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Small Scale Thermal Violence Experiments For Combined Insensitive High Explosive And Booster Materials

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Abstract. A small scale cook-off experiment has been designed to provide a violence metric for both booster and IHE materials, singly and in combination. The experiment has a simple, axisymmetric geometry provided by a 10 mm internal diameter cylindrical steel confinement up to 80 mm in length. Heating is applied from one end of the sample length creating pseudo 1-D heating profile and a thermal gradient across the sample(s). At the opposite end of the confinement to the heating block, a machined groove provides a point of rupture that generates a cylindrical fragment. The displacement of the external face of the fragment is detected by Heterodyne Velocimetry. Proof of concept experiments are reported focusing on HMX and TATB formulations, and are described in relation to confinement, ullage and heating profile. The development of a violence metric, based upon fragment velocity records is discussed.

Introduction

For improved safety during development and scale-up of new and modified explosive formulations data are required on the response of the materials to credible handling and storage scenarios including cook-off [1].

Interest in cook-off behaviour endures throughout the final article lifetime, with experiments and modeling being fielded in pursuit of a predictive response capability. Standard studies include sentencing tests such as burning tube and fuel fire which are qualitative and present results as numerical response levels [2]. Other experiments can be used to generate validation data and a predictive capability for models, such as STEX [3] and SITI [4]. However, the explosive masses required for these experiments and their general complexity makes them difficult to field on new explosive formulations where typically only few hundred grams are manufactured.

Regulatory drivers require the selection of adequately performing materials with the lowest sensitiveness, explosiveness and mass. One difficulty is defining explosiveness or violence for small-scale reactions. The approach taken here is to quantify metal pushing ability. The challenge was therefore to develop a small scale cook-off experiment where violence of response, from a benign confinement rupture through deflagration to detonation is measured quantitatively and reproducibly.

Scoping experiments were conducted using a LANL thermal cook-off gun assembly using TATB and other formulations. The experiment had previously been optimised to study the cook-off response of PBX9501 under varying inertial (projectile) and static (burst disk) confinement [5, 6].

Since candidate materials included TATB, a material known to pressure burst confinements prior to DDT, it was decided that a strongly sealed system was required to retain reactive intermediates for exothermic gas phase reactions [7]. A variable point of weakness, or rupture disc, was designed to be accelerated by the reaction products and act as a quantitative metric of violence.

Previous work on deflagration to detonation transition (DDT) in granular HMX gives a good indication of the strong confinement and porosity required to achieve a short run distance to detonation [8, 9]. Extensive damage from thermal conditioning is necessary to reduce the run distances to the scale of this experiment. Work on granular HMX with approximately 30% porosity achieved run distances of 19 mm for heavily confined experiments and recent work at LANL shows that with relatively low porosities run distances of 50-100 mm are observed [10].

In order to achieve maximum extent of reaction and still use the minimum sample masses, it was necessary to investigate a range of sample cavity lengths and temperature soak profiles.

Experimental

A small scale cook-off experiment, figure 1, was designed to provide a high fidelity violence metric for both IHE and booster materials, singly and in combination. The experiment has an integral burst disk to allow thermal conditioning of formulations for extended periods without breaching of the confinement.

An axisymmetric geometry with a 10 mm internal diameter cylindrical 070M20 mild steel (1018 US Grade) confinement up to 80 mm in length was designed. Confinement is maintained up to several GPa by an internal pressure inconel c-seal. Heating is applied from one end of the sample length via two/three 150W cartridge heaters in a copper or brass heating blocks. Temperature control is provided by a thermocouple in the heating block. Additional thermocouples were inserted into the steel confinement. At the opposite end, a machined groove provides a point of rupture that generates a cylindrical fragment. The displacement of the external face of the fragment is detected by heterodyne velocimetry and is triggered by a peizoelectric pin.

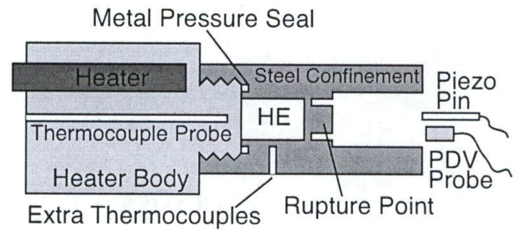

Fig. 1. A schematic of the experimental assembly.

Table 1. Formulations & PBXs used in cook-off experiments

Label	Composition data	Pellet length (mm)
L	Isostatically pressed 91:8 HMX/NC	4
M	Isostatically pressed TATB/Fluoropoylmer	4
N	Die-pressed 92:8 HMX/HTPB shear mixed	8 & 10
P	Die-pressed 92:8 HMX/HTPB	8 & 10
Q	Die-pressed Tetryl	10
R	Die-pressed 91:8 HMX/NC	10

Integration of the velocity vs. time history allows the fragment displacement to be found. Thus, the movement of the disc while still blocking the passage of reaction gasses to the atmosphere can be distinguished from free fragment flight. Once in free flight, rapid expansion of the product gases will occur by escaping around the sides of the fragment.

Calculations using the Sandia Hydrocode CTH were undertaken during the confinement design stage to verify that under both deflagration and full detonation, the rupture plug was correctly sheared from the body and would strike the piezo trigger pin.

For the experiments, the samples were subjected to a pseudo 1-D heating profile at rates of 1°C min^{-1} and 10°C min^{-1}. Extended thermal soaking at 170°C or 180°C was performed on HMX

Table 2. Relationship of the groove depth to shear area and rupture pressure.

Groove depth / mm	Shear area / mm^2	Rupture Pressure / MPa (kpsi)
5	25.1	134 (19.4)
4	50.2	316 (45.8)
3	75.2	$>$ 400 (58)

based samples in ramp-soak type experiments and at 250°C for TATB based samples. Materials studied are listed in table 1.

Upon rupture, a projectile would be generated. The velocity was dependent on the violence of reaction of the HE. Velocity was recorded by a Tektronix TDS 6804B 8GHz 'scope and Doppler velocimetry (Het-V), using a 4mW, 1550 nm diode laser [11, 12]. The data generated by the Het-V system were processed using a Fast Fourier Transform (FFT) method in an Igor Pro routine.

Results

Static pressure rupture testing

Several prototype confinement vehicles were manufactured with differing burst disk configurations. The designed point of weakness relies on a machined groove on the outside of the body. The depth of this groove determines how much steel the growing reaction must fracture in order to release the pellet. Thus a 5 mm groove leaves less material and offers less confinement than a 4 mm groove, etc. The area of material to be sheared is shown in table 2 together with the measured quasi-static burst pressure for the three groove depths.

Cook-off: 10 mm length sample confinements

Cook-off experiments were undertaken using pellets of composition L as a source of well characterized material from which reactions of appreciable violence in confined cook-off could be expected. Pellet length was fixed at 4 mm as the samples were legacy machined material. Two pellets were used in each experiment leaving a 2 mm long void space at the burst disc end of the sample volume equivalent to 19% ullage. Originally it was thought that the

Table 3. The conditions for 10mm cook-off experiments. The ramp rate was set at 10°C min^{-1} for both ramp-to-failure and 1.5 hr soak experiments. * 5 hour soak, ** soak at 190°C.

Expt.	Steel groove (mm)	Profile	Expt.	Steel groove (mm)	Profile
L1	5	soak	Q2	4	rtf
L2	4	soak	LM1	5	rtf
L3	4	soak*	LM2	4	rtf
L4	4	soak**	P1	4	soak
L5	4	rtf	P2	4	soak
L6	3	rtf	N1	4	soak
L7	3	soak	N2	4	soak
M1	4	rtf	Q1	4	rtf

10 mm long body would allow sufficient extent of reaction to distinguish between material responses without transitioning to detonation.

From figure 2 it may be seen that the experiment differentiated well between HMX based composition L samples subject to different soak times but under the same confinement. These data suggest that there is an optimum extent of degradation which gives the greatest reaction violence. This result is intuitive, as a violent reaction is expected to require significant porosity to support ignition but also enough material remaining for the reaction to propagate.

However, the 3 mm groove depth confinement has the effect of reducing the pellet velocity compared to a 4 mm vehicle under the same conditions. It is most likely that this results from the 3 mm confinement requiring more of the available energy to promote fracture and therefore results in a lower velocity.

For comparison, the results from a TATB based composition M cook-off suggest a slow, ductile failure in the manner of a pseudo-static pressure burst.

Experiments with a single pellet of composition L against the heater block followed by a pellet of composition M as shown in figure 3 (HMX:TATB). A 2 mm ullage space was left (20%) and a 4 mm groove depth confinement was used.

Experiments LM1 and LM2 follow the trend of the composition L data for similar confinement and conditions, despite having half the mass of HMX

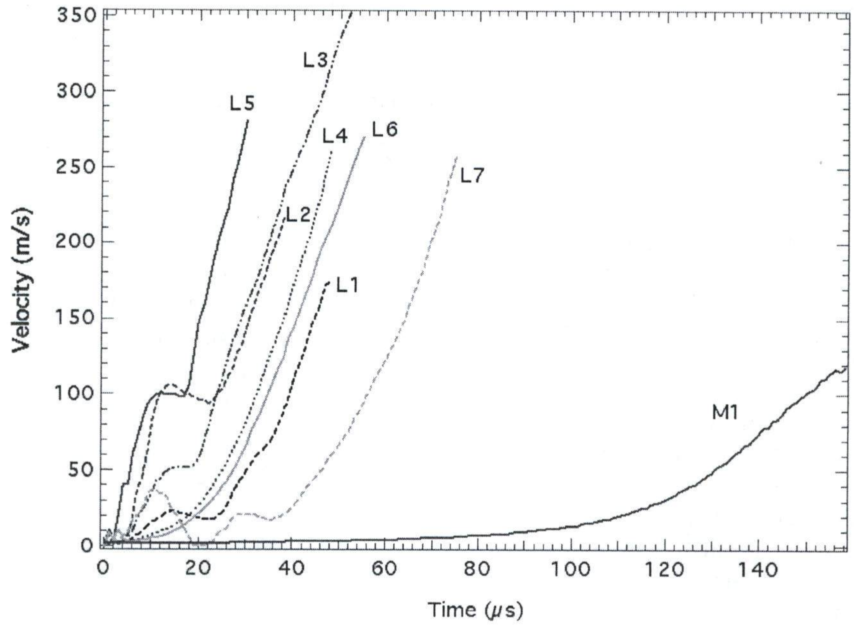

Fig. 2. Velocity/time profiles for HMX based composition L cook-offs in which confinement, soak duration and soak temperature were varied. A TATB based composition (M) cook-off is included for comparison.

present. There appears to have been little contribution from composition M (TATB) with unburnt material scattered around after rupture. In both cases the fragment velocity is influenced by the mass of HMX present.

Fragments from some of the 10 mm cook-off experiments were examined for evidence of shear localization during fracture. It has previously been shown that in 1018 steel, shear localization is suppressed unless the material had been shock prestrained [13]. This prior finding was confirmed for the experiments presented here where optical microscopy of the rupture surfaces showed no evidence of adiabatic localization. Additionally, it was observed that the more violent cook-offs produced cleaner fracture surfaces with less sign of plastic deformation of surrounding material.

Cook-off: The effect of ullage in 10mm sample length confinements

In an effort to determine the influence of ullage on the violence of response in these particular experiments, the HMX/HTPB compositions N and P were tested with and essentially without ullage.

In response to the applied thermal profile a volume change of approximately 7% [14] resulting from the $\beta - \delta$ phase change of HMX crystals would be expected [15]. Additionally there would be a small volume change due to thermal expansion of all constituents [a]. The configuration of the experiment delivers a thermal gradient across the sample such that the additional thermocouples show a temperature difference of 20°C across the sample confinement. This means that, assuming a linear gradient, the sample volume heated to the threshold for $\beta - \delta$ phase change will only be of the order of 3.5 mm^3 or 0.45% of the total. Also, since HMX does not begin appreciable degradation until approximately 170°C, the gas generation of these samples during soak will be low. Thus, the reduction in the ullage after heating is from approximately 20% to 19% and relatively little gas will be generated from the HMX to expand internal voids and pores within the thermally damaged HMX. In this way, the applied

[a]This excluded any compressible porosity that may also be generated.

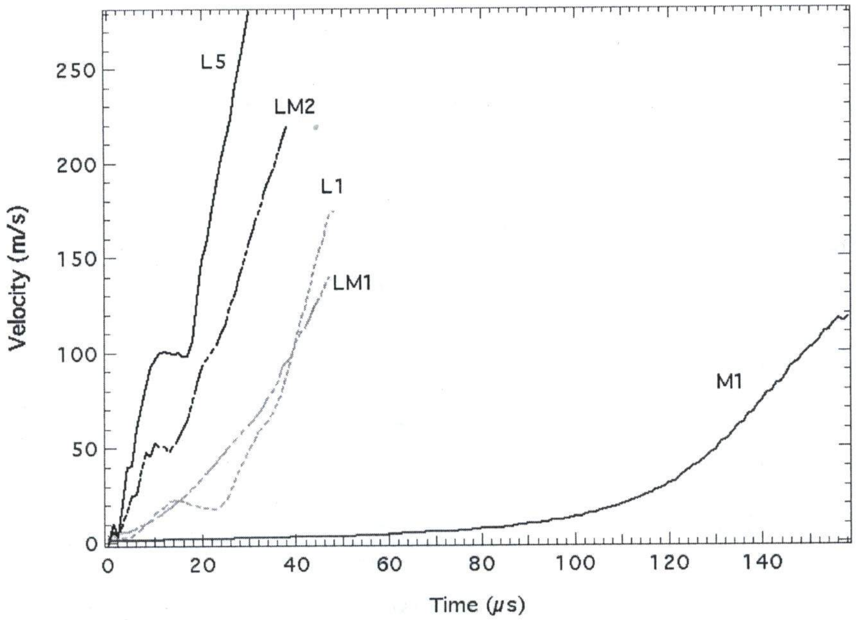

Fig. 3. Velocity/time profiles for composition L & M mixed cook-offs in comparison with separate cook-off of composition L and composition M

thermal profile is effective in raising the bulk temperature of the material without excessive material damage, gas pressure increase prior to burning or HMX mass loss.

Results of the HMX/HTPB based experiments are shown in figure 4. The trend for lower projectile velocities in experiments with no void space is followed followed by all samples. All of the experiments show relatively low projectile velocities which is attributed to incomplete burn of the sample.

The inclusion of Tetryl in the data set illustrates the issue of the sample length used in the experiment influencing the violence of response. Tetryl will run to detonation from cook-off in a distance of 70mm [16]. Sample lengths are restricted to 10mm in these experiments and similarly low velocities have been observed for both Tetryl and HMX/HTPB formulations. This implies that, like Tetryl, the HMX/HTPB formulations may show a greater response, and possibly distinction between formulations, with longer sample lengths.

Cook-off: 40mm to 80mm HE sample confinements

A table of cook-off experiments with fast and slow heating profiles that were performed on HMX based composition R pellets is shown in table 4. The groove depth in all cases was 4 mm. The body was insulated using glass fiber tape. In some experiments the insulation only extended half way along the body while in others the whole body was insulated. This lagging changed the thermal gradient along the explosive run length. The profile was recorded using up to four equispaced thermocouples.

It was determined that in order to obtain the best possible coupling of explosive response to projectile velocity, an alteration of the brass heater was necessary. It had been observed in some experiments that the brass heater had become prematurely detached from the confinement body despite the rupture of the fragment. The brass heater was altered to allow a short brass projection (2 mm long) to enter the sample containing tube. This was done in an effort to reduce the dynamic force experienced by the sealing face and the threads by swelling of the

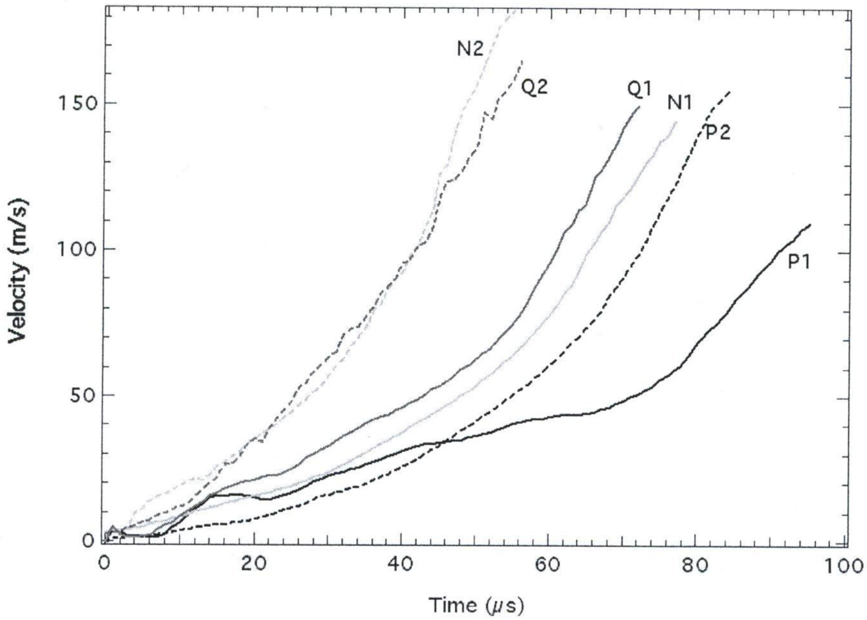

Fig. 4. Velocity/time profiles for HMX/HTPB and Tetryl.

Table 4. A table of 40 mm and 80 mm experiments with and the associated thermal profile applied. RTF is continuous temperature ramp-to-failure. The profile for the 1.5 hr soak at 180°C was rampt to hold 10°C min^{-1} followed by 10°C min^{-1} to reaction.

Ramp	Half body lagging	Full body lagging
RTF 10°C min^{-1}	R2 *R4* *R5*	
RTF 1°C min^{-1}		*R7* *R8* (80 mm)
180°C 1.5 hr + RTF 10°C min^{-1}		R1 *R3* *R6*

brass projection under high pressure. The modification was successful in delaying separation of the heater from the body until such time as the rupture disc was in free-flight.

All confinement bodies had sample cavities 40 mm in length except experiment R8 which had a sample cavity of 80 mm. Experiments in italics in table 4 represent those conducted with the altered brass heating block. With the 2 mm intrusion into the confinement the full complement of 4 × 10 mm pellets could not be accommodated. Therefore 3 × 10mm pellets were used leaving an 8 mm long void space at he burst disk end. All other experiments had no ullage.

Referring to figure 5, it can be seen that experiments with the same thermal profile exhibit velocity profiles which are similar after the heater modification. At early times in the velocity profile, before the fragment is in free-flight, the curves almost overlap. Further, the data indicates that the length of the insulation (lagging) on the experiment is negligable in comparison to the influence of ullage and thermal profile.

In the overlapping trace cases, the curves indicate the intrinsic response of the HE under the given conditions. This intrinsic response may then be used to derive a violence metric based on the gradient of the early velocity record. While the fragment is still blocking the flow of most reaction gases to the atmosphere, the motion will be heavily influenced by the mechanical strength of the steel which depends entirely on the strain rate induced by the hot gases generated in the confinement and temperature. Essentially, this is the rate and extent of reaction of the explosive.

Once the fragment is in free flight, the motion will be a complex mix of acceleration from gases coming from a now pressure relieved reaction, air drag and residual velocity from the fracture process. This part of the data are therefore less useful. As previously described, the timing of separation of the heater block from the confinement was variable, but always occurred after the projectile had entered free flight after the modification.

Experiment R7 shows the highest projectile velocity. However, early time data for velocity profiles R4, R5 and R7 almost overlaps. Some other traces (R1 & R2) show lower velocities, however, in these cases the heater modification was not used. Some experiments were repeated (R4 & R5 and R3 & R6) and show good reproducibility.

One of the aims of the experiments was to obtain a quantitative measure of violence with respect to metal-pushing ability. The gradients of the velocity/time profiles up to free flight offer the best basis for such a metric. It must be determined if other formulations converge on a single intrinsic velocity profile per confinement vehicle as HMX based composition R does.

Cook-off: Confinement expansion

The sectioned confinement vehicles from experiments with differing velocity records were examined.

The R7 vehicle in figure 6 shows evidence of an ignition(1) and growing reaction running in the direction of the burst disk up to a maximum (2). The deformation near the burst disk end appears to reach a steady velocity (3) but measurement of the vehicle show that this point corresponds to the end of the explosive pellet and the region of ullage. The position of the inside face of the burst disk is shown by (4) and the position of the bottom of the machined groove by (5). There is no evidence to suggest that DDT was reached.

In order to provide a comparison with a true detonation, an experiment was undertaken with a sample of C4 and an L1A1 detonator. A velocity profile was recorded and the fragments of the vehicle were collected. A photograph of the pieces is presented in figure 7. The fragmentation was extensive with the greatest damage at the heater end where the detonator was located. None of the cook-off experiments undertaken show any confinement fragmentation approaching that of the detonated confinement. The velocity record for the detonative test, S1 in figure 5 shows that the velocity profiles for the series of composition R exhibit much lower gradients.

A different pattern of deformation was found in the confinement vehicles of experiments where relatively low velocities were observed, an example (R2) is shown in figure 8.

Steel Hardness Testing

There is a well known reversible $\alpha - \epsilon$ phase transition in iron and iron based alloys at $\approx$ 13 GPa [13]. Upon shock release back to 1 bar, residual microstructural changes remain that increase the hardness of the material. It was wondered if this harness 'signature' might be useful in detecting localized, and ambiguous, regions of detonation in steel confinements such as used in this research.

Vickers hardness (HV) tests were undertaken on as-received confinement steel and the average was found to be 195 $\pm$ 5 HV. The heated confinements in which deflagration had occurred had undergone significant plastic deformation at pressures significantly below the $\alpha - \epsilon$ phase change. The measured hardness from inside to outside the cylinder wall dropped from $\approx$ 260 to $\approx$ 240 HV. Considerable scatter in the values ($\pm$10 HV) was seen owing to the dirty nature of this steel but measurements were made in three confinements at a total of nine locations. Similar measurements were undertaken on a recovered fragment from the detonation experiment where the confinement was cold prior to ignition. The measured hardness was approximately the same across the wall thickness and had a value of 220 $\pm$ 10 HV. The detonated fragment showed less overall plastic deformation than the deflagration confinements.

From this it is clear that the hardening associated with plastic deformation of the confinement is greater than that from the shock hardening process. Therefore, measurements of steel hardness are not a suitable method of deciding if local detonation has occurred in low carbon steel confinements.

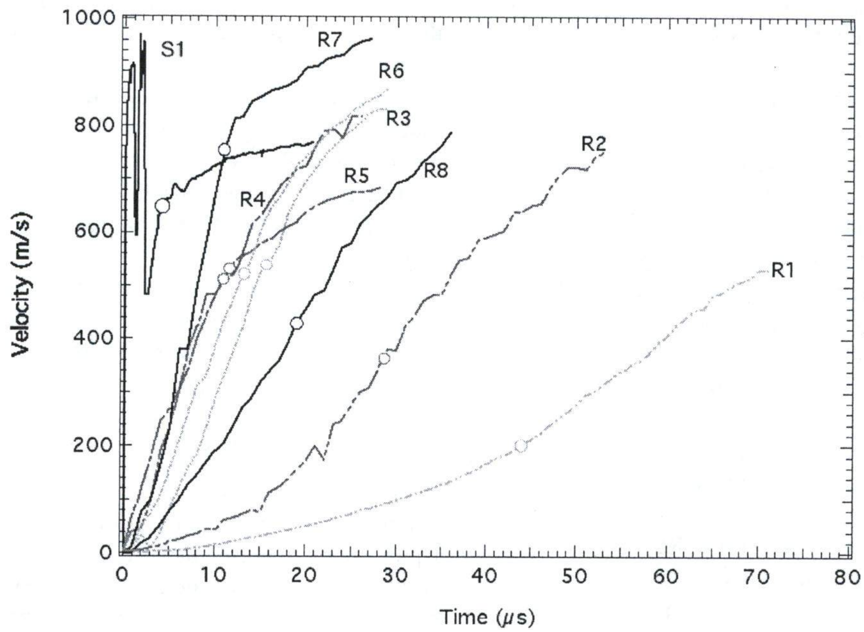

Fig. 5. Velocity / time data for the 40 mm and 80 mm experiments. Circles indicate the time at which the projectile has moved 3 mm from the starting position, after which it is considered to be in free flight. A detonative test (S1) is added for comparison.

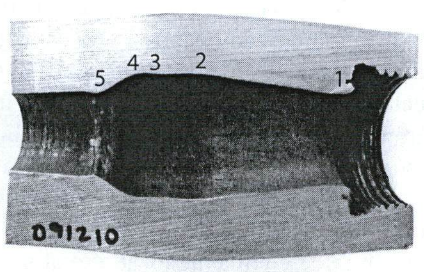

Fig. 6. The R7 confinement vehicle in section

Fig. 7. Fragments of the vehicle in the detonative test.

Conclusions

The data presented here demonstrates the difficulty of generating DDT events in small masses of high density secondary explosives even held under extremely strong confinement and heated using aggressive thermal profiles. It has been shown that run lengths of less that 10mm are not capable of fully exploring the response of the material. Although the results do show differences between materials, these differences in the velocity profiles are not significant enough to draw firm conclusions regarding the relative violence of reaction of the materials. This demonstrates that under the reported level of confinement pressure, burst occurs before the materials have fully reacted.

Further, it has been shown that ullage influences the extent of reaction and thus the velocity profile observed. The presence of ullage allows greater expansion and cracking of the sample, particularly

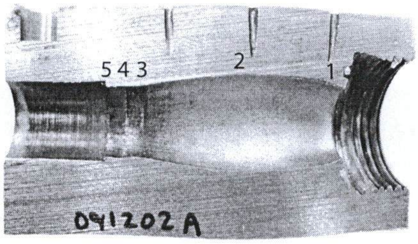

Fig. 8. The R2 confinement vehicle in section

when HE crystalline phase transitions are encountered, this increases the surface area available to the initial burn part of the DDT process and gives space for product gases to expand into. These factors allows the reaction to progress to a greater degree resulting in a more violent rupture. Additionally, there is greater PV energy stored in the pressurized gas within the chamber also increasing the velocity.

The 40 mm sample lengths have been shown to be adequate to explore the full extent of reaction possible in HMX with this level of confinement. Additionally, it has been shown that even with significant lagging and a slow heating rate allowing a greater degradation of the sample, 80 mm was not sufficient to induce a detonation owing to the high density of the undamaged starting explosive.

The data from the 40 mm experiments appears to show the maximum possible reaction of the material under these experimental conditions. From these data the early part of the velocity profile shows the greatest potential to be developed into a violence metric. The gradient of the velocity profile prior to free flight of the projectile is a function of the extent and rate of reaction of the explosive and the work done on the confinement vehicle to generate the projectile. Thus the gradient is the best indicator of the violence of reaction of the material and its ability to do work on a metal.

Data on the cook-off of different formulations is required to verify the suitability of this metric.

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