lot BAE02D145-004. The compacting pressure transmitted by a 1-5/8-in.-diameter piston was 20,000 psi, and the powder was heated at 90°C before and during compaction. Densities of the recovered solid explosives and the time they were held at pressure are plotted in Fig. 3. The figure shows that PBX 9501 compacts at a rate much slower than the time required applying the compaction pressure, usually several seconds. The compaction continued but at a decreasing rate with increasing dwell. This plastic-like behavior of PBX 9501 is significant; it means that there is no point in expending effort to apply pressure rapidly, and once at pressure, there is no hurry for releasing the pressure.

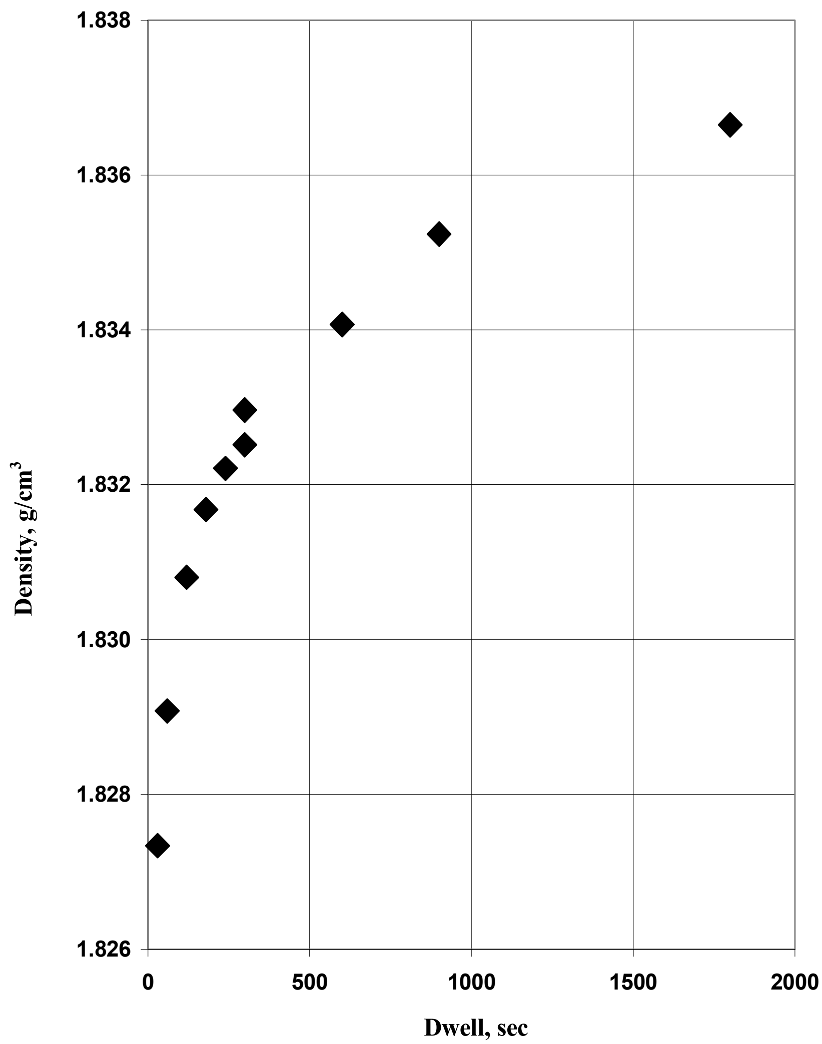

Figure 3. The density of PBX 9501 compacted to 20,000 psi and 90°C for the various lengths of time held at pressure, called dwell.

Linear Relation between Density and the Log of Dwell Time

In Fig. 4 the density data are plotted with the natural logarithms of their dwell times in seconds. The data appear linear:

$$\rho_x = a + b \times \log(dwell) \ , \tag{1}$$

where ρ_x is the density of the explosive. This same phenomenon is observed for PBX 9502 and with compactions done in other presses. The linear approximation allows the compaction densities involving other variables to be specified for a large range of dwell times from only two data points. The linear relation between density and the log of dwell extrapolates to infinite density, however, which not physically possible.

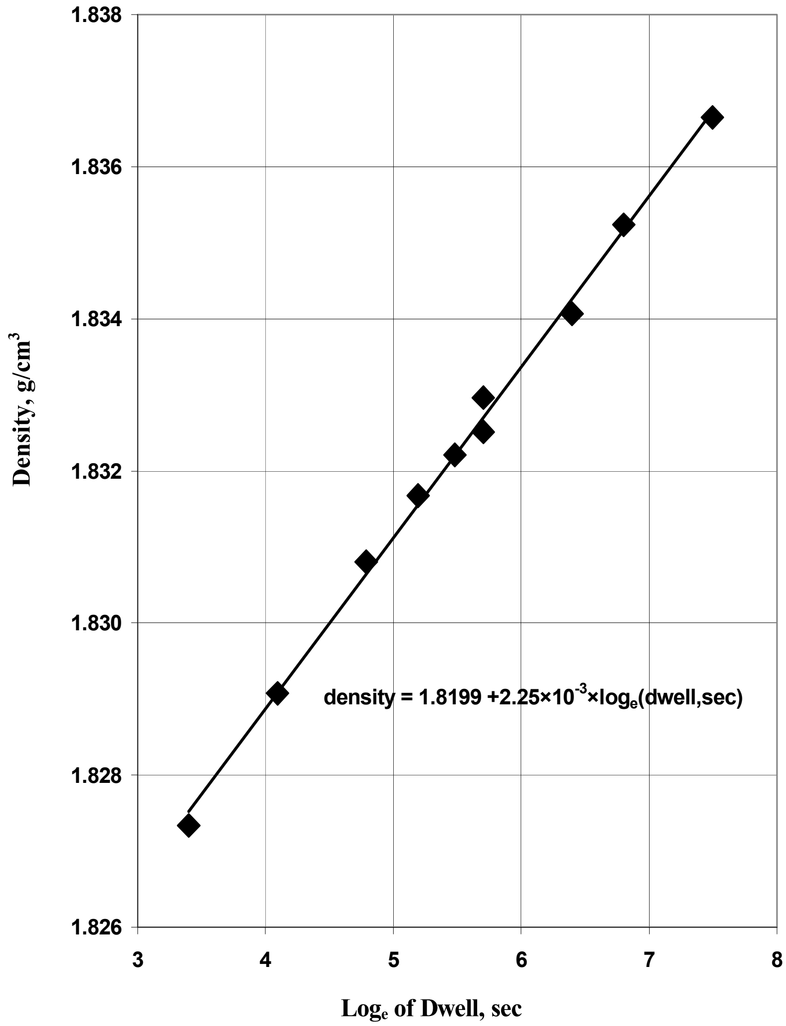

Figure 4. The density of PBX 9501 compacted to 20,000 psi and 90°C plotted against the natural logarithm of dwell.

Porosity and Density

Though density is the property by which PBX compaction is judged, the physics of compaction is driven by porosity. As the porosity approaches zero, achieving higher densities becomes more difficult; it requires more time at pressure, or more pressure, or increased temperature, or more applications of pressure. The maximum density, the theoretical density with zero-porosity, is calculated from the explosive components, 1.859 g/cm^3 for PBX 9501 and 1.942 g/cm^3 for PBX 9502. The porosity fraction, F_p, is

$$F_p = \frac{Theoretical\ Density - Density}{Theoretical\ Density} \quad . \tag{2}$$

As the dwell approaches infinity, F_p approaches zero, or the reciprocal of the porosity fraction, $1/F_p$, approaches infinity. Unlike the density expression above, this is physically possible. In Figs. 5 and 6 are the same data as in Figs. 3 and 4, but now the density is in the form of the reciprocal of the porosity fraction. These figures show that although $1/F_p$ extrapolates to the correct value as the dwell goes to infinity, it has no obvious function with respect to dwell, and it is not linear with respect to the $\log_e$ of dwell.

Though the porosity fraction is recognized as the principal parameter for compaction, the linear behavior between measured densities and the log of their dwell time will continue to be used as an extrapolation tool within the practical limits of the presses. The fact that the measured densities do not meet expectations of response can be readily explained by looking at Figs. 1 and 2. These compacted explosives do not have uniform densities; the material does not yield to achieve homogeneous pressure inside during compaction. The bulk densities that are used in the analyses do not reflect the internal densities; they are averages.

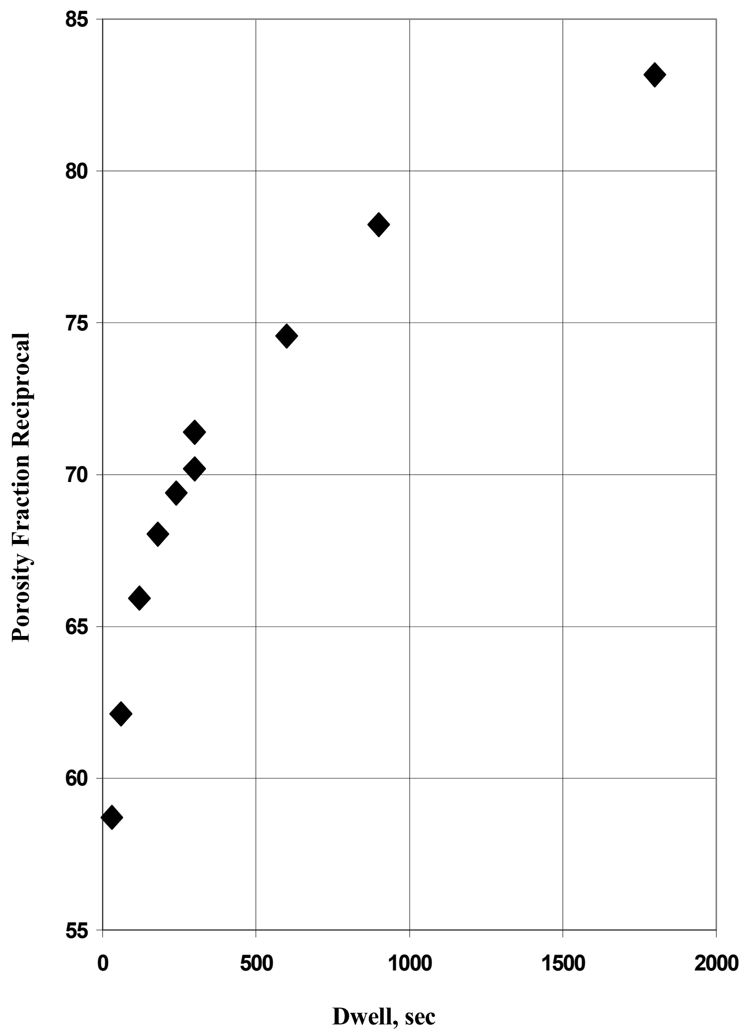

Figure 5. The porosity fraction reciprocal for PBX 9501 compacted to 20,000 psi and 90°C for the various dwell lengths.

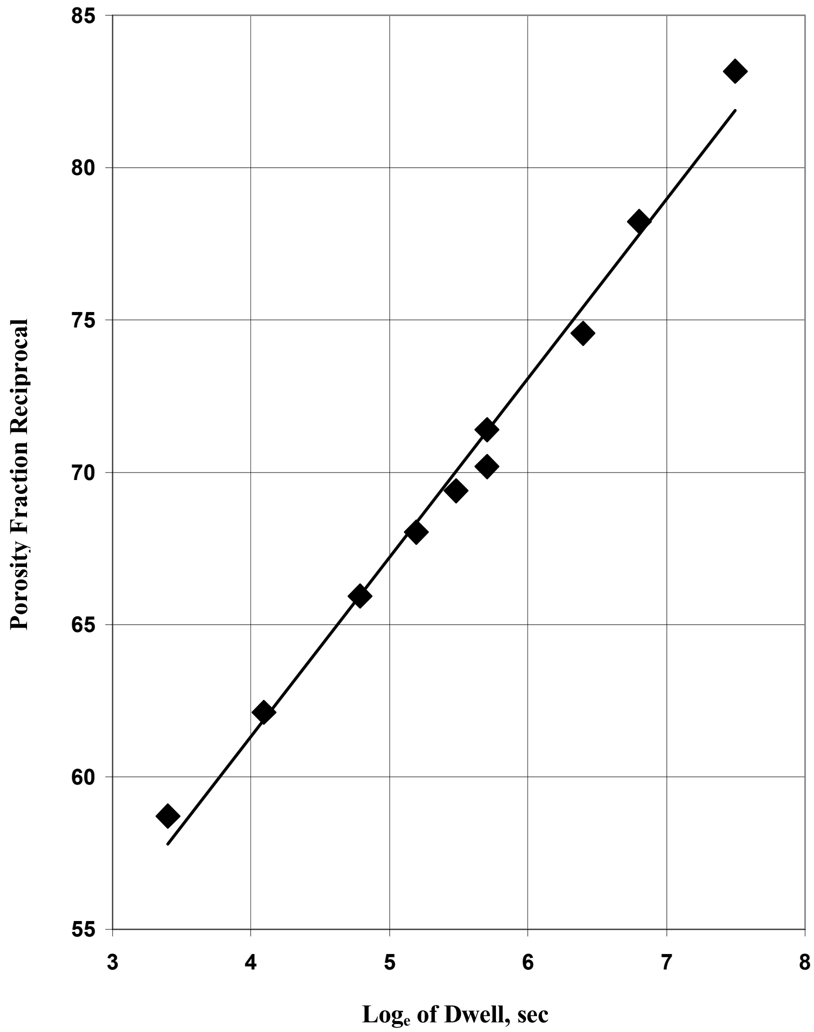

Figure 6. The porosity fraction reciprocal is plotted against the natural logarithm of dwell. The fit is linear, but the data are not.

Influence of Pressure on Compaction Density

PBX 9501 was compacted into 1-5/8-in.-diameter and 2-3/16-in.-long cylinders at 12,000, 14,000, 16,000, 18,000, and 20,000 psi with 200-, 600-, and 1,800-s dwell times. The powder and compaction temperature was 90°C. The measured densities are plotted in Figs. 7 and 8 with the dwell and the log of the dwell. The curves in Fig. 7 are from the linear fits to the data in Fig. 8.

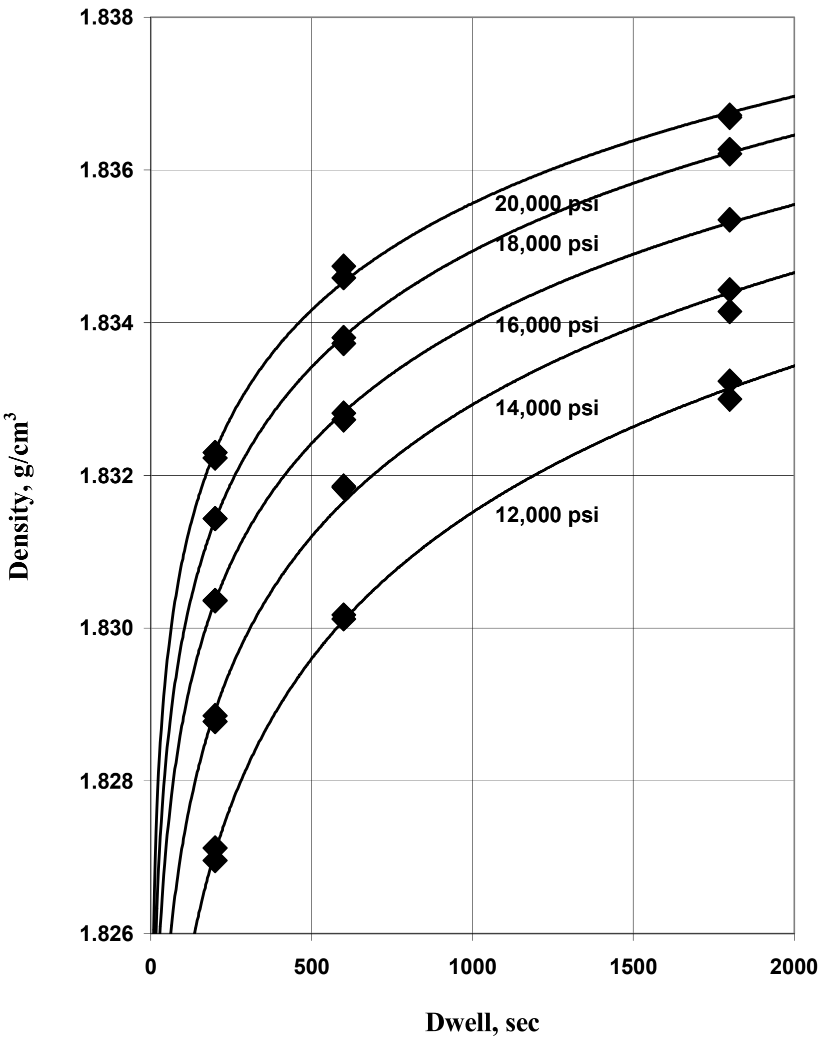

Figure 7. Densities of PBX 9501 compacted at different pressures and dwell times at 90°C, plotted as a function of the dwells. The curves are from the linear fits in Fig. 8.

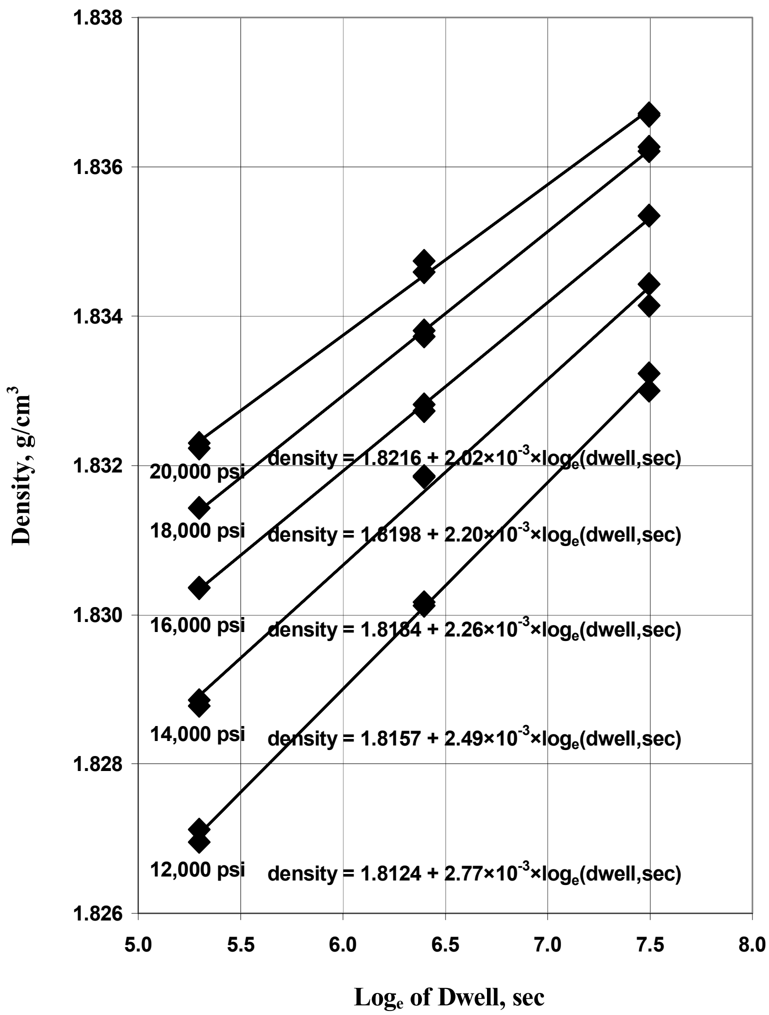

Figure 8. Densities of PBX 9501 compacted at different pressures and dwells at 90°C, plotted as a function of the log of the dwells. The linear fits to the data are listed.

Several phenomena are observed in these data. A given density can be reached in a shorter time at higher pressure. Higher compaction pressure reduces porosity more quickly. The plastic behavior observed earlier is shown to occur at many different pressures. Specifically, the linear relation between density and the log of dwell is found to hold over a wide range of compaction pressures.

From these data, another compaction relation can be found. For a given dwell, the density can be accurately estimated from the reciprocal of the compaction pressure by

$$\rho_x = c - \frac{d}{P_c} \quad , \tag{3}$$

where ρ_x is the explosive density, and P_c is the compaction pressure. From these data, for instance, for a 10-min dwell, c = 1.8406 and d = 0.1246, with P_c in units of k-psi.

Temperature Effect on Compaction

In Fig. 9 are plotted data for 200-, 600-, and 1,800-s dwells for the same lot of PBX 9501 compacted at 20,000 psi into 1-5/8-in.-diameter and 2-3/16-in.-long cylinders, but at two different temperatures, at the usual 90°C and then at 60°C. Apparently, the material strength resisting compaction is greatly diminished by a temperature increase from 60°C to 90°C. This is another characteristic of a plastic material. This data may help determine temperature-dependent yield constants for this viscoelastic material. A comparison shows that the 20,000-psi, 60°C fit is nearly identical to the compaction fit for 14,000 psi, 90°C, in Fig. 8. However, the effects of temperature and pressures are different. High temperatures soften the Estane™-nitroplasticizer binder, increasing the molding powder ductility or deformability and decrease the binder viscosity, increasing its ability to fuse when compacted under pressure.

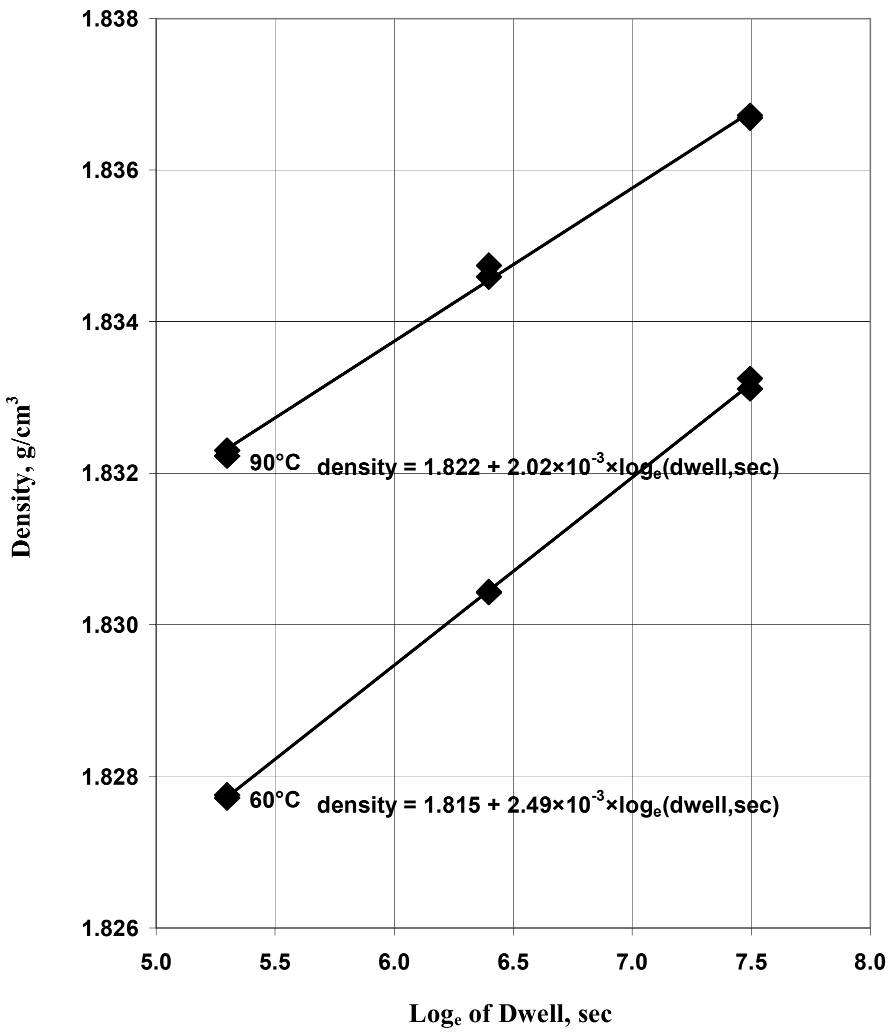

Figure 9. PBX 9501 compacted at 20,000 psi, heated before and during compaction to 60°C and 90°C.

Lot-Dependent Effects on Compaction

In dealing with explosive molding powders, a lot is a large quantity of molding powder, usually 5,000 to 10,000 pounds, that is made up of individual batches, usually 500 pounds each, made as described in the sections on molding powder. The batches are blended together to give the lot of molding powder uniform properties. The compaction characteristics of each lot can vary greatly.

In Fig. 10 the pressing characteristics of three lots of PBX 9501 molding powder are compared. These lots were all recently made by the Holston plant in Tennessee managed by British Aerospace Engineering (BAE). The lots were made from the same sources of HMX and plastic binder material. They are composed of different sets of batches. Molding powder from the three powder lots was compacted under the same conditions, 20,000 psi and 90°C, into 1-5/8-in.-diameter and 2-3/16-in.-long cylinders. The differences in the densities achieved between different lots are as great as those for the same lot compacted with 4,000 psi difference in pressure. As we shall see, a powder lot with poor pressing characteristics requires more time or more cycles to achieve a given density.

The fact that some powder lots are more readily compacted has been long recognized. But the reasons why are a mystery. There are as many explanations as there are people who compact explosives.

Dwell Required to Achieve Given Density for Various Conditions

The influence of pressure, temperature, and properties of the powder lot on the kinetics of compaction can be demonstrated by comparing the dwell times required to reach a particular density, in this case 1.836 g/cm^3, calculated from the linear fits shown in the figures above. Tables 2, 3, and 4 show the dwell times required for different lots to produce 1.836 g/cm^3 density at different compaction pressures and temperatures.

The reciprocals of the dwell times are a measure of the plastic flow of the plastic-bonded explosive as the pores collapse. For example, for each 2,000 psi increase in compaction pressure, the rate of compaction is increased by 1.4 times. Increasing the temperature from 60° to 90°C speeds up compaction by almost 4 times. And differences in the properties of molding powders from different lots can slow down compaction 2 times.

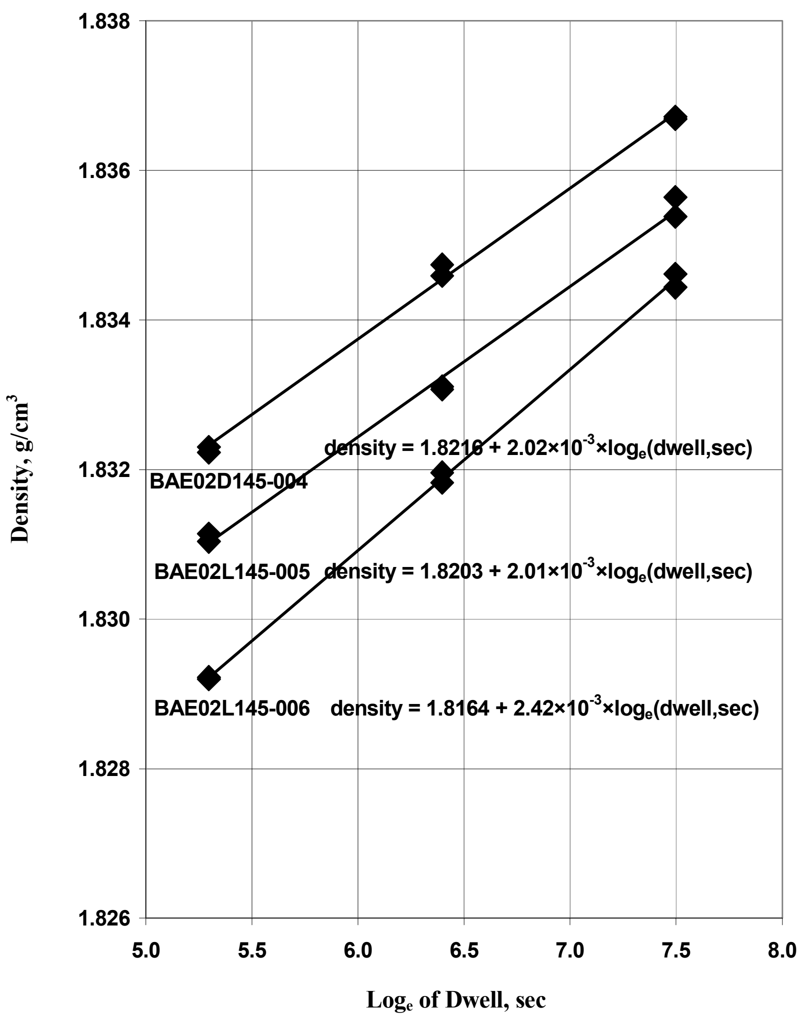

Figure 10. Three different lots of PBX 9501 made from the same sources of HMX and plastic binder material compacted at 20,000 psi and 90°C.

Table 2. Dwell Times to Reach 1.836 g/cm^3 at Various Compaction Pressures

Lot	Pressure (psi)	Temperature (C°)	Dwell Time (s)
BAE02D145-004	20,000	90	1,247
BAE02D145-004	18,000	90	1,578
BAE02D145-004	16,000	90	2,411
BAE02D145-004	14,000	90	3,473
BAE02D145-004	12,000	90	5,013

Table 3. Dwell Times to Reach 1.836 g/cm^3 at 90° and 60°C

Lot	Pressure (psi)	Temperature (Cº)	Dwell Time (s)
BAE02D145-004	20,000	90	1,247
BAE02D145-004	20,000	60	4,600

Table 4. Dwell Times to Reach 1.836 g/cm^3 with Different Molding Powder Lots

Lot	Pressure (psi)	Temperature (C°)	Dwell Time (s)
BAE02D145-004	20,000	90	1,247
BAE02L145-005	20,000	90	2,467
BAE02L145-006	20,000	90	3,292

Cycling

Once compacted, the means to achieve greater densities in the shortest time is through a compaction procedure called cycling. During cycling, at the end of a dwell, the pressure is released for a period of time, the rest, and then applied again, usually to the same pressure used for the first dwell and as long as the first dwell. In order to understand what effect each step of the cycling process has on the density of PBX 9501, a sequence of a 300-s dwell, 300-s rest, and 300-s dwell was interrupted at successive times by ejecting the compacted PBX 9501 from the press. The compacting pressure was 20,000 psi and the temperature was held at 90°C at all times. As usual, the compacted cylinders were 1-5/8 in. in diameter and 2-3/16 in. long.

The densities of the explosive at various times along the two cycles are shown in Fig. 11. Data for the first 300-s dwell were taken from Fig. 3. Unexpectedly, during the rest period with no pressure on the explosive, the density increased with time. During the rest period, the time required for the increase in density is about the same as required for the first dwell period. During the second application of pressure, the second cycle, the density increased in a time much shorter than for the first dwell and for a much smaller increase in density, about 0.002 g/cm^3 or about 0.1%. However, when the density increase during the rest period of the two cycles is included, the total increase is 0.005 g/cm^3 or about 0.25%.

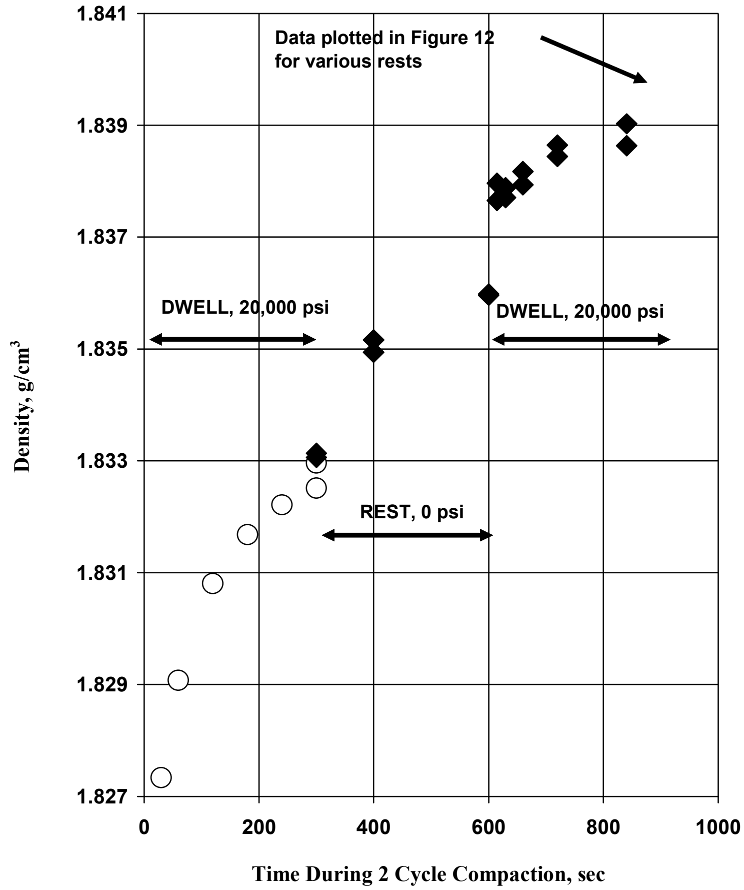

Figure 11. Densities of PBX 9501 compacted at 20,000 psi, 90°C, during a 300-s dwell, a 300-s rest at 0 psi, and a second 300-s dwell. Note the increase in density during the rest period with no pressure on the explosive.

Effect of Rest Period on Compaction

In Fig. 12 are plotted the densities achieved when the molding powder is compacted for 5 min at 20,000 psi, 90°C into 1-5/8-in.-diameter and 2-3/16-in.-long cylinders. Next, the pressure is released for rest periods of 15, 30, 60, 300, 600, and 1,200 s, and then 20,000 psi is re-applied for an additional 5 min. As the rest period increases, the densities at the end of the second dwell increase, rapidly at first, but diminish with longer rest periods.

The most efficient density increase is achieved after "resting" for a period roughly equivalent to the time required to compress the molding powder during the first dwell. In Fig. 12, the data from Fig. 11 are used to illustrate how the density increases from the end of the first 20,000 psi, 300-s dwell, during the 0 psi, 300-s rest period, and then increases again during the second application of 20,000 psi for 300 s. For the remainder of the data at longer times, the second dwell curve would remain the same, but the curve for the density-increase during the rest period would become larger. The data for the density at the end of the second cycle can be linearly fit with the logarithm of the length of the rest.

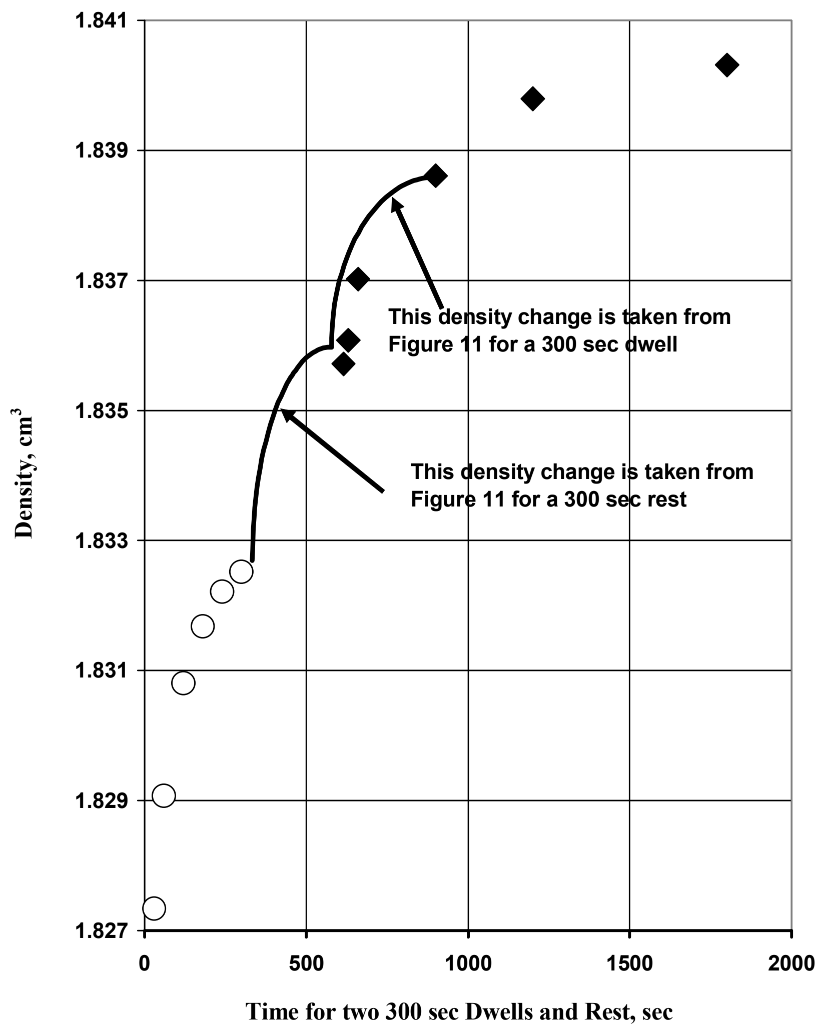

Figure 12. The data are the densities at the end of two cycles with a 300-s dwell, a rest period of increasing length, and a second 300-s dwell. The two curves show the locus of densities during the specific 300-s rest and the second 300-s dwell.

Effect of the Number of Cycles on Compaction

In Fig. 13 are plotted the densities measured for PBX 9501, Lot BAE02D145-004, compacted with 1 through 6 cycles, each cycle with a 300-s dwell and a 300-s rest. The PBX 9501 was compacted at 20,000 psi, 90°C, into 1-5/8-in.-diameter and 2-3/16-in.-long cylinders. These measurements are compared to the density of the same material for one continuous dwell. The largest gain in density is during the second cycle. Subsequent gains in density above the single dwell rapidly diminish. The re-application of pressure after the fourth cycle appears to gain little over compaction due to constant pressure.

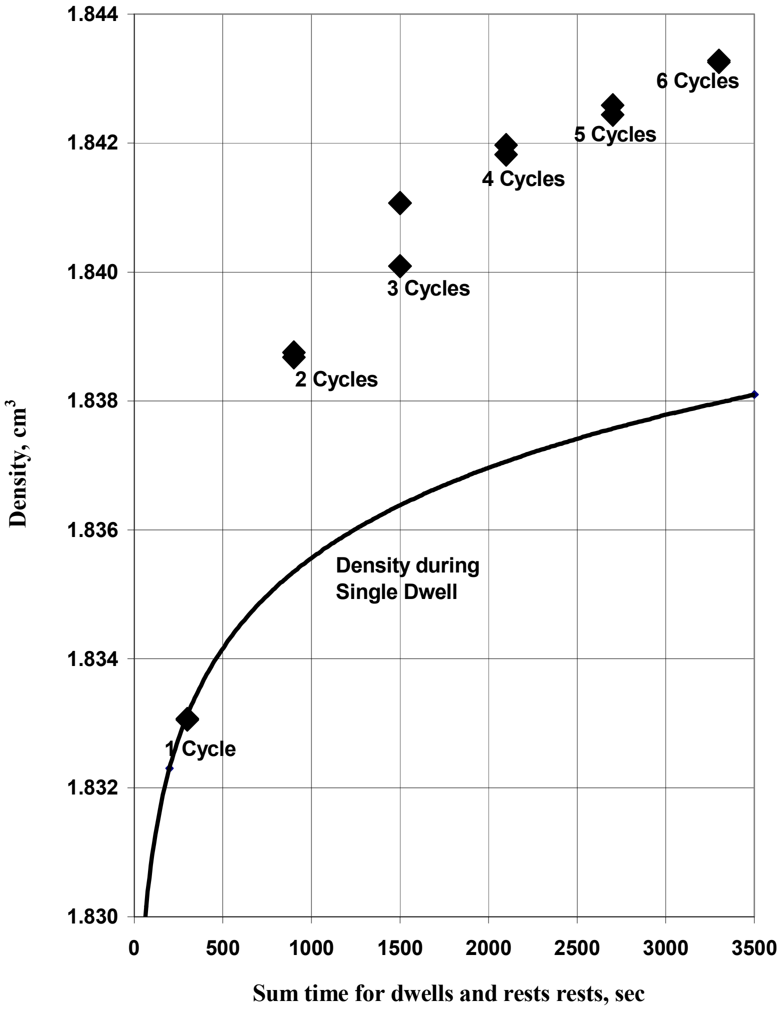

Figure 13. Densities achieved at the end of a sequence of dwells and rests. Compacting pressure was 20,000 psi and temperature was 90°C. Dwells and rests were 300 s long. The curve is the locus of densities during one dwell.

Efficient Distribution of Dwell and Rest Periods

It is obvious from these studies that one of the important parameters in compacting explosives molding powders is time in the press, whether pressing or resting. What is done with that time can optimize the density. In this study a specific period of time, 30 min, was used to subject the molding powder to different dwell and cycling periods, and then determine its density. As usual, the work was with the steel-die press that compacts PBX 9501 molding powder from Lot BAE02D145-004 into 1-5/8-in.-diameter and 2-3/16-in.-long cylinders weighing 136 g. The powders were heated before and during compaction to 90°C and compacted with 20,000 psi.

The results, shown in Table 5, agree with earlier observations that equal times for both dwells and rests maximize the density provided they are not less than the compaction time. In addition, the measurements support that the density increase during the rest periods is as great or greater than during the dwell periods. Surprisingly, explosives compacted with three 10-min dwells with 1-min rests were no denser than those with one 5-min dwell followed by a 25-min rest in the heated press. The densest explosive was produced by dwells and rests of equal length, three 6-min dwells with 6-min rests.

Table 5. Densities for Various Dwell and Rest Periods Lasting 30 Minutes

Dwell 1	Rest 1	Dwell 2	Rest 2	Dwell 3	Density
1,800 s					1.8366 g/cm^3
870 s	60 s	870 s			1.8386 g/cm^3
600 s	600 s	600 s			1.8403 g/cm^3
60 s	1,680 s	60 s			1.8390 g/cm^3
560 s	60 s	560 s	60 s	560 s	1.8395 g/cm^3
360 s	360 s	360 s	360 s	360 s	1.8406 g/cm^3
60 s	810 s	60 s	810 s	60 s	1.8397 g/cm^3
300 s	1,500 s				1.8395 g/cm^3

Additional Compaction Studies with Larger Cylinders of PBX 9501

The PBX 9501 compaction studies to this point were conducted with cylinders 1-5/8-in. diameter and 2-3/16 in. long weighing 136 g. They were repeated with 4-in.-diameter and 4-in.-long cylinders weighing 1,800 g to investigate what effect size has on the densities of compacted plastic-bonded explosives. For these studies, Lot BAE02L145-006 molding powder was used instead of Lot BAE02D145-004, which was used in the earlier described studies.

Figure 14 shows the densities of PBX 9501 plotted with linear functions of the log of the dwell for compacting pressures of 12,000 psi, 16,000 psi, and 20,000 psi. The densities are lower than in Fig. 8 because of the powder lot used and not due to the sample size, as is shown in Fig. 10.

Figure 15 shows the densities of PBX 9501 molding powder compacted into 4-in.-diameter and 4-in.-long cylinders at 20,000 psi, pre-heated and held in the steel-die press at 70°C, 80°C, and 90°C, and kept at those conditions for various dwells. Several data at 70°C and 80°C at higher dwells are not shown.

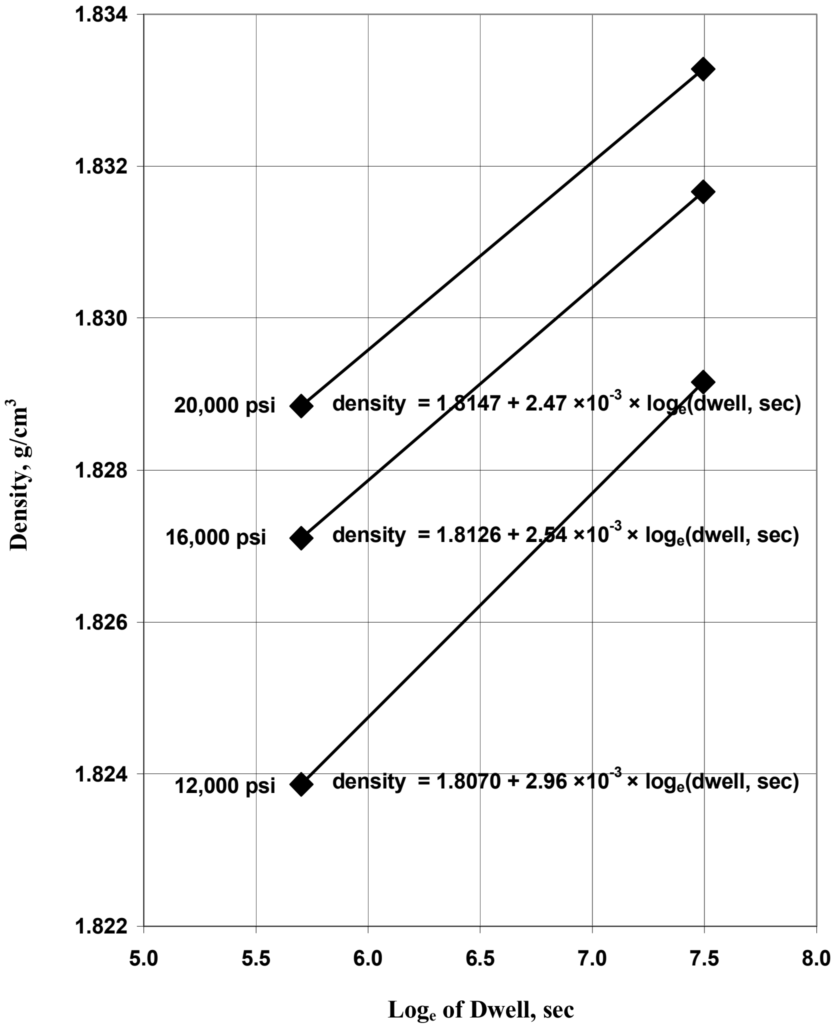

Figure 14. Three pressures were used for compacting PBX 9501 into 4-in. by 4-in. cylinders at two different dwells.

The relation between compaction density and the compaction temperature for a given dwell can be derived from the linear fits in Fig. 15. The densities for 300-, 600-, and 900-s dwells from the logarithmic fits for 70°C, 80°C, and 90°C are plotted in Fig. 16 as a function of temperature for given dwell times. In order to get a sense of the increase in the compaction density for an increase in the compaction temperature, a linear fit was forced through the calculated values. The slopes show the compaction density increases about 0.0002 gm^3 for each °C increase in powder temperature.

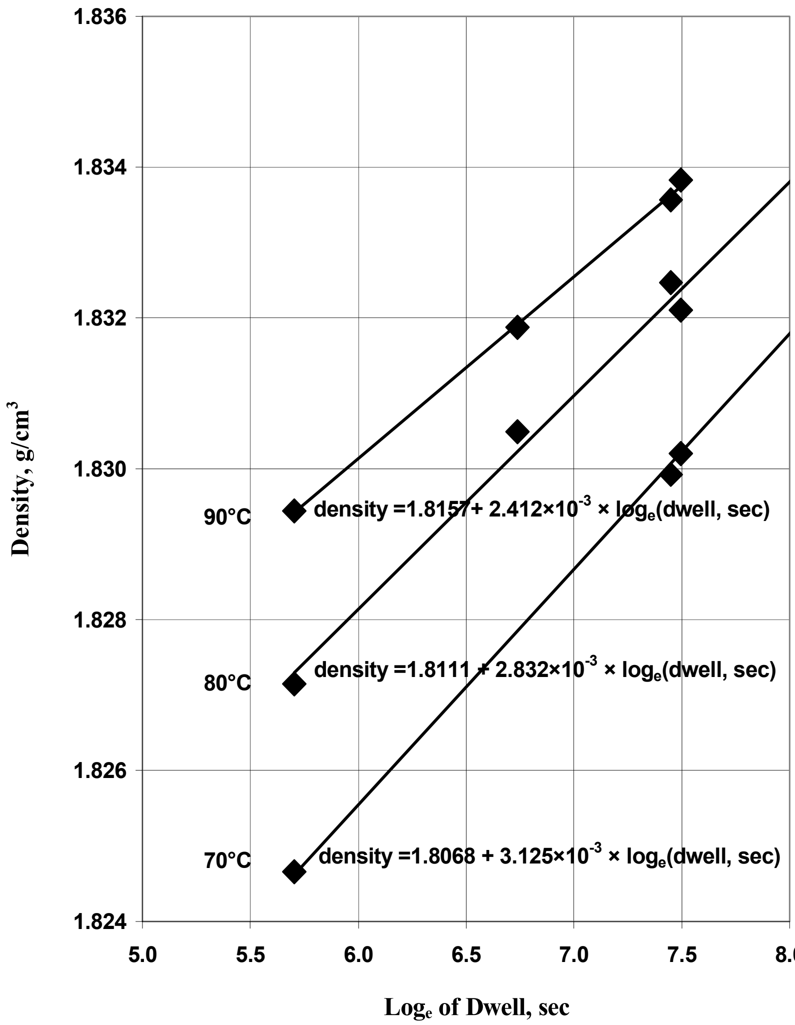

Figure 15. Compacted densities plotted as a function of the logarithm of dwells for temperatures of 70°C, 80°C, and 90°C.

In Fig. 17 are the densities of the 4-in.-diameter and 4-in.-long cylinders at various times during the compaction of PBX 9501 molding powder at 90°C, 20,000 psi for two cycles. During the first cycle dwell the PBX was held at pressures for 300 s, then the pressure was released while the explosive remained under vacuum and held at 90°C for a 300-s rest period, and finally the pressure was re-applied for an additional 300 s. The densities for the first 300-s dwell are calculated from the 90°C fit in Fig. 15. Densities of cylinders recovered during the 300-s rest period show, as with 1-5/8-in.-diameter and 2-3/16-in.-long cylinders in Fig. 11, the density continued to increase despite the absence of pressure. But the effect was much less than what was observed for the smaller cylinders. Size appears to matter in regard to the density increase during the rest period. When pressure was re-applied, the density rapidly increased and then leveled off in a logarithmic fashion.

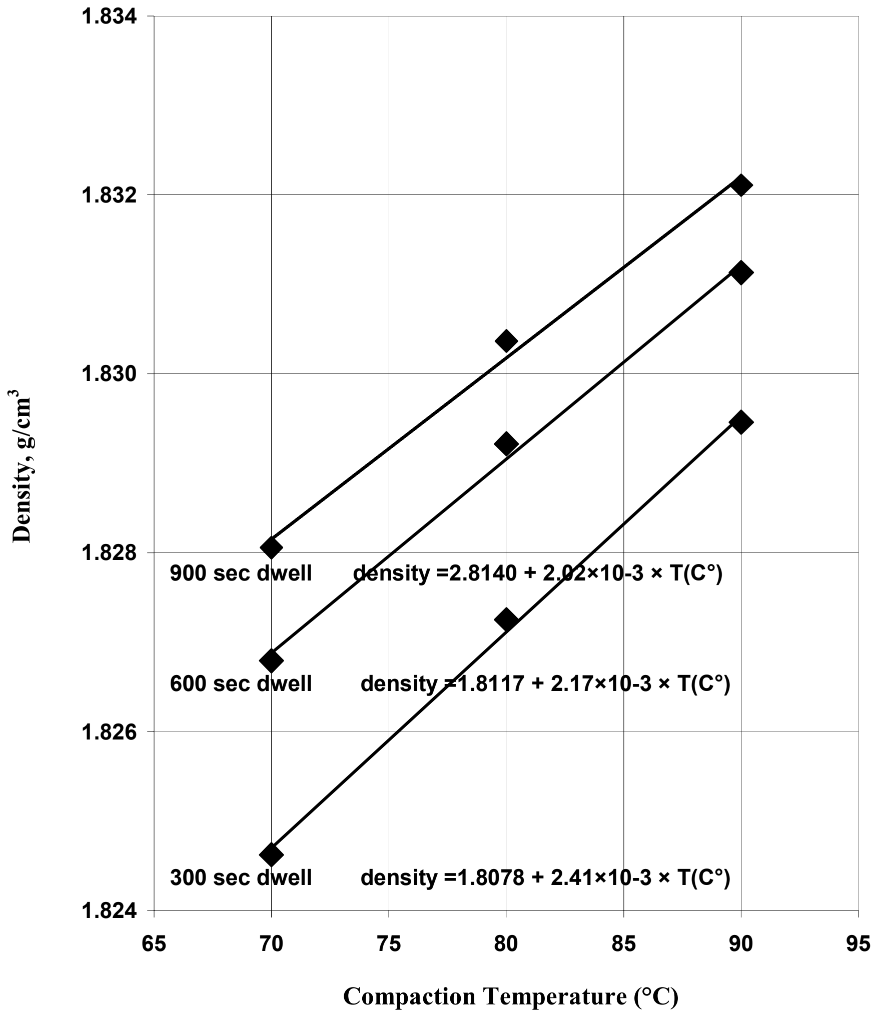

Figure 16. Points are taken from linear fits in Fig. 15 and used to establish a trend of compaction temperature and density.

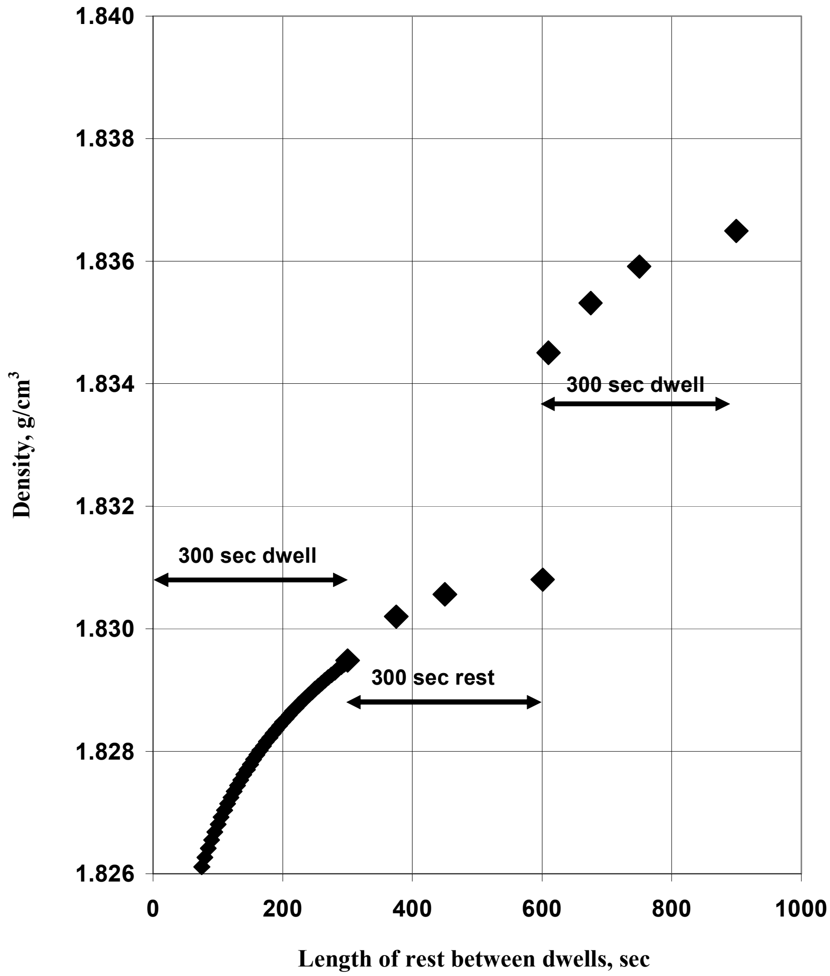

Figure 17. Densities of PBX 9501 compacted at 20,000 psi, 90°C, during a 300-s dwell, a 300-s rest at 0 psi, and a second 300-s dwell.

However, in the earlier studies using the 1-5/8-in.-diameter and 2-3/16-in.-long cylinders, we found that the density at the end of the second cycle increases rapidly with an increase in the rest period between cycles and could be linearly fit with the logarithm of the time for the rest period. We attributed this phenomenon to some sort of relaxation in the plastic-bonded explosive. We found this same phenomenon again with the 4-in.-diameter and 4-in.-long cylinders. The densities of cylinders recovered after various lengths of rest are plotted in Fig. 18. Again, they can be fit linearly with the logarithm of the rest period duration.

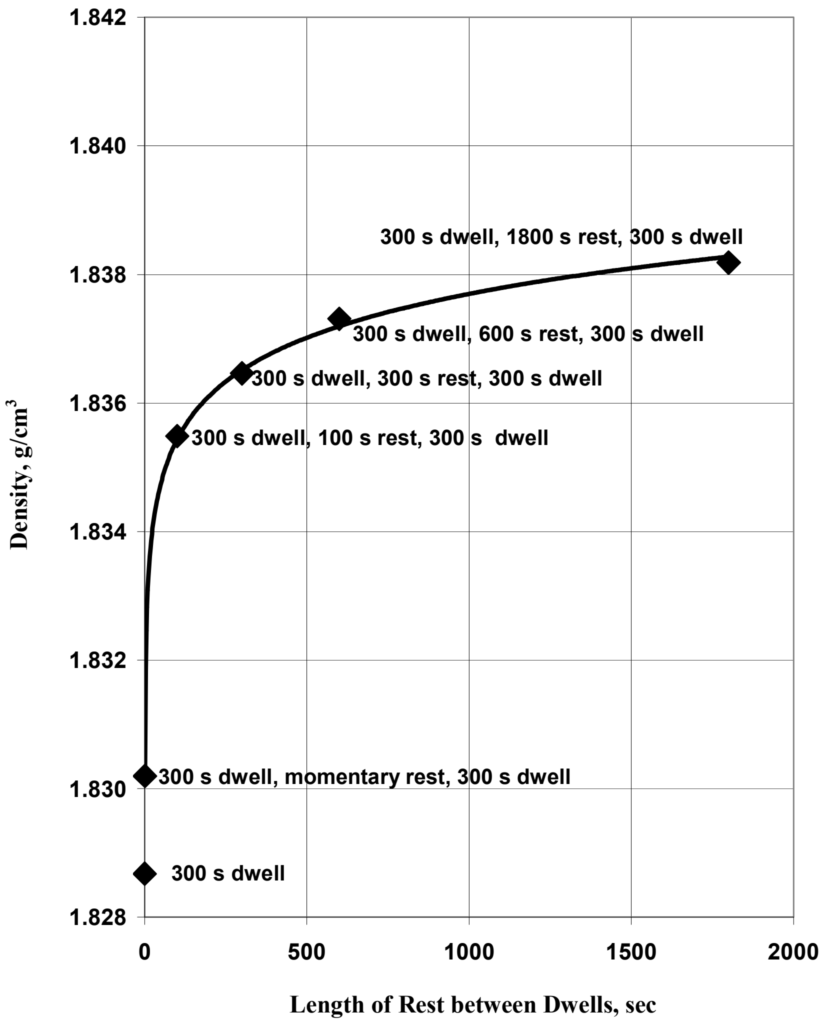

Figure 18. Densities of PBX 9501 after a 300-s dwell, a rest of varying length, and a 300-s dwell. The curve is a linear fit to the density and the logarithm of the rest length.

Compaction Studies of Large Cylinders of PBX 9502 and Comparisons to PBX 9501

In Fig. 19 are plotted the densities of compacted 4-in.-diameter and 4-in.-long cylinders of PBX 9502 at its normal powder temperature for pressing, 110°C, as a function of logarithm of the dwell in seconds, for 12,000, 16,000, and 20,000 psi. As with PBX 9501, the relationship is approximately linear. For comparison, in Fig. 20 the densities for PBX 9501 are shown for the same density range and dwell times at its normal powder temperature for pressing, 90°C. As can be seen, changes in compaction pressure cause much larger changes in density in PBX 9502 than in PBX 9501. The difference in response to compaction pressure for these two plastic-bonded explosives probably can be attributed to the difference in their porosities. PBX 9502 is three times more porous than PBX 9501, leaving more room for further compaction. The increase in density due to dwell is also greater for PBX 9502 than it is for PBX 9501, probably because of the porosity difference.

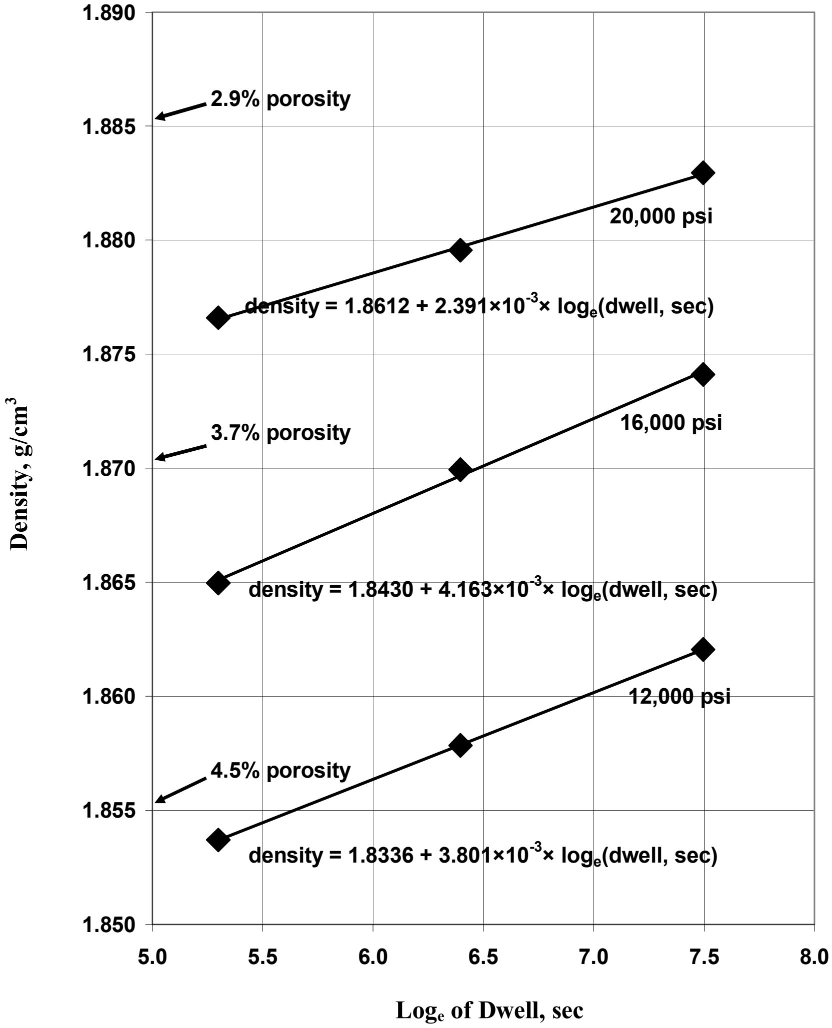

Figure 19. The compaction of PBX 9502 at 110°C, and 12,000, 16,000, and 20,000 psi. Porosity is indicated for selected densities.

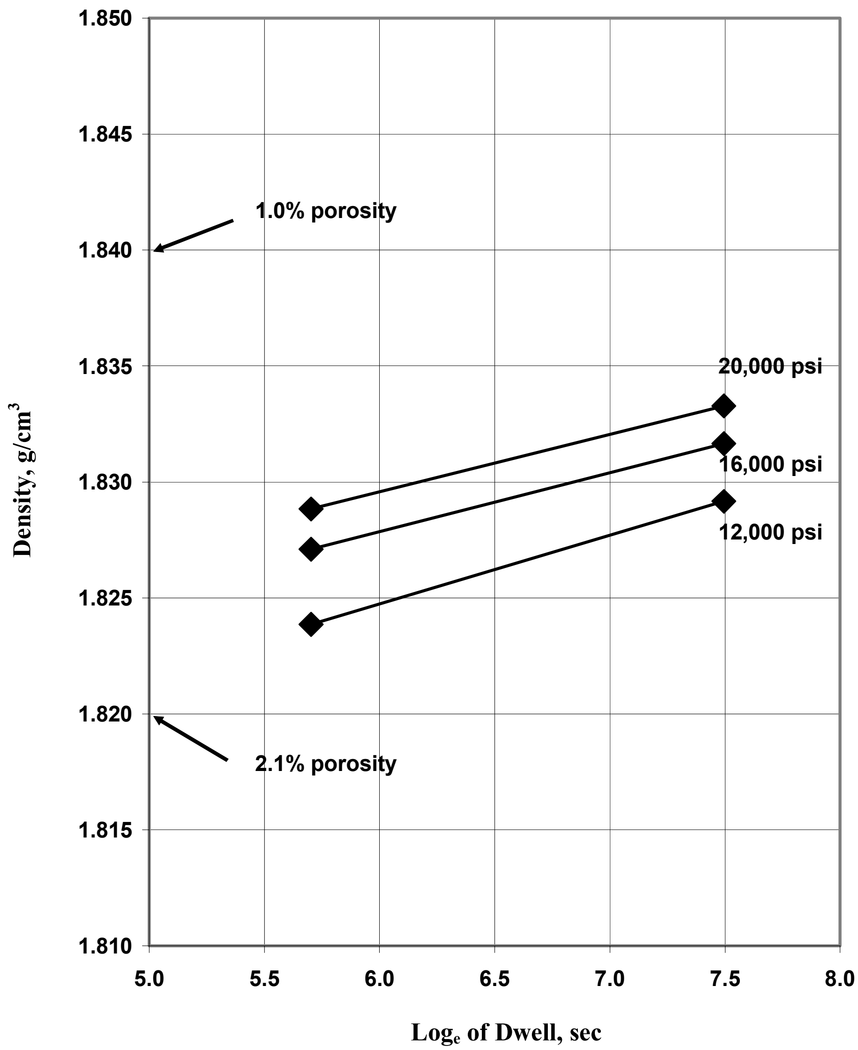

Figure 20. The compaction of PBX 9501 at 90°C, and 12,000, 16,000, and 20,000 psi. Porosity is indicated for selected densities.

The next set of data, displayed in Fig. 21, is the densities of PBX 9502 pressed at 20,000 psi, at 90°C, 100°C, and 110°C, as a function of the logarithms of the dwell times. Figure 22 shows similar data for PBX 9501 for 70°C, 80°C, and 90°C. Contrary to the effects of compaction pressure and dwell time, temperature has a greater effect on the compaction density of PBX 9501 than on the PBX 9502 density. This difference is particularly important when considering the effect of heat lost during hydrostatic pressings. The PBX 9502 density will be less affected by a decrease in temperature than will the PBX 9501 density.

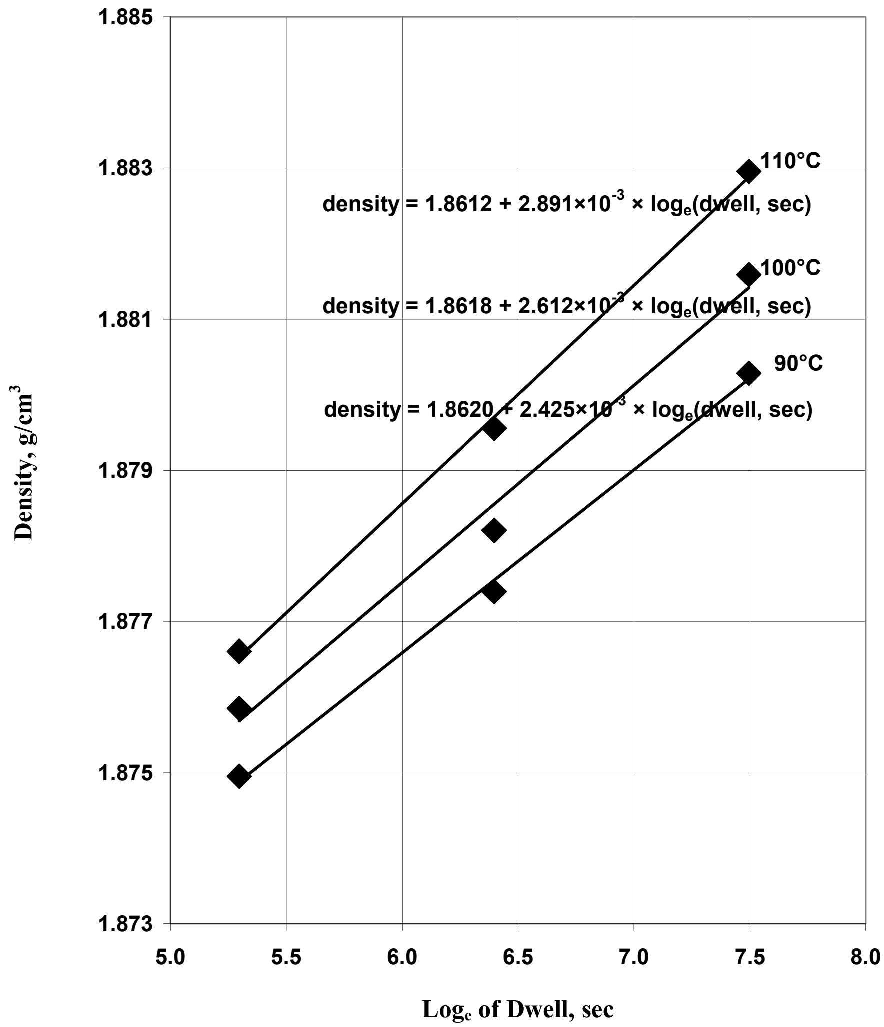

Figure 21. The compaction of PBX 9502 at 20,000 psi and 90°C, 100°C, and 110°C.

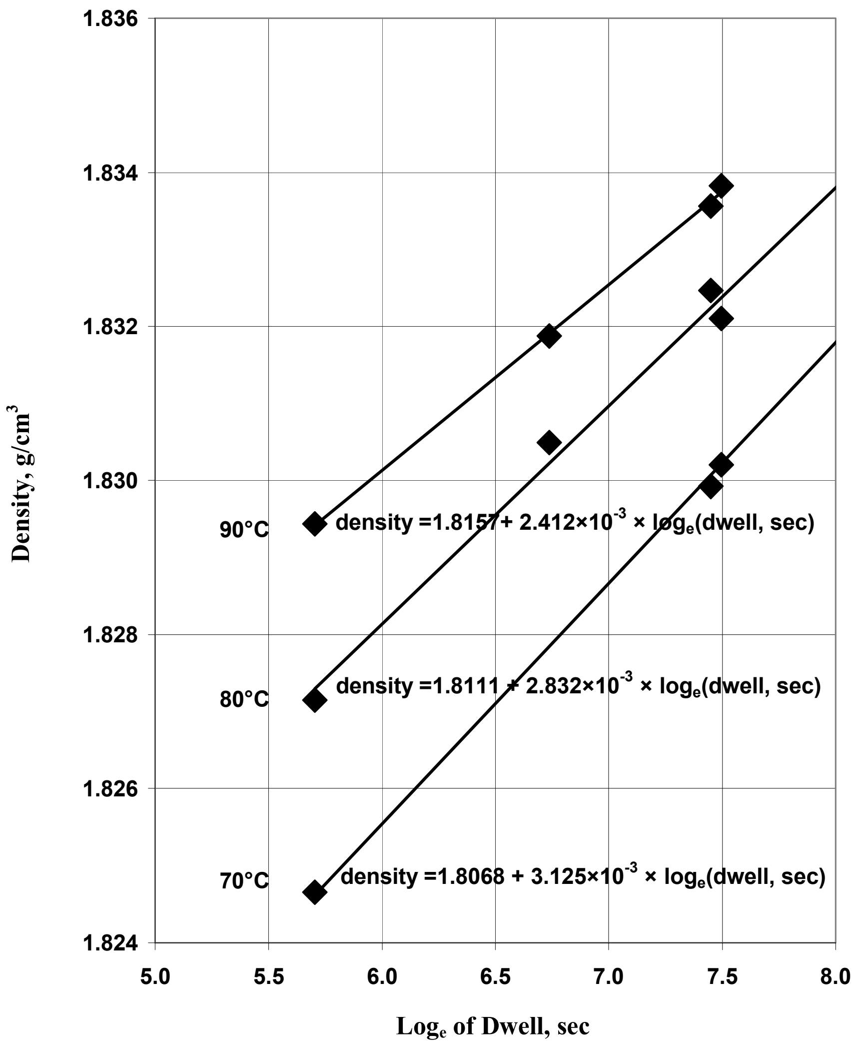

Figure 22. The compaction of PBX 9501 at 20,000 psi and 70°C, 80°C, and 90°C.

As was done with PBX 9501, densities of PBX 9502 were measured for various times during a 300-s rest period following a 300-s dwell. These densities for both PBX 9502 and PBX 9501 are shown in Figs. 23 and 24. The compacting pressure was 20,000 psi, and the temperature was 110°C for PBX 9502 and 90°C for PBX 9501. The responses of both explosives were similar, with the greater density increase for PBX 9502 attributed to its greater porosity. Both explosives showed little of the density increase during the rest period as was displayed earlier by the smaller compacted cylinders of PBX 9501.

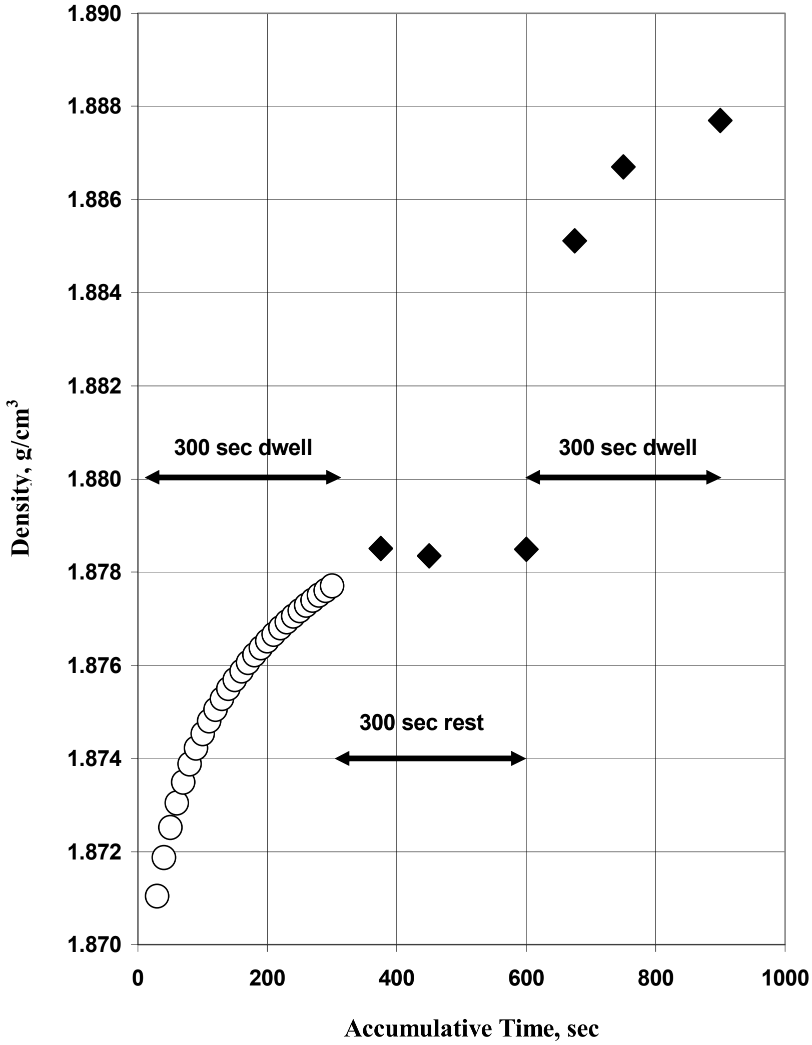

Figure 23. Densities of PBX 9502 at various times during a 300-s, 20,000-psi dwell, a 300-s, 0-psi rest, and a 300-s dwell, all at 110°C.

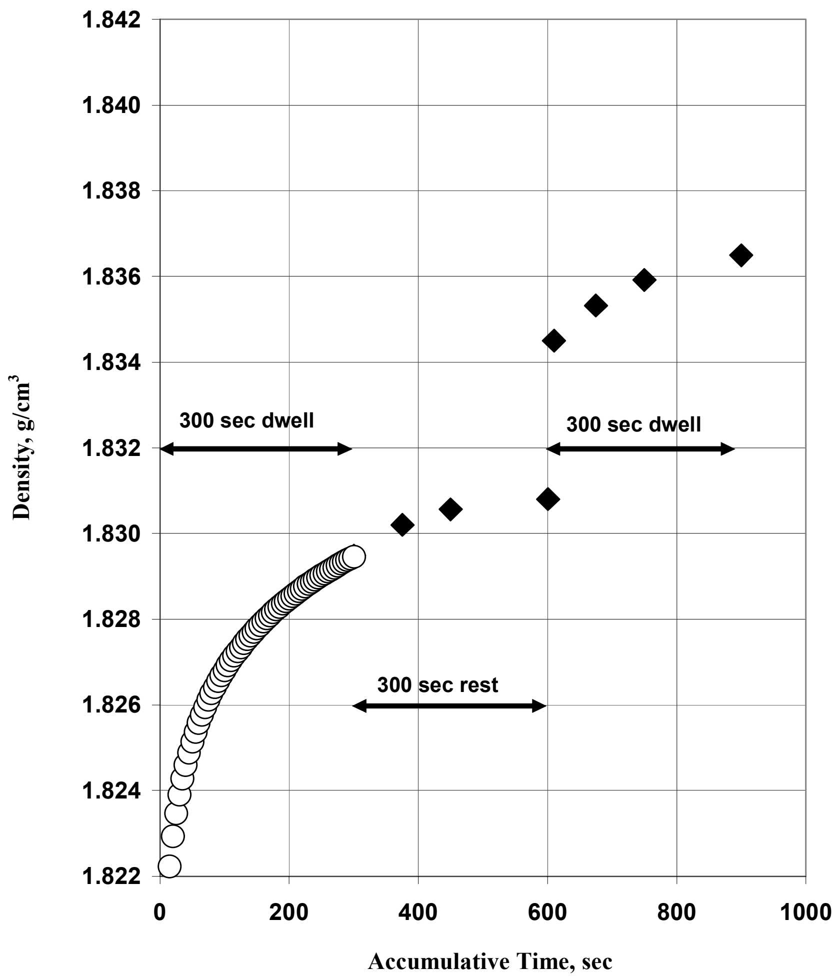

Figure 24. Densities of PBX 9501 at various times during a 300-s, 20,000-psi dwell; a 300-s, 0-psi rest; and a 300-s dwell, all at 90°C.

In the study of PBX 9501, longer rest periods resulted in greater densities at the end of a second dwell period. The increase diminished with rest period increase as the logarithm of the rest length. The most efficient gain was found to be for equal dwell and rest periods for PBX 9501. In Figs. 25 and 26 are densities for PBX 9502 and PBX 9501 for an initial dwell of 300 s, rest periods ranging from a few seconds to 800 s, followed by a second 300-s dwell. Both explosives were compacted to 20,000 psi; PBX 9502 was heated and pressed at 110°C and PBX 9501 at 90°C.

The response to cycling for PBX 9502 was different than for PBX 9501. The density at the end of the second cycle increased rapidly with increasing the rest period to 90 s and then suddenly ceased and even decreased according to these measurements. This result suggests that periods of dwell and rest for maximizing the density increase in PBX 9502 should include 5- to 10-min dwells followed by 90-s rests. Though PBX 9502 requires briefer rest periods than PBX 9501, it does require rest periods of up to 90 s for significant gains in density. This difference in the responses of the two explosives to the rest period suggests different compaction mechanisms for the two explosives.

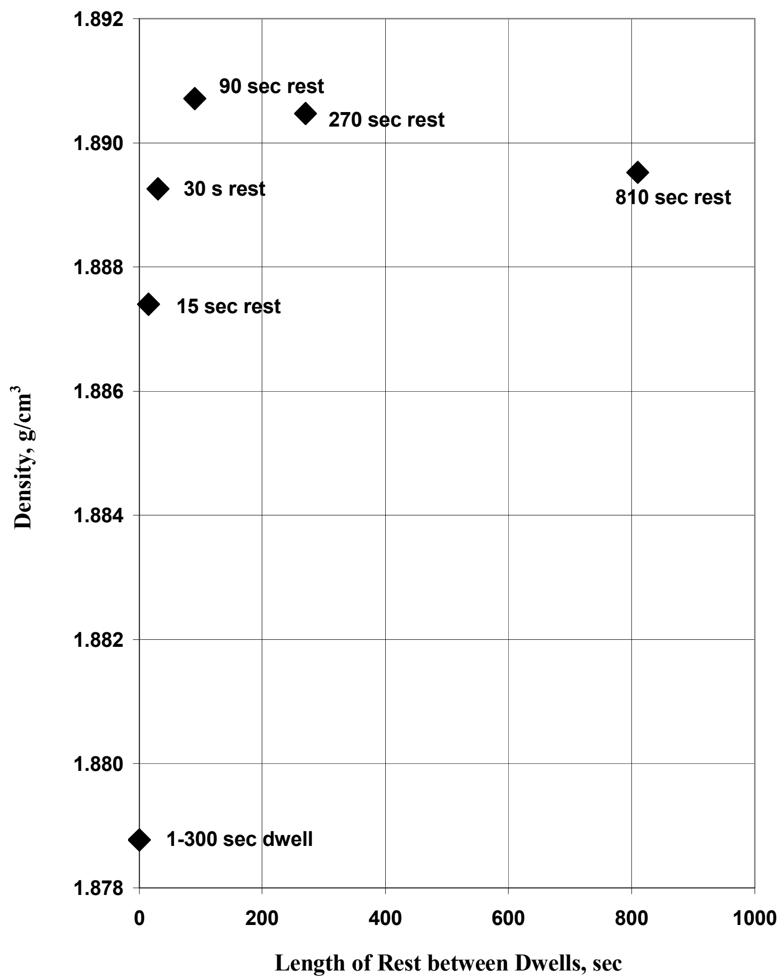

Figure 25. Densities of PBX 9502 compacted at 20,000 psi, 110°C, after a 300-s dwell, a rest of different lengths, and finally another 300-s dwell.

The results of the comparison of the compaction properties of PBX 9502 and PBX 9501 can be summarized as follows.

1. The porosity of PBX 9502 is about three times greater than that of PBX 9501 for given conditions.
2. The density increase with compaction pressure is about three times greater for PBX 9502 than for PBX 9501.
3. The density increase caused by dwell is 50% greater for PBX 9502 than for PBX 9501.
4. The compaction density of PBX 9501 is more influenced by temperature than that of PBX 9502.
5. Both explosives require a period of rest in order to gain a substantial density increase during cycling. PBX 9502 requires about 1 min whereas PBX 9501 requires 5 to 10 min for maximum efficiency.

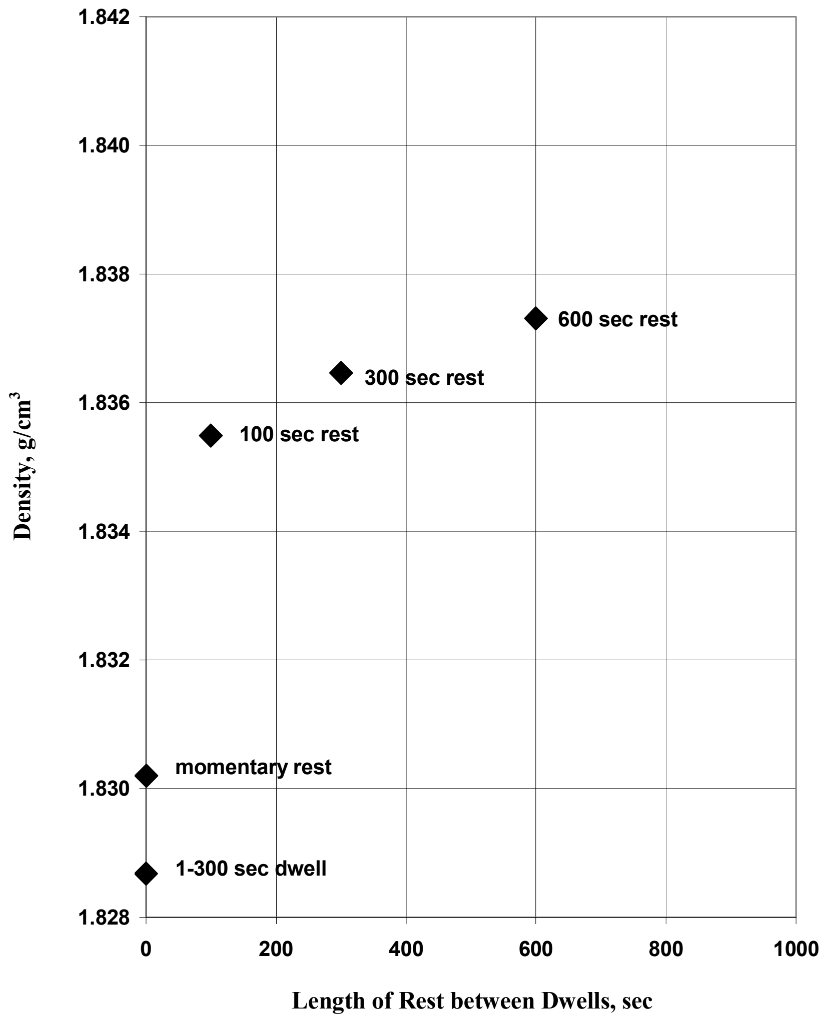

Figure 26. Densities of PBX 9501 compacted at 20,000 psi, 90°C, after a 300-s dwell, a rest of different lengths, and finally another 300-s dwell.

Some Characteristics of Hydrostatic Compaction of PBX 9501

To this point the compaction properties of plastic-bonded explosives have been based on studies done with steel-die presses. However, many explosive charges are pressed in either of the two hydrostatic presses, the 3H (#3 Hydrostatic) or the Carbox™ press. As described earlier, with the hydrostatic or dry-bag method the powder was packed hot into a thick rubber-like mold called a liner that was in turn set in a thinner rubber sack. The press lid sealed the sack opening, and the powder was evacuated through the lid. Pressure was applied with the oil in which the sack was immersed. Mandrels can be set into the powder during packing.

For characterizing the compaction of PBX 9501 in the hydrostatic presses, Lot BAE02L145-006, was chosen. A 10-in.-diameter hemispheric shape was used for the documentation. The powders were heated to 90°C before being packed into the cold liners and powders were compacted to 20,000 psi.

Two different characterizations of the presses were documented. The first was the increase in density of the charges as a function of the dwell time. In Fig. 27 are plotted the densities of the hemispherical charges from both the 3H and Carbox™ presses held at 20,000 psi for 200, 600, and 1,800 s. Their densities were found to increase as the logarithm of the dwell time, and their slopes were comparable to those for the 4-in.-diameter and 4-in.-long cylinders, the 1,800-g cylinders pressed with the steel-die.

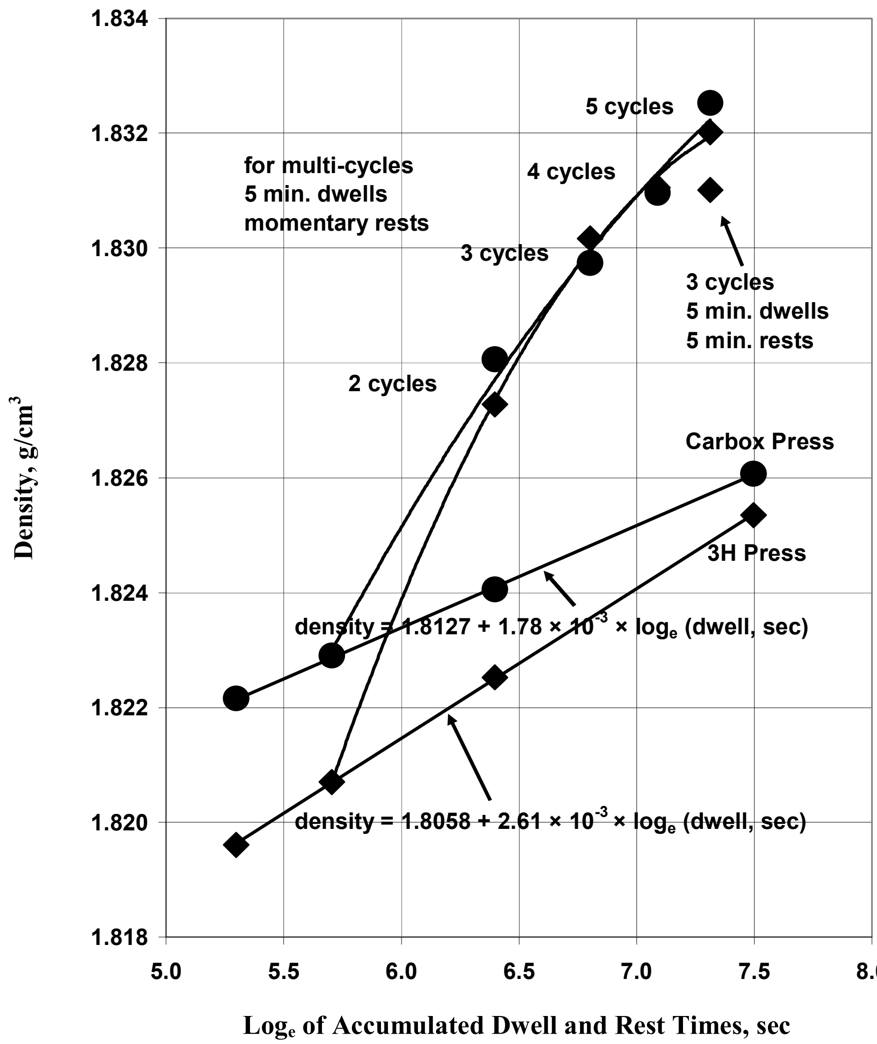

Figure 27. Densities of PBX 9501 compacted in the 3H (diamonds) and Carbox™ (circles) hydrostatic presses. The densities for a single dwell of various lengths increase as the logarithm of the dwell. The difference in densities from the two presses diminishes after three cycles. Dwells were 5 min long, the rests were momentary.

The second characteristic measured for the presses was the effect of cycling on density. Figure 27 also displays these data; the dwell times used for the multi-cycles are the accumulated dwell times. Historically, only momentary rests were used, so this was what was documented. The pressure was released to 0 psi and immediately reapplied, taking perhaps 15 s. The dwells used during the cycling operation were 300 s long.

A major difference between steel-die pressed charges and hydrostatically pressed charges was the greater density of the former. The density of a 1,800-g cylinder, pressed in a steel-die to 20,000 psi, 90°C for one cycle with a dwell of 300 s is 1.8285 g/cm^3. The density of a 10,500-g hemisphere pressed hydrostatically to 20,000 psi starting at 90°C for one cycle with a dwell of 300 s is 1.8229 g/cm^3.

However, during cycling, the density of the cylinders increased from 1.8285 to 1.8302 (Δ0.0017) g/cm^3, using momentary rest (Fig. 15). Here the density increased from 1.8229 to 1.8281 (Δ0.0052) g/cm^3, for the same rest at the same dwell and temperature (Fig. 27). This difference could be caused by the pressing technique, or, more likely, by the lower starting density in the hydrostatic pressings. Though the Carbox™ press initially compacts explosives to higher densities than the 3H press (differences in pressures, perhaps undetected by faulty pressure gauges), cycling causes the differences to disappear.

Prolonged Rests during Hydrostatic Cycling

Studies with steel-die presses showed that prolonged rests led to substantial gains in density during cycling. This phenomenon was studied for hydrostatic pressing. To determine the effect, a hemisphere was compacted at 20,000 psi with powder heated to 90°C in the 3H press for three cycles with 5-min dwells, and with 5-min rests instead of momentary rests. The result is shown in Fig. 27, 1.8310 g/cm^3. This density is the same as that for four cycles with momentary rests, but the total compaction time is the same as five cycles with momentary rests that achieves a density of 1.832 g/cm^3.

Density Distribution in Hydrostatically Pressed PBX 9501

A large continuous charge having a uniform radius from the pressure medium was pressed. It was a 10-in.-diameter hemisphere of PBX 9501. The powder was pre-heated to 90°C and compacted at 20,000 psi for five cycles with 5-min dwells and momentary rests. The resulting density distribution of the cross-section is shown in Fig. 28. The bulk density is about 1.830 g/cm^3, and the density variation is remarkably low, less than 0.002 g/cm^3. Almost all the low- and high-density regions are near the hemisphere’s equator; the remainder of the volume is uniform.

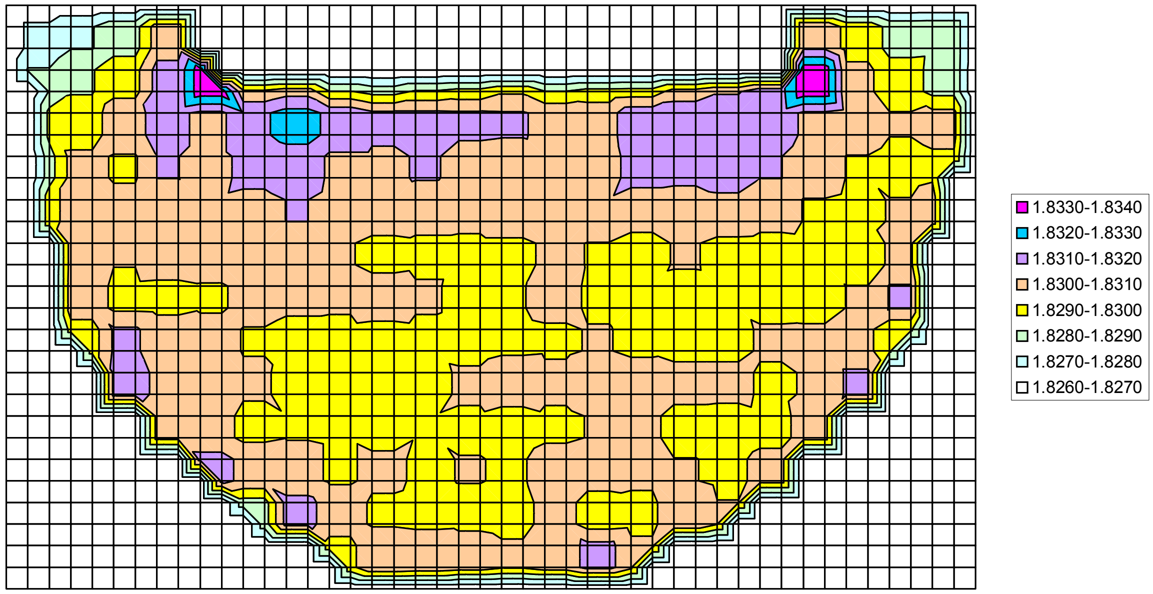

Figure 28. Ten-in.-diameter hemisphere of PBX 9501. The powder was preheated to 90°C and compacted at 20,000 psi for 5 cycles with 5-min dwells and momentary rests.

The Reciprocal of Porosity Fraction Increases Logarithmically with the Number of Cycles

High densities in plastic-bonded explosives are efficiently achieved by repeatedly applying and releasing and reapplying pressure. Each application of pressure is called a cycle, the time the explosive is held at pressure is called the dwell, and the periods of time at zero pressure between dwells are called rests.

In the **Porosity and Density** section, an attempt was made to describe the density increase as a function of dwell time in terms of the porosity fraction, the ratio of the difference between the theoretical density and the actual density to the theoretical density. The porosity faction is the property of the compacted PBX we are attempting to reduce to zero; it has a definite limit and is increasingly difficult to achieve as it is reduced. The value of the reciprocal of the porosity fraction goes to infinity with the dwell time or its logarithm. Attempts at finding a constrained fit, a linear equation, using dwell as a variable did not succeed (see Figs. 5 and 6).
The logarithmic increase in density with dwell proved to be a better description of the data (see Figs. 3 and 4) despite the fact that it fails at infinite dwell time where the density is also infinite.

Here the problem of describing the density increase caused by cycling is addressed in terms of the porosity fraction. The data available were those collected from the mold type (MT) 157s and from MT 800s. These data are from compaction operations that are very different. The MT 157s are 1-5/8-in. × 2-in. cylinders compacted in the 50-ton steel-die press. They were held at 90°C before and during compaction. Each cycle consisted of 20,000 psi for 5 min and then no pressure for 5 min. The MT 800s are hollow hemispheres, much larger than the MT 157s, compacted in the 3-H and Carbox hydrostatic presses encased in thick rubber bladders.

The molding powder was heated to 90°C before compaction, but was not heated during compaction. Each cycle consisted of 20,000 psi for 5 min with momentary rests (a few seconds) before returning to pressure.

The figures on the next page on the left side (Figs. 29, 31, and 33) display the densities for the cycles applied. On the right side (Figs. 30, 32, and 34) are plotted the porosity fraction reciprocals and the logarithms of those cycles. For the porosity fractions, the theoretical density (TD) is 1.859 g/cm^3 for PBX 9501. The linear fits for the data are shown as lines, and the fits are listed at the bottom of the figures. In all cases, the data are tightly distributed along the fits. Therefore, the reciprocal of the porosity fraction seems to increase logarithmically with the number of cycles. This relationship would also hold for accumulative time, such as that of the dwells or of the dwells and rests. The conditions of each of the cycles of a compaction are assumed to be the same as all the other cycles. The intercept and slope of the linear fits depend on cycle compaction conditions, such as pressure, temperature, and lengths of the dwells and rests. The curves on the left-hand figures were calculated from the linear fits in the right-hand figures.

This linear relationship between the reciprocal of the porosity fraction and the logarithm of the number of cycles enables us to estimate accurately the density at the end of a number of cycles based on the densities measured for a PBX compacted with just one and two cycles. We have already thoroughly studied the conditions needed to achieve a density at the end of the first cycle, the intercept of this linear fit. From the density after the second cycle, the slope is determined; its value depends mainly on the length of the rest period. In the section, **Efficient Distribution of Dwell and Rest Periods**, it was shown that dwell and rest periods of equal length during cycling is the most efficient means to achieve high density (see Table 5).

What physically occurs during cycling is not understood. We have measured increases in the density of the PBX during the rest period when the explosive is not under pressure. We did not expect this result. The PBX apparently compacts while it relaxes after being under stress. In addition, the compacted PBX under stress appears to develop high internal surface friction that gradually reduces during the rest period, permitting the next application of pressure to compact the explosive a greater amount than if it had been retained at pressure.

Density Dependence on Cycling Period and Cycling Pressure

In the previous section, the linear relation between the reciprocal of the porosity fraction,

$$\frac{(Theoretical.Density)}{(Theoretical.Density - Density)} \equiv \frac{TD}{TD - D} \quad , \qquad (4)$$

and the logarithm of the number of compaction cycles is demonstrated,

$$\frac{TD}{TD - D} = a + b \times \log(\#cycles) \quad . \qquad (5)$$

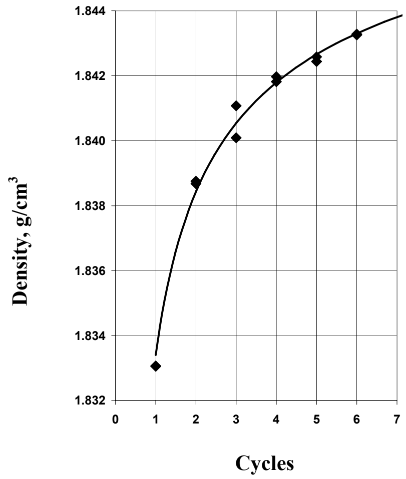

Figure 29. MT 157, density and cycles (50-ton steel-die press).

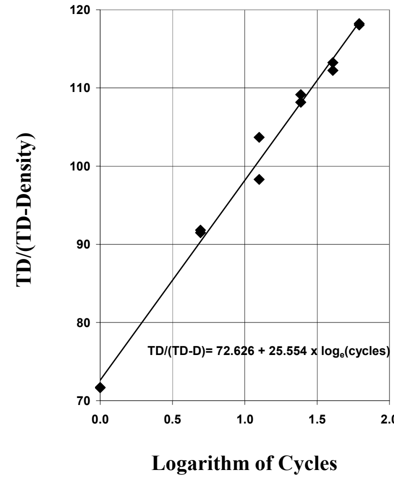

Figure 30. MT 157, TD/(TD-density) and logarithm of cycles (50-ton steel-die press).

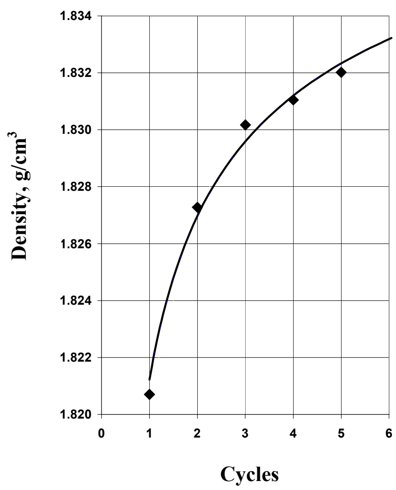

Figure 31. MT 800, density and cycles (#3 Hyrostatic Press).

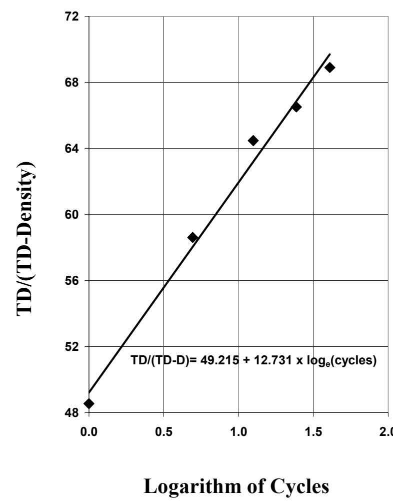

Figure 32. MT 800, TD/(TD-density) and logarithm of cycles (#3 Hyrostatic Press).

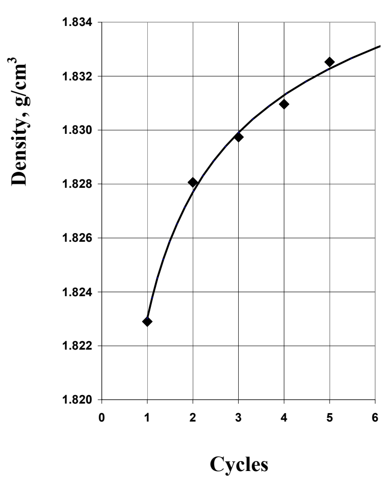

Figure 33. MT 800, density and cycles (Carbox Press).

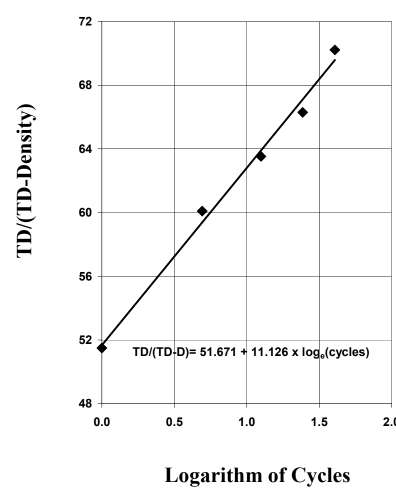

Figure 34. MT 800, TD/(TD-density) and logarithm of cycles (Carbox Press).

Using this phenomenon, we can determine the linear constants by measuring the densities of explosives that have undergone different numbers of compaction cycles. Having determined the linear constants, then the number of cycles required to achieve a given density can be determined.

The densities of hemispherical shells compacted in the #3 Hydrostatic (3H) Press using mandrels for the mold type 800 were measured for two different numbers of cycles. The material lot of PBX 9501 used was BAE03L145-007. The powder was heated to 90°C and compacted at 20,000 psi. Densities were measured for 1 and 5 cycles. From the determined linear constants, we found that for dwell and rest periods of 2 min, 7 cycles are required to achieve a density of 1.833 g/cm^3. For dwell and rest periods of 1 min, 12 cycles are required. The time required to compact PBX 9501 was not substantially reduced by changing the period of the cycle.

The same material was compacted using 2-min rest and dwell periods, but at 10,000 psi instead of 20,000 psi. At this pressure, 25 cycles were required to achieve 1.833 g/cm^3. Reducing the compaction pressure by one-half required 3 to 4 times longer achieve a given density. This finding is similar to the increase in time required to achieve a given density using one cycle at two different pressures, Table 2.

Compaction-Temperature and Dwell and the Compressive and Tensile Strengths of PBX 9501

Density is the principal criterion for judging the quality of a compacted plastic-bonded explosive (PBX). PBX molding powder is heated to a temperature that softens the plastic binder and is compacted under vacuum at high pressure for a period of time called the dwell. Usually the pressure is released for another period called the rest and then reapplied. Each application of pressure is called a cycle. Pressure, temperature, dwell, rest, and cycles are the variables involved in compaction. These variables have been studied to understand their contributions to the explosive's density. Different combinations of the variables can be used to achieve a given density.

The mechanical properties of compacted explosives are also important. Explosives must have sufficient strength to withstand severe shock-and-vibration conditions and strong positive and negative forces caused by accelerations. Though various combinations of the compaction parameters can be used to achieve a given density, the resulting mechanical properties for these combinations may not be the same. This study was the first of a series of experiments to document the effects of the compaction parameters on the material properties of PBX 9501. Temperature during compaction was the first parameter studied because higher temperatures soften the plastic binder making it more weldable between adjoining powder granules during compaction, resulting in a more cohesive plastic-bonded solid explosive.

Changing any one of the compaction parameters to measure its influence on compressive or tensile strength also alters the density of the compacted explosive. The fundamental compaction response of plastic-bonded explosives is a logarithmic increase in bulk density with the compaction dwell length. In Fig. 35 are the plotted the densities of PBX 9501 with the logarithm of dwell compacted at 20,000 psi (138 MPa) with 5-min and 30-min dwells and with the powder preheated and heated during compaction at 3 different temperatures, at 70°C, 80°C, and 90°C. The PBX 9501, from lot BAE02L145-006, was compacted in a 500-ton steel-die press into cylinders 4-1/4-in. high and 4-1/4-in. diameter. This figure shows the interdependence of density, dwell time, and temperature. A study of the effects of the temperature of compaction on compressive and tensile strengths also requires the effects of density and dwell on these strengths to be considered.

The dwell times can be well determined, and because the molding powders are preheated to the temperatures at which they are compacted, the compaction temperatures are also accurately known. The density of the compacted explosive can also be determined accurately and precisely to within 0.0001 g/cm^3 by permitting the explosive to reach thermal equilibrium with the immersion water and using silicon crystals as standards. However, the material strength of the explosive during compaction prevents the piston pressure from being achieved throughout the explosive. Figure 36 graphically illustrates this result in the density distribution in a steel-die compacted cylinder of PBX 9501.

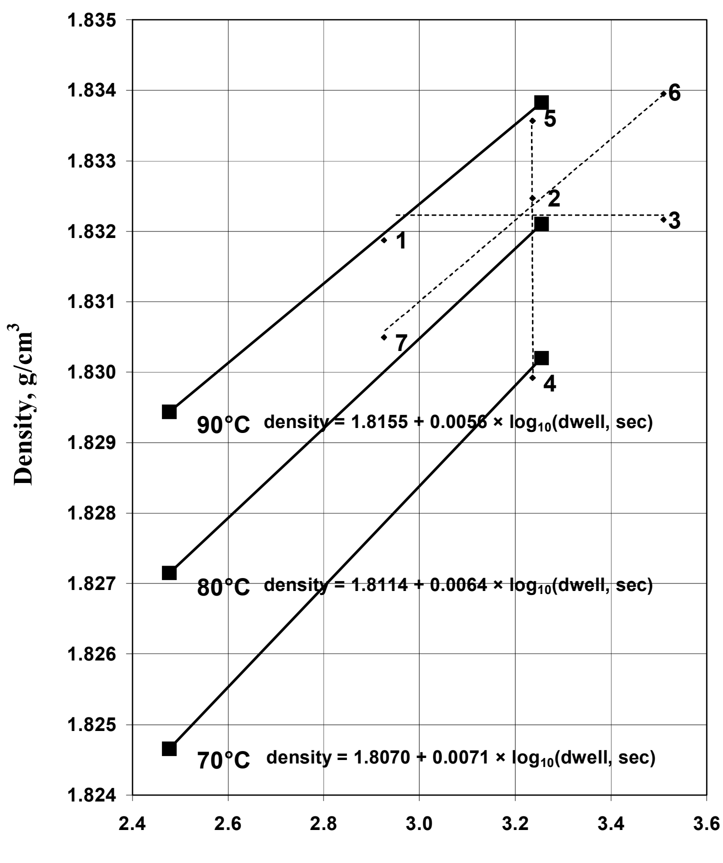

Figure 35. Density of PBX 9501 increases linearly with the logarithm of the dwell for 70°C, 80°C, and 90°C. The material compacted for material strength measurements are the diamonds and are numbered.

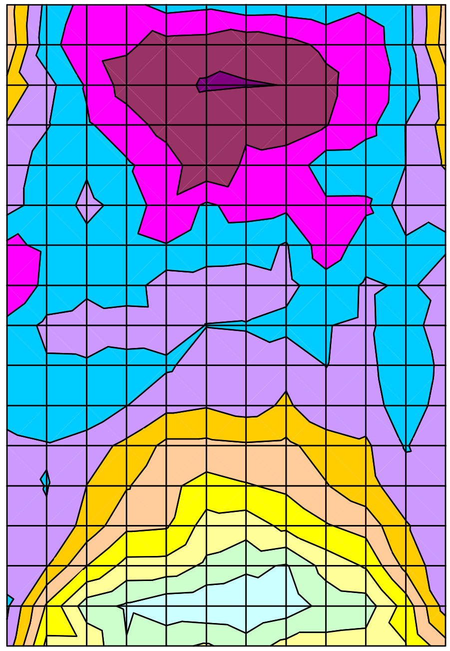

Figure 36. Density distribution in a steel-die-compacted cylinder. It was determined from the elements of a diameter slice of the cylinder.

The bulk density of this cylinder is 1.8325 g/cc, but along the axis it ranges from 1.835 near the top to 1.830 near the base. The compaction pressure is applied against the base. All cylindrical steel-die-compacted explosives have the same density distribution pattern. From each cylinder, 3 tensile specimens were cored parallel to the axis, and 4 compression specimens were machined from along the axis (Fig. 37). Because four compression specimens were cut from a core along the axis, they each have a unique density. The tensile specimens from the same cylinder have a density distribution within them, but they should be all similar. The region subjected to failure within the tensile specimens is a region of rather uniform density.

Because density is dependent on dwell and temperature, a plan for isolating the effects of temperature, density, and dwell was made based on the linear fits in Fig. 35. Conditions for the first group of samples were selected to have their densities the same, 1.832 g/cm^3, by adjusting the dwells so that they could be compacted at different temperatures—70°C, 80°C, and 90°C. The dwell times determined from the linear fits were 843 s at 90°C, 1,720 s, at 80°C, and 3,235 s at 70°C. The measured bulk densities at these conditions are labeled 1, 2, and 3 in Fig. 35. Next, conditions were chosen so that the dwell times were the same for samples compacted at 70°C, 80°C, and 90°C. The densities at these conditions are labeled 4, 2, and 5 in Fig. 35. Finally, conditions were selected for samples to be compacted at the same temperature, to have the same dwell as samples 1 and 3, and to have the same densities as samples 5 and 4. These are samples 6, 2, and 7.

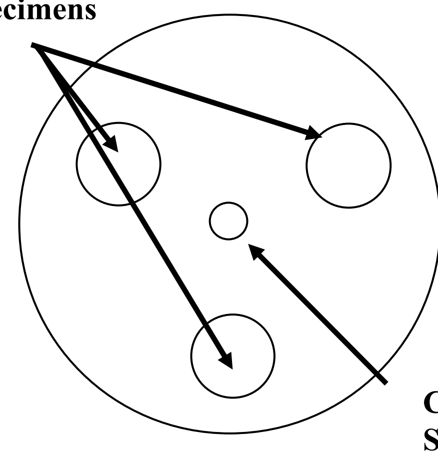

Figure 37. Location and size of cores cut from 4-1/4-in.-diameter cylinders. One specimen was cut from each tensile core and four specimens from the compression core.

Quasi-static compression and tension specimens were machined from the cylinders at locations shown in Fig. 37. The density of each specimen was determined to a precision and accuracy of 0.0001 g/cm^3. Compression specimens were 0.953 cm in diameter and 1.91 cm in length. Graphitic molybdenum-sulfide lubricant was rubbed on the ends of the specimens to minimize frictional confinement. Tension specimens were 7.62 cm in length with cylindrically tapered ends and a gauge length of 1.27 cm diameter, 3.81 cm long. Quasi-static, uniaxial tension and compression tests were conducted on an Instron™ 5567 using two oppositely mounted, knife-edge extensometers. Tests were conducted in strain rate control using the averaged strain output of the two extensometers. All tests were conducted at 0.0001 s^{-1} (a velocity of the length of the specimen per 10,000 s) and 23°C.

The data are the simultaneous measurements of tensile or compressive stress and the resulting strain. The resulting stress and strain data were characterized by three parameters: the maximum stress, σ_m, defined as the highest stress achieved before failure of the material; the strain at maximum stress, ε_m, the strain value at σ_m; the elastic modulus, E_{40}, the slope of the linear elastic portion of the stress-strain curve at a stress value of 40% of σ_m. Because of the initial, uneven seating of the specimen under load, the low-strain region of the resulting stress-strain curve can sometimes be affected by an experimental toe artifact. A toe correction is performed by calculating the strain intercept value at zero stress for the E_{40} tangent line, then shifting the strain data by this value so that the tangent line passes through the origin. The toe-corrected value of ε_m is then determined.

The ultimate tensile and compressive stresses are the principal material strength parameters of interest. These are indicative of the forces the explosive can be subjected to without being damaged. In Figs. 38 and 39 are the values of these stresses plotted with the dwells and compaction temperatures of the test specimens. Those compacted at 90°C are plotted with the symbol "■," at 80°C with "▲," and at 70°C with "♦." Linear fits were made for each temperature group to see the trend of the data because the data are few and scattered. In addition, loci of conditions that result in the same densities are plotted as dashed lines. These were calculated from the linear fits in Fig. 35 to calculate the logarithms of the dwells for densities 1.831 g/cm^3, 1.832 g/cm^3, and 1.833 g/cm^3. Then, from the linear fits in Figs. 38 and 39 and the calculated logarithms, the ultimate stress coordinates for the iso-density loci are calculated.

Several conclusions can be made from the plot in Fig. 38. As density in Fig. 35, the ultimate tensile stress increases with dwell. Also, as density, compaction temperature increases ultimate tensile stress. The iso-density loci reveal yet another important observation. The changes on the ultimate tensile stress level caused by lower compaction temperature are greater, but opposite, from those caused by increasing dwell in order to maintain a constant density. For a given density, PBX 9501 compacted at higher temperatures has greater ultimate tensile stresses. The effect that compaction temperature has on the ultimate tensile stress, though real, is not large. Increasing the compaction temperature from 80°C to 90°C increases the ultimate tensile stress only 7% for a given density. Because increased dwell and compaction temperature cause greater density, ultimate tensile stress can also be described as increasing with density and, in addition, with the compaction temperature.

The ultimate compressive stresses plotted in Fig. 39 show that, like the ultimate tensile stresses, they increase with both dwell and compaction temperature. However, the loci of compaction conditions resulting in the same densities show that ultimate compressive stress levels are insensitive to the compaction temperature, unlike the ultimate tensile stress levels. As Fig. 39 shows, the changes on the ultimate compressive stress level caused by decreasing temperature are about equal, but opposite, to those caused by increasing dwell in order to maintain a constant density. That means the ultimate compressive stress is mainly dependent on the density. A recent analysis of the ultimate compressive stress data from "Compression Properties of PBX 9501 at Densities from 1.60 to 1.93 g/cc" (Tom Meyer, Richard Browning, Robert Spence, Feb 2000) shows that the ultimate compressive stress magnitudes increase linearly with the logarithm of ratio of the explosive's theoretical density to its difference with the actual density.

The results of the data plotted in Figs. 38 and 39, when they are compared with the density plot in Fig. 35, show that this study was unduly complex. The strategy was to hold a specific variable constant while measuring the effects of the others. A better strategy would have been to simply use the cylinders compacted to determine the density projections for the compaction temperatures, perhaps adding another cylinder in the center of each projection to provide more and a greater array of samples.

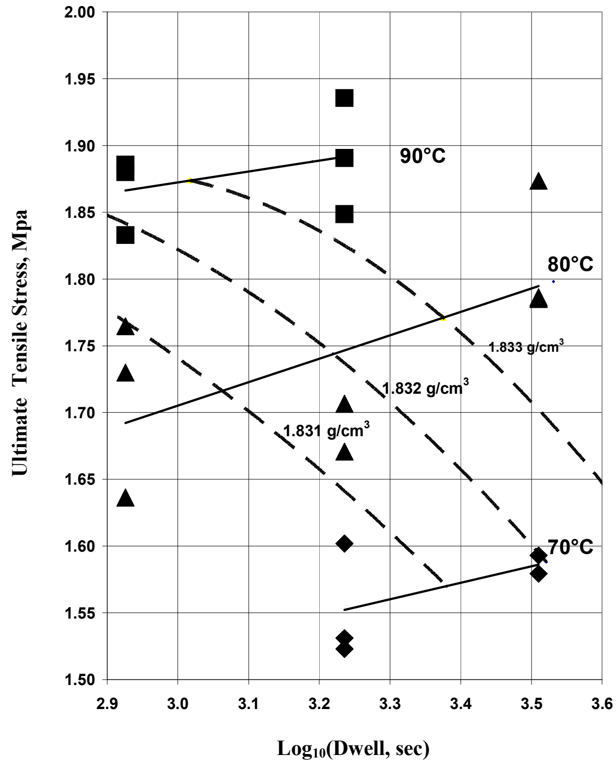

Figure 38. Ultimate tensile stresses of specimens are plotted with the logarithms of their dwells at 20,000 psi. Linear fits are for data from specimens compacted at 70°C (♦), 80°C (▲), and 90°C (■). The dashed line indicates conditions producing the same densities.

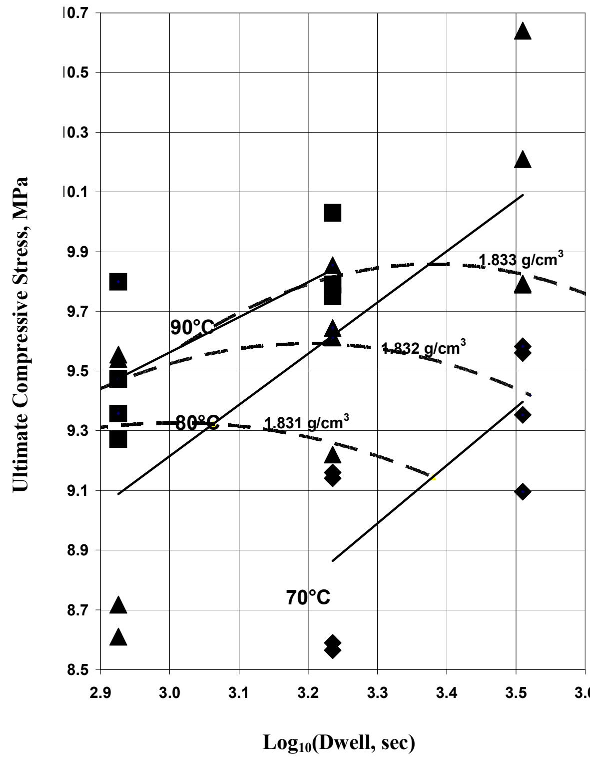

Figure 39. Ultimate compressive stresses of specimens are plotted with the logarithms of their dwells at 20,000 psi. Linear fits are for data from specimens compacted at 70°C (♦), 80°C (▲), and 90°C (■). The dashed line indicates conditions producing the same densities.

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