THICK WALLED DDT TUBE EXPERIMENTS[1]

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ABSTRACT

In this paper we present initial results from a new series of deflagration-to-detonation transition (DDT) experiments in which we study the piston-initiated DDT of heavily confined granular cyclotetramethylenetetranitramine (HMX). These experiments were designed to be useful in model development and evaluation. Initial experiments have examined the effect of density, piston speed, and ignition delay on the DDT event. Often, in previous work, little material characterization is reported which makes modeling and interpretation of the experiments more difficult. In this work we measure the particle size distribution of the original granular explosiove, as well as the size distribution of pressed (higher density) samples. Scanning electron microscope (SEM) pictures are presented and are useful in interpreting the size distribution measurements of the HMX and in more fully characterizing their initial condition. We find that the particle size distribution changes significantly with pressing. That is, particles are highly fractured and damaged at higher pressed densities. Also, we have found that sample preparation can significantly affect size distribution measurements. For the base case (65% TMD) two DDT experiments were performed yielding nearly identical results, indicating that reproducibility is good. Run-to-detonation, as indicated by color change and deformation of the inner wall, for the three densities (65%, 75%, and 85% TMD) considered shows a slight decrease, going from 65% to 75% TMD, but a significant increase in 85% TMD. This result is qualitatively compared with similar results reported previously for thermally ignited DDT tubes. Increasing the piston speed decreased the distance to detonation, as expected, and the effect of the ignition delay (tighter packing of the titanium boron gasless igniter) extended the distance to detonation somewhat. Future experiments are described.

INTRODUCTION

It is widely known that damaged explosives can be more sensitive to initiation by low-speed impacts than undamaged materials. Granular explosives have often been used as a simulant of pre-damaged explosives. It is far easier to characterize the materials and identify the DDT process in granular materials than in actual damaged explosives. DDT tubes, initiated thermally or by low-speed impact of a piston, have been used for years to study the DDT process in granular explosives (Griffiths and Groocock 1960; Korotkov, Sulimov et al. 1969; Bernecker, Sandusky et al. 1981; Samirant 1981; Bernecker, Sandusky et al. 1985; Samirant 1985; Bernecker 1986; Bernecker 1989; Samirant 1989; Luebcke, Dickson et al. 1995; Luebcke, Dickson et al. 1996). Much has been learned concerning the DDT process in granular materials from these experiments, however significant gaps remain between the desired data base and available data for model development and evaluation.

The run-to-detonation can be a strong function of density (or porosity), and is therefore of significant interest. The effect of density on run-to-detonation for granular HMX was examined in the early experimental study of Griffiths and Groocock (1960). For several reasons, however, these data are of only qualitative value. First, a hot-gas-producing igniter was used, which significantly complicates the interpretation of the experimental results and subsequent modeling. Further, data scatter is large, indicating poor repeatability. Even when less intrusive hot-wire ignition is used, the ignition front may not be planar, causing significant quantitative differences from run-to-run (Samirant 1981). Further, the particle size distribution was not well-characterized by Griffiths and Groocock (1960) and data were reported as a function of measured permeability instead of density. No conversion was provided to obtain density from the measured permeability, however the data do appear to indicate a minimum distance at a certain permeability (or density) with longer runs-to-detonation at lower and higher densities ("U-shaped" plot). Other data also have been reported for run-to-detonation as a function of density (e.g., Korotkov, Sulimov et al. 1969; Luebcke, Dickson et al. 1995; Luebcke, Dickson et al. 1996), but thermal ignition was used and granular HMX was not considered. Interestingly, some recently reported data do not show a U-shaped density dependence in granular CP (Luebcke, Dickson et al. 1996).

Because of the complex nature of the ignition, and early conductive and convective burning in thermally ignited DDT tubes, piston-driven experiments have been used here at Los Alamos National Laboratory (LANL) (McAfee, Asay et al. 1991) and elsewhere (Sandusky 1983). In the LANL experiments a combustion-driven piston ignites particles in the granular bed by compaction, which starts all the processes in a single plane at one end of the tube. These experiments were heavily diagnosed using a variety of pins, transducers, and x-radiography and provide a detailed picture of piston-initiated DDT for a narrow set of conditions (e.g., only a single density was considered). Although these results give a detailed picture of the DDT event for the conditions considered, little information is obtained concerning the effect of several parameters that are known to be important. This

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makes the development and evaluation of improved models difficult.

As part of the explosives safety program at LANL our group has worked to develop models to describe the DDT of granular HMX explosives. A main goal of this effort is to develop truly predictive models. To accomplish this, a well-characterized set of data is needed that spans a more significant parameter space. The aim of this work is to design and perform several experiments (approximately 30 tests) that significantly extends the experimental parameter space. The experiments are diagnosed as completely as possible given time and cost constraints. It is critical that these experiments be highly reproducible and that the materials used be well-characterized. Consequently, attention has also been paid to both these issues. In this paper we report initial experimental results that establish the repeatability of the experiments and report the effect of varying the density as well as the effect of piston speed and ignition delay on the DDT event. We describe the experimental setup in the next section, followed by results from the particle characterization and DDT experiments.

EXPERIMENTAL

The apparatus used in the DDT tube experiments is shown schematically in Fig. 1. The DDT tube used was a thick walled 77.6 mm o.d. stainless steel 304 tube with a 96.5 mm length and 6.4 mm i.d. The 143:1 inner to outer cross sectional area provided enough inertial confinement to contain the DDT event radially. The piston was loaded into the bottom end of the tube and set flush with the bottom surface. HMX was incrementally loaded directly into the tube in 3 mm lengths, and a lexan plug was set in the end to temporarily confine the bed.

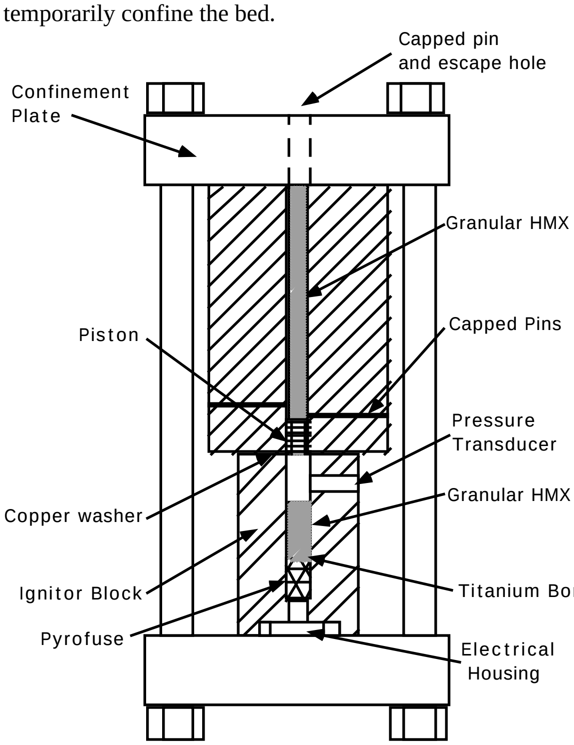

Fig. 1 Diagram of DDT tube experiment.

The ignitor block was made of vascomax 250 maraging steel. A PYROFUSE® was set into the bottom of the chamber with a thin mixture of titanium and boron, a gasless pyrotechnic, covering the bridge wire. A load of loose HMX in the burn chamber was ignited by the burning titanium and boron and the burning HMX powder raised pressures enough to drive the piston into the bed of HMX leading to initiation. A pressure transducer (PCB 109A02) was mounted into the side of the burn chamber, and in the first two tests, side closure pins were positioned at four locations near the bottom of the tube. The mating lip between the ignitor block and main tube was sealed with a thin copper washer to prevent venting, and the entire assembly was held together with four one inch thread bolts. A small hole in the upper confinement plate allowed visual inspection of tube for residual explosive before disassembly. On the four later shots, a capped pin was set in the top of the tube as an indicator of when the detonation had reached the end of the tube. All pressure data were collected using a pair of LeCroy 9400A Dual channel oscilloscopes. The PYROFUSE® was ignited by a low voltage CDU, and the capped pin data were collected by a time interval meter.

PARTICLE CHARACTERIZATION

It is widely accepted that particle size distribution in granular beds of HE plays a large role in DDT events. Particle size analysis in the past has been performed with little or no detail. To better understand the effects of particle size distribution, extensive analysis has been done on the batch of course granular HMX (class A 920-32) used in these experiments and in other related experiments. This analysis helps to clarify the initial state of the particles, as well as extend the understanding of the effects of high density pressing on the particle size and shape.

The goal of the particle size analysis was to measure the crystal size distribution of the HMX as accurately as possible. Basic analysis was done using a Coulter LS 230 particle size analyzer which uses light scattering by particles to measure size distributions. Samples were taken from solutions of about 0.1 g HMX in a bath of 10 ml of distilled water. Due to quick settling of the larger particles, special care was taken to obtain samples representative of the entire distribution by rapidly stirring the solution with a magnetic stir bar. Samples of approximately 1 ml were quickly transported from the solution to the particle analyzer using a dropper.

A series of HMX samples was pressed to various densities ranging from 1.24 g/cc to 1.81 g/cc (65% to 95% TMD). The samples were pressed using exactly the same procedure as in the packing of the DDT tube. Several runs were taken of each sample to verify accuracy and repeatability. The original particle size distribution was found to have a mean of about 270 µm, whereas samples pressed to the highest density (1.81 g/cc) had means of about 120 µm. The measured size distributions (by volume percent) are shown in Fig. 2a. Taking the difference between the original distribution and the pressed distributions it becomes clear that certain sized particles were being broken. (see Fig. 2b). We see that particles around 270 µm are being broken up, forming smaller particles whose peak is near 40 µm. The statistical parameters obtained from these measurements are listed in Table 1.

The higher density samples formed pellets which were easily broken apart, however possible agglomeration due to pressing needed to be addressed. A sonication bath has often been used in particle sizing to disperse HMX grains in the solution and to break up agglomerates formed during the pressing procedure. This operation, however is performed at the risk of breaking up individual crystal structures as well.

An analysis of the effect of the duration of sonication on the particle size distribution was investigated using the original unpressed HMX. Figure 3 illustrates the effects of sonication on the original material. A sample of HMX was tested without sonication, and subsequently after 1 minute, 5 minutes, 15 minutes, and 30 minutes of sonication. The samples that were sonicated for only one and five minutes showed distributions only moderately changed from the original distribution. Significant effects were evident, however, in the 15 minute bath as the mean particle size shifted from 270 µm to 92 µm. Extended periods of sonication resulted in the formation of smaller particles.

To better disperse the samples while leaving HMX particles undamaged, the pressed samples used in the first set of tests were sonicated for one minute and re-entered into the analyzer. The results of this series of measurements on the effect of pressing and short-duration sonication are shown in Fig. 4. The distribution statistics are shown in Table 2. Dramatic effects are observed. Highly fractured particles from the higher pressing densities are easily broken into smaller particles by the short-duration sonication. As pressing density is increased, the particles become increasingly fractured and fewer facets can be identified. Clearly sample preparation can significantly affect size distribution measurements.

The damaged state of the pressed samples was verified by SEM and optical microscope images. Figure 5a shows typical unpressed HMX crystals with rough surface textures and various inherent surface flaws. The structures, however, appear to be solid with no major cracks. Significant loss of structural integrity becomes apparent, however in the 75% TMD crystals as shown in Fig. 5b. Part of the original crystal surface remains in tact as evidenced by the rough surface on the lower portion of the crystal. The upper faces of the particle, however, are smoothly cut due to recent cleaving, and show large cracks throughout the structure. Further cracking and brittle failure occur at higher densities as shown in Fig.5c and Fig.5d.

DDT TUBE EXPERIMENTS

Table 3 lists the DDT tube experiments performed and list the conditions considered. Figure 6a is a photo of a cross-sectional view of the base case (shot #1). There is a change from dark smooth walls to lighter walls, followed by darker pitted walls downstream. The color changes are visible in the photo, but are more identifiable visibly. Past analyses at LANL and elsewhere (see Luebcke, Dickson et al. 1996) have correlated the color changes to events in the transition process by comparing pin and slit-camera records with color changes on recovered walls. For example, the first dark to light change appeared to correspond to the intersection of a rearward traveling wave (driven by the detonation) and the initial burning wave in DDT experiments (Luebcke, Dickson et al. 1996). The second color change (light to dark) corresponded to the onset of detonation. Our analysis uses both color change and wall profile to define points that can be correlated to the DDT process (see Fig. 6b). Light intensity analysis with a reflective light microscope was used to accurately define the point of transition between light and dark regions, while digitized data from scanned images of the cross sections yield the wall profile. The wall profiles obtained have many of the same characteristics as previous thermally ignited tubes (Luebcke, Dickson et al. 1996). One distinguishing feature of these tubes is the widely expanded region followed by a region where the expansion decreases. This likely indicates that detonation occurred in the pre-compacted region, producing higher pressures, as well as, in the initial low density material. This scenario is consistent with highly diagnosed DDT experiments by (McAfee, Asay et al. 1991) who, under similar conditions, observe detonation beginning within the material compacted by the initial compaction wave. This indicates there are some qualitative differences between thermally ignited and piston-initiated experiments.

Closure pin data was obtained near the initial location of the piston in some of the experiments. This is shown in Fig. 7 for shots #1 and #2. There was a long ignition delay (about 400 ms compared with delays <100 ms in the other experiments) due to tight packing of the titanium and boron in the ignitor block. This delay was probably accompanied by some burning in the chamber which pushed the piston into the bed slightly. Nevertheless, by shifting the initial time to correspond to the closure of the first pin in both shots #1 and #2 reasonable comparison between the two sets of pins is obtained. A wave speed of about 300 m/s is observed which probably corresponds to the initial compaction wave. The ignition delay, and probable small movement of the piston from it's original position before full ignition appeared to shift identifiable points further downstream in the tube by 3-4 mm consistently. This is seen in Table 4 where the identified points are listed for the six experiments reported here.

Burn chamber pressure was also measured in some of the tests (see Fig. 8). The pressure rises quickly as the loose HMX burns in the chamber. The pressure attained in the high chamber load (shot #4) is slightly more than a factor of two higher than the base case (shot #1). The pressure is slightly higher in the 85% TMD case (shot #6) than in the base case (shot #1) due to the increased resistance to the movement of the piston by the higher density bed. Also shown in Fig. 8 is the time when detonation was indicated by a closure pin near the end of the tube.

An important objective of this work was to have a very repeatable experiment. Consequently, we repeated shot #1 as closely as possible (shot #3) and compared the results. We found that the two experiments were very close as indicated by the wall deformations reported (see Fig. 9) and other points of reference (see Table 3). These results are an indication that the experiments are fairly repeatable. Additional tests will hopefully verify this further.

Figures 10a,b summarize the results obtained so far. Figure 10a shows the location of the first color change and Fig. 10b shows the second color change for the six cases. These color changes have been correlated to the transition event, as discussed above. It is clear that increasing the burn chamber pressure (piston speed) yields a much shorter run-to-detonation, as expected. A slight decrease is observed at 75% TMD compared with 65% TMD, however, at 85% TMD there is a significant increase in the run-to-detonation. Although more data is needed to better define this curve, results appear qualitatively similar to those obtained by (Griffiths and Groocock 1960). Interestingly these results were obtained in piston-initiated DDT experiments that circumvent the initial convective burning mode. Convective burning issues have often been used to explain much of the U-shaped plot, however, convective burning is very likely irrelevant in these experiments. Increasing the density yields more rapid pressurization because more explosive is available in a given volume leading to a shorter run-to-detonation. There is a limit, however, after which further increases in density inhibits the formation of ignition sites. This type of effect has been explored to explain plug formation (Son, Asay et al. 1995).

SUMMARY

Initial results from a new series of experiments in heavily confined granular HMX were presented. These experiments were designed to be useful in model development and evaluation. The effect of density (3 densities so far), piston speed (2 speeds), and ignition delay on the DDT event have been considered. We have measured the particle size distribution of the original granular explosive and pressed (higher density) samples. Scanning electron microscope (SEM) pictures were presented for the materials used. These pictures were used to interpret the size distribution measurements and to more fully characterize the initial condition of the materials used. The size distribution changes significantly and the particles become increasingly fractured with pressing. Further, we found that sonication can significantly affect size distribution measurements. This effect becomes more apparent for the more fractured materials (higher pressed densities). For the base case (65% TMD) two DDT experiments were performed yielding nearly identical results. This indicates that reproducibility was good. Run-to-detonation, as indicated by color change and deformation of the on the inner wall, for the three densities (65%, 75%, and 85% TMD) considered show a slight decrease in run-to-detonation going from 65% to 75% TMD and a significant increase at 85% TMD. Increasing the piston speed decreased the run-to-detonation. The effect of the ignition delay (tighter packing of titanium and boron) extended the run-to-detonation.

Several additional experiments are planned. The initial density will be varied over a broader range to better define the “U-shape” run-to-detonation plot. A few thermally ignited experiments, using the same tubes used here, will be performed to further examine the differences from piston-ignited experiment. RDX is very similar to HMX, but burns very differently. For example, the “stand-off” distance of gas phase reactions in burning HMX is much larger than for RDX, at least at low pressures. To investigate this effect on the DDT event, RDX (with nearly the same size distribution) will be used in place of HMX also. As we press the granular HMX we are changing the density as well as the particle size distribution. To separate these two effects we plan to press to a higher density, deconsolidate the material and test the pre-pressed material at our standard low-density (65% TMD). To better understand the effect of particle size on DDT, nearly monosized distributions of coarse and fine HMX particles will be tested. These experiments will use the same HMX as palnned for use in gas gun experiments. Also, to determine the significance of wall effects, similar tests will be run with tubes of larger i.d., while longer tubes may be implemented to confirm the development of a steady detonation wave. There remains some ambiguity concerning the interpretation of the pin and wall records. To clarify these issues we plan to use microwave interferometry to continuously measure piston velocity (at least part of it), compaction wave, and finally the detonation wave.

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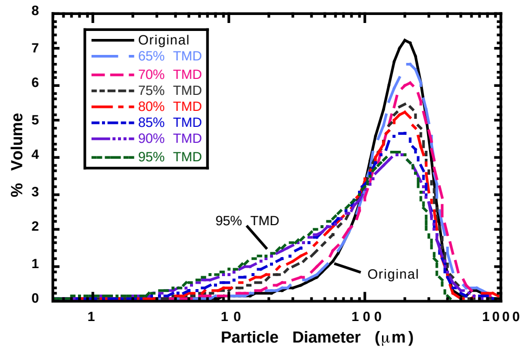

Fig. 2(a) Particle size distribution of original HMX.

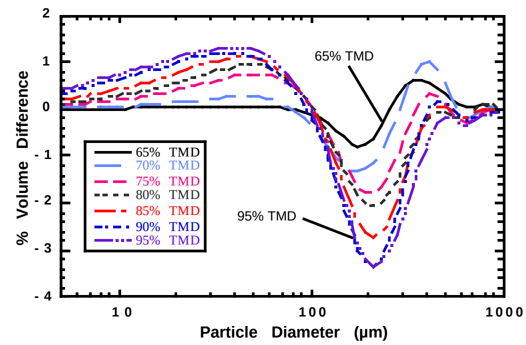

Fig. 2(b) Original size distribution subtracted from pressed particle distributions.

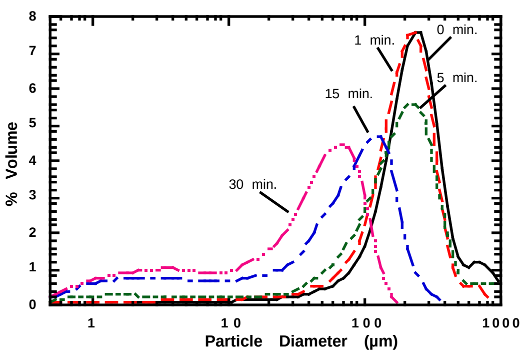

Fig. 3(a) Particle size distribution after various durations.

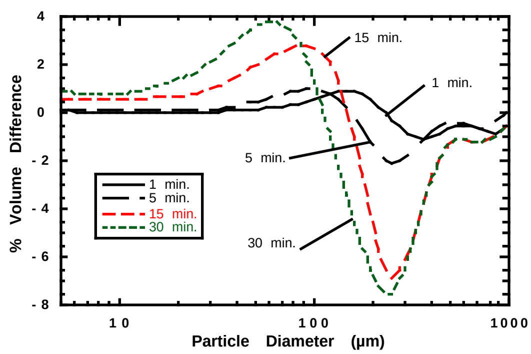

Fig. 3(b) Original size distribution subtracted from sonicated particle distributions.

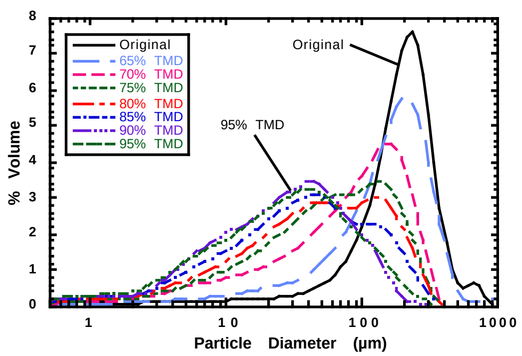

Fig. 4(a) Particle size distribution of pressed HMX after one minute in low powered sonicating bath.

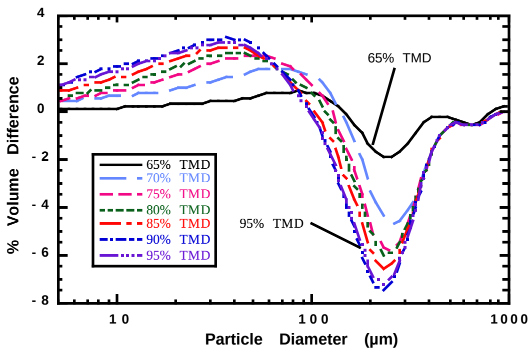

Fig. 4(b) Original size distribution subtracted from pressed samples that were sonicated for 1 minute.

Table 1 Particle size statistics for pressed HMX.

% TMD	Mean (μm)	Median (μm)	Mode (μm)	S.D. (μm)	Skewness	Kurtosis
Poured	192.8	178.2	203.5	127	2.99	19.3
65%	200.3	181.9	223.4	136	2.6	16.8
70%	220	181.8	223.4	222	4.23	23.9
75%	161.9	136.2	203.5	140	3.22	25.5
80%	160.8	137.0	203.5	155	4.24	31.7
85%	144.0	121.0	185.3	128	2.8	19.3
90%	130.8	104.8	185.3	115	1.3	2.4
95%	113.2	92.61	168.8	93	.794	-0.129

*Statistics are according to particle diameter.

Table 2 Particle size statistics for pressed HMX sonicated for one minute

% TMD	Mean (µm)	Median (µm)	Mode (µm)	S.D. (µm)	Skewness:	Kurtosis
Poured	217.0	204.7	245.2	132	1.29	3.58
65%	204.6	170.6	223.4	213	4.27	24.9
70%	108.2	92.32	153.8	85.2	0.745	-0.124
75%	85.03	61.56	140.1	75.8	1.15	0.858
80%	76.15	49.66	140.1	74.1	1.34	1.42
85%	62.02	37.86	45.75	66.3	1.72	2.95
90%	41.70	28.88	41.67	41	1.8	4.35
95%	45.41	29.05	41.67	49.1	2.06	5.29

*Statistics are according to particle diameter.

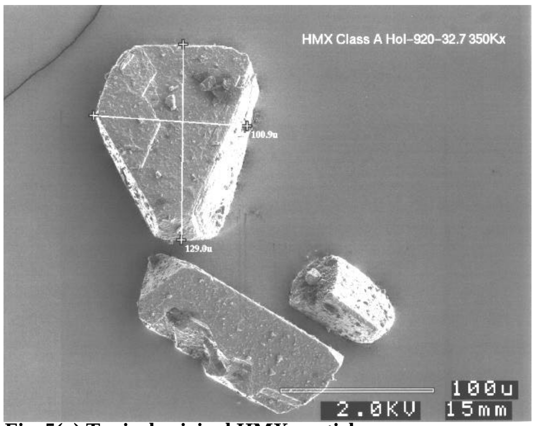

Fig. 5(a) Typical original HMX particles.

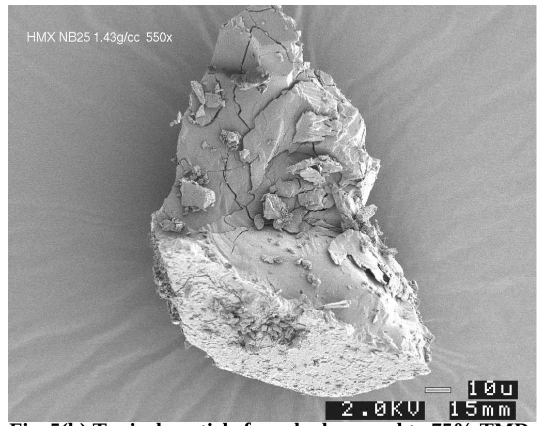

Fig. 5(b) Typical particle from bed pressed to 75% TMD.

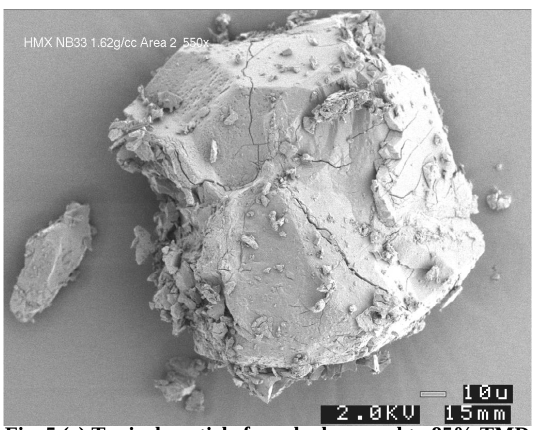

Fig. 5 (c) Typical particle from bed pressed to 85% TMD.

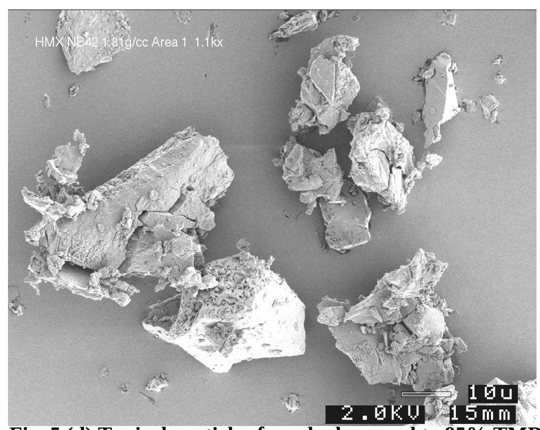

Fig. 5 (d) Typical particles from bed pressed to 95% TMD.

Table 3 Summary of DDT experiments.

Shot #	% TMD	Load In Burn Chamber (g HMX)	Capped Pins	Pressure (kpsi) *	Comments
1	65%	0.64	yes	-	base case
2	65%	0.64	yes	-	tightly packed TiB
3	65%	0.64	no	39	repeat of base case
4	65%	1.19	no	107	increased piston speed
5	75%	0.64	no	-	high density bed
6	85%	0.64	no	47	higher density bed

*Maximum pressure in burn chamber before detonation

Fig. 6(a) Cross section of base experiment (shot #1) using 65% TMD HMX powder.

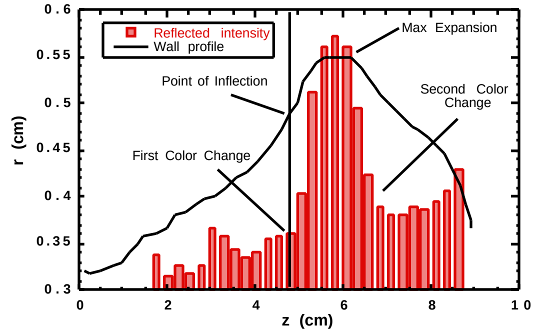

Fig. 6(b) Base experiment (shot #1) wall profile and reflected intensity (arbitrary units) as a function axial location, z.

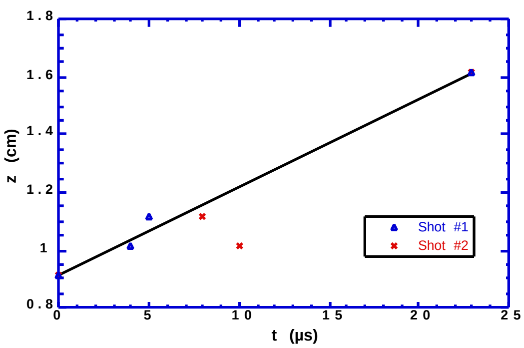

Fig. 7 Pin closure times of first two experiments (shots 1&2).

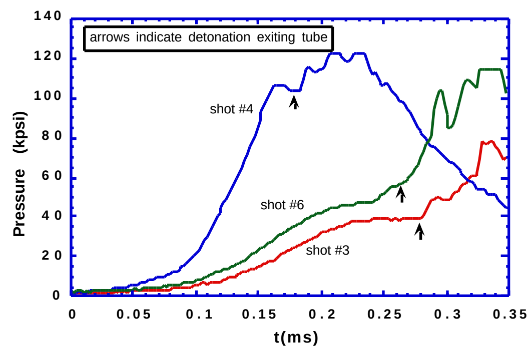

Fig. 8 Burn chamber pressure traces. Arrows indicate time of side closure pin reporting.

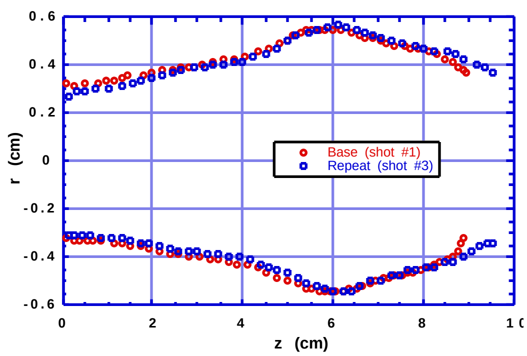

Fig. 9 Overlay of shots 1&2 inner wall profiles.

Table 4 Color and profile data taken from tube record.

Shot	1st Color Change	2nd Color Change	Point of Inflection	Point of Max Expansion	Max Expansion	Extrapolation to I.D.*
1	4.6	6.2	4.66	5.94	0.544	2.39
2	5.0	6.7	5.10	6.20	0.531	2.90
3	4.7	6.4	4.82	6.11	0.546	2.90
4	3.3	5.4	3.28	4.93	0.546	1.28
5	4.5	6.1	4.59	6.00	0.599	2.20
6	6.6	8.1	5.84	7.68	0.568	-0.21

*Extrapolation taken at point of inflection back to original i.d. using slope measured at the point of inflection.

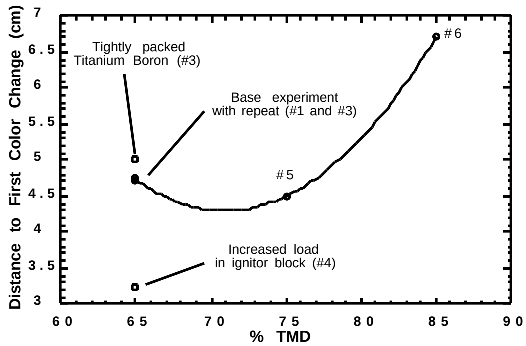

Fig. 10(a) Distance to first color change.

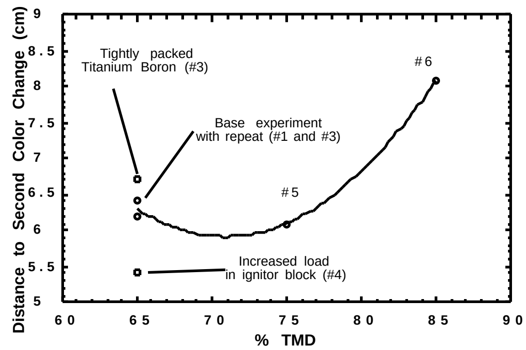

Fig. 10(b) Distance to second color change.

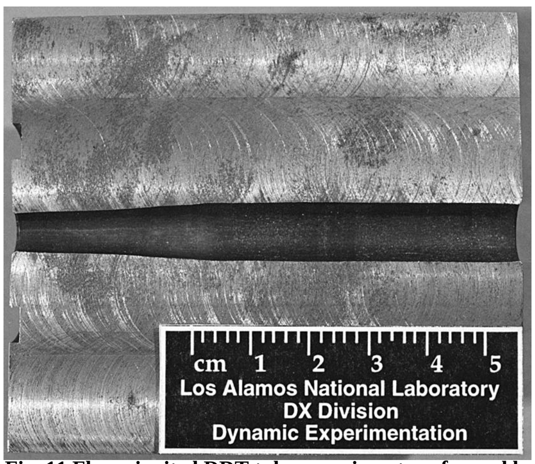

Fig. 11 Flame ignited DDT tube experiment performed by Blaine Asay and Gary Laabs using 65% TMD HMX powder (unpublished result).