SAND2018-7274C

Development of a small scale thermal violence test

Malcolm D. Cook*, Christopher Stennett**, and Michael L. Hobbs#

*AWE, Aldermaston, Reading, Berkshire, UK RG7 4PR

**Centre for Defence Chemistry, Cranfield University, Defence Academy of the UK, Shrivenham, Wiltshire, UK SN6 8LA

Sandia National Laboratories, Albuquerque, NM 87111 USA

Abstract. The One-Dimensional-Thermal-Violence (ODTV) experiment, described in this paper, was primarily devised to determine Cook-Off violence while providing data for models. The ODTV capsule employs a spherical sample up to 30mm in diameter which is considered large enough to ensure that the true violence of the test material is captured. An important design feature is its high confinement to ensure that all the energetic material including gaseous intermediates, liquid and solid material are retained within the test vehicle up to the point of final event. A number of methods to heat the vehicle have been explored including a hot molten metal alloy bath and induction heating. The capsule is typically preheated to a set temperature, but it can also be heated over any profile. Firings have been carried out for a range of HMX based explosives some of which used HTPB as the binder and others using nitrocellulose and K10 as the binder In addition to the fragmentation of the capsule, Photon Doppler velocimetry (PDV) has been deployed to capture the mid-point cylindrical wall velocity. Smooth velocity traces were achieved in nearly all firings. A good correlation was found between the PDV derived wall velocity and the number of capsule fragments recovered. This demonstrates that PDV can be used as a metric of violence. Our data has been used to validate a pressure-dependent Cook-Off model developed at Sandia National Laboratories. This work emphasizes the importance of accounting for pressure effects during Cook-Off of HMX based explosives.

Introduction

If a system containing an explosive is heated to elevated temperatures, the time-to-explosion, the temperature profile and amount of material that undergoes the thermal explosion can be predicted using heat flow codes with embedded chemistry. This method can be applied for an explosive article in any thermal environment. Prediction of thermal violence is much more challenging since the mechanisms and processes involved are not fully understood. Small-scale experiments are required to identify mechanisms, provide input data and validate models.

The literature contains many papers that describe charge scale Cook-Off tests which range in size, shape and configuration[1-3]. Of these, the LLNL One-Dimensional-Time to eXplosion (ODTX) test is the smallest that is commonly used[1-3]. The ODTX experiment nominally considers 2g of explosive that is near the theoretical maximum density (TMD) and occupies about 1ml. Problems occur if there is

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leakage especially when the Cook-Off mechanism is dominated by gas-phase reactions.

Despite its small size, the ODTX test has proven to be very useful and is a classic experiment for determining reaction rate parameters for Cook-Off models to predict the Time-To-eXplosion (TTX), especially if the mechanism is not pressure dependent. ODTX is one-dimensional employing a small 12.5 mm diameter spherical explosive sample and has well-defined boundary conditions and confinement all of which lends it to modelling. The method involves rapidly placing the sample in aluminium anvils, preheated to a set temperature, until an explosion is observed. Repeating this procedure over a range of temperatures allows the reaction time versus temperature to be plotted. This effectively provides a measure of reaction rate with temperature, which can be plotted as log reaction time versus reciprocal temperature. However, it is difficult to obtain definitive violence data from the test since, at the point of thermal explosion, much of energy is used in deforming and expelling the copper seal that is placed between the anvils. In addition, it is quite common to observe a number of minor explosions before the final event due to loss of gas and reassertion of the anvils.

The One-Dimensional-Thermal-Violence (ODTV) experiment, described in this paper, was primarily devised to determine Cook-Off violence while providing data for models.

In this paper we give details of the ODTV test and results from some test firings on HMX based explosive compositions. We also present finite element model simulations of our experiment with predictions of thermal history, pressure history, and final ignition time.

ODTV overview

Like the ODTX the ODTV experiment employs a spherical sample[1-3]. The current ODTV capsule can hold samples up to 30mm in diameter which is considered large enough to ensure that the true violence of the test material is captured. It has a high confinement to ensure that all the energetic material including gaseous intermediates, liquid and solid material are retained within the test vehicle up to the point of final event. This is achieved via a double shell arrangement such that the two barrel shaped halves of the inner vehicle are orthogonal to two outer rings[2] (see Figs. 1 and 2).

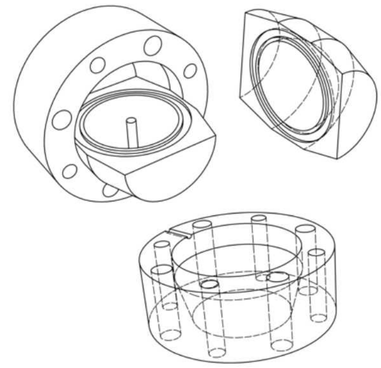

Fig. 1. ODTV capsule design, showing one inner shell (with instrumentation pocket) fitted into the compression ring, and the other shell and compression ring separately.

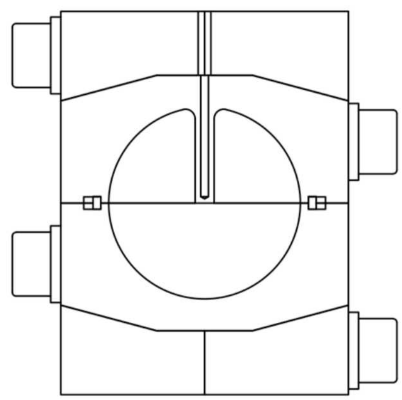

Fig. 2. Cross-section of ODTV capsule, showing tapered profiles of inner shells. The capsule axis is horizontal in this view

These outer locking rings are pushed and bolted together over the inner barrels crushing a copper sealing ring housed within machined grooves. The inner barrels house the spherical explosive charge. By this means the hoop strength of the outer rings controls the confinement rather than the strength of the bolts. A number of methods to heat the vehicle have been explored including a hot molten metal alloy bath and, more recently, induction heating. The capsule is typically preheated to a set temperature, but it can also be heated over any profile. By repeating over a range of initial temperatures until thermal explosion is observed, the experiment can replicate the smaller ODTX test. However, unlike the ODTX test, it does not require a dedicated machine and can be deployed in a standard firing facility.

Photon Doppler velocimetry (PDV) has been deployed to capture the mid-point cylindrical wall velocity. Smooth velocity traces are typically observed. Preliminary experiments showed good correlation between the PDV derived wall velocity and the number of capsule fragments recovered. This demonstrates that PDV can be used as a metric of violence.

Experiment description

The filled ODTV capsule was placed within a 70mm diameter coil (six turns) of hollow copper tube covered by glass fibre sleeving. Just enough space was left between the copper tube windings to allow two 'K' type thermocouples to be placed into the thermocouple port in the capsule body. A 0.25mm thermocouple was placed at the bottom of the port with its tip at the centre of the explosive charge. A 1.6mm thermocouple was placed alongside it such that is just touched the outside of the inner barrel shaped aluminium pieces holding the explosive sample. A further 1.6mm thermocouple was placed in an adjacent hole to a depth of 5mm to provide the feedback to the induction heater control unit.

The thermocouples were supported by a rectangular metal frame held in place using two of the capsule bolts. This frame also allowed the PDV fibre-coupled collimator lens to be held normal to the rounded capsule surface in order to achieve a strong reflected signal.

The ODTV capsule complete with thermocouples, PDV fibre and wound copper heater coil was placed inside a strong welded mild steel box on a piece of chipboard. The copper tubes were passed through a small hole and connected to a Draper 1KW induction heater switching at 70kHz. The induction heating was controlled by means of the 'K' type thermocouple placed in the body of the capsule connected to an ESM 4450 closed-loop process controller, capable of following a programmed heating rate profile.

The two instrumentation thermocouples were connected to a Picolog TC-08 logger, recording at one sample per second throughout the experiments. A piezo-electric trigger pin was placed alongside it to act as a trigger for the oscilloscope which served to record the PDV trace. The fibre optic probe was connected to a purpose-designed PDV. The beam of the laser was directed between two windings of the heater coil and onto the surface of the capsule. The aim point was 2 mm from the mid-plane of the capsule. The instrument layout is shown in Fig 3.

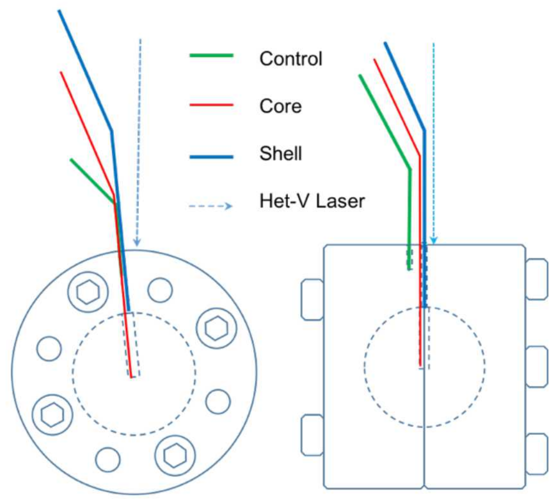

Fig. 3. Instrumentation layout showing the locations of the core, shell and control thermocouples. The dotted arrow shows the laser beam path.

Test Materials

We have previously reported the results of firings on a range of HMX based explosives employing HTPB as the binder[4]. In this work we describe firings on a high performance HMX based explosive that has a 1% nitrocellulose, 8% K10 binder. Such a high performance material would be expected to produce violent events and is therefore ideal to examine the ability of the ODTV experiment to capture such reactions.

Machining spherical samples

The explosive was supplied as a block of material. This was cut into sections using a remotely operated band saw and further cut into cubes using a custom made remotely operated fine-blade saw. The cubes were placed on the flat based vacuum chuck of a modified CNC milling machine that allowed capture of the swarf. A hemispherical shape was achieved by means of a flat-bottom cutter programmed to produce a slightly oversized shape. The final hemisphere was achieved using a profile cutter having the exact surface curvature. The sample was then turned upside down and placed in a purpose 3D printed hemispherical vacuum chuck and the process repeated. A single hole was drilled to the centre of the sample to allow the insertion of the thermocouple port. In this manner, samples were manufactured to exactly fit the capsule.

The copper sealing rings were filed to close to tolerance and tested such that they just allowed light to be observed when the two inner capsules halves were held tightly together.

The inner capsule having the thermocouple port was placed onto the sample such that it aligned with the drilled hole. The other halve with copper sealing was then placed on top and the capsule carefully inserted into one of the sealing rings. The capsules are precision manufactured and so care is required to align the two inner barrel halves exactly and normal to the outer ring. Once in place the other ring was pushed on and the whole pushed together. Eight bolts (four from either end) were used to hold the rings in place. The bolts are mild steel and would be expected to be preferentially heated by the induction mechanism.

Experimental Results

Fig. 4 shows the natural logarithm of the time to explosion versus the inverse set-point temperature. The quoted temperatures were measured at the centre of the specimen (at the bottom of the instrumentation well) as a long-term average of the steady temperature that was reached after the initial heating phase.

The digitized velocity-time records from the heterodyne velocimeter were further processed, by integration to produce displacement velocity relationships, and these are shown in Fig. 5. Following Wardell[5] and Maienschein[6] the radial velocity at a given displacement was used as a speculative measure of violence. In our method, the velocity at a displacement of 1.5 mm (designated $U_{1.5}$) was used, this being the estimated extent of capsule expansion for which the capsule had not yet de-confined. A higher velocity at 1.5 mm displacement would therefore be an indicator of a more violent response.

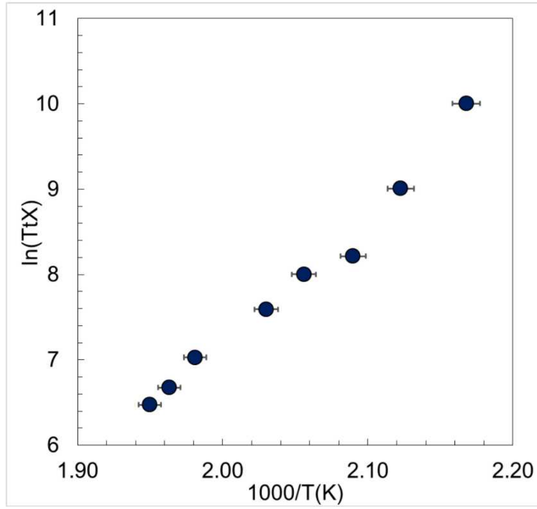

Fig. 4. Time to explosion data for ODTV firings. Horizontal error bars indicate the variation in 'steady' temperature, following the initial ramp.

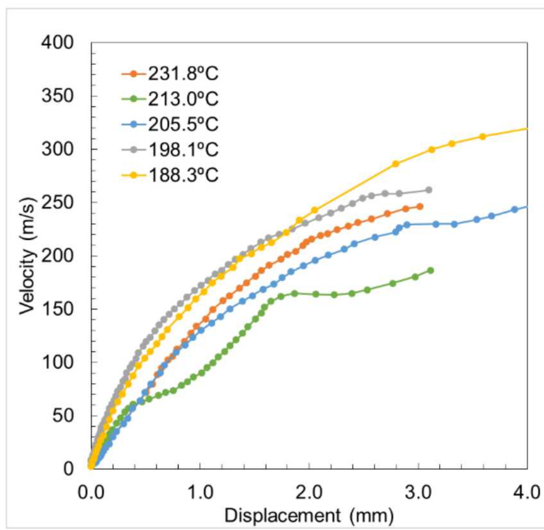

Fig. 5. Expansion velocity (measured by heterodyne velocimetry) as a function of radial displacement of the confinement.

Fragments of the capsule were recovered from the test arena and examined. It was found that in all experiments the compression ring fragments were easily distinguished from the remainder, having become separated at the thin web of material adjacent to the retaining bolt holes. These formed 16 similar fragments of approximately equal weight. The fragments from the inner shell, however, showed different degrees of damage depending on the set-point temperature, and the fragment counts are plotted in Fig. 6. $U_{1.5}$ velocites for the corresponding experiments are also included in this plot.

There is a relatively clear relationship between set-point temperature and degree of fragmentation of the inner shell, with a tendency to more numerous (and smaller) fragments at lower temperatures. Similarly, there is a general trend of increasing $U_{1.5}$ velocity with a decrease in temperature, although the data point at 231.8 ºC lies away from this trend. These relationships are depicted in Fig 6.

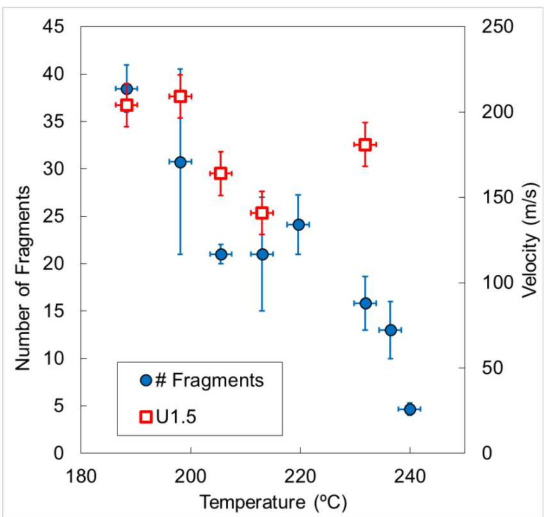

Fig. 6. Degree of fragmentation of the capsule as a function of temperature, and $U_{1.5}$ velocity.

The expansion velocity records generally the same smooth 'S' shaped profile shown in Fig, 7(a). In one experiment, at 231.8ºC, Fig 7(b), however, the indistinct and irregular record suggested poor alignment of the laser, and we speculate that in this experiment the Het-V was aimed too close to the joint at the mid-plane of the capsule and recorded the motion of several different surfaces during expansion.

Fig 7(c) shows the record for the experiment at 213.0ºC, in which the acceleration of the confinement was not steady, but was nevertheless smooth and continuous, and possibly indicates a non-steady development of pressure within the confinement during ignition.

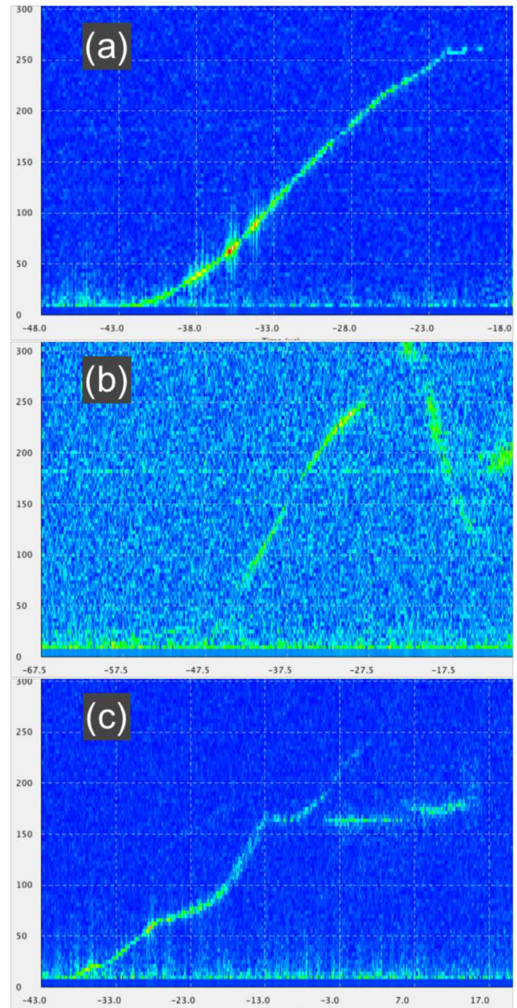

Fig. 7. Velocity spectragram profiles. Histories were typically of the profile (a); histories (b) and (c) are described in the text

Modelling ODTV

Our data has been used to validate a pressure-dependent PBX 9501 (95% HMX, 2.5% Estane, 2.5% BDNPA/F) Cook-Off model developed at Sandia National Laboratories[7]. The PBX 9501 decomposition mechanism is presented in Table 1 with more details provided in Ref. 7.

The 5-step global decomposition model includes drying, binder decomposition, and HMX decomposition. The products from the binder are allowed to interact with the explosive decomposition products. The kinetics use distributed activation energies, and some of the reactions are pressure dependent. Pressure dependence implies that there is at least one rate limiting gas-phase reaction. The pressure is implemented into the Cook-Off model by simply multiplying the rates by normalized pressure raised to a power. Vented systems are modeled with the pressure exponent set to 0. Sealed systems are modeled by specifying the pressure exponent as in reference 7.

The 9501 model was applied to the HMX formulations in the current work by accounting for the specific weight percent of HMX and assuming the binder is either reactive, or non-reactive. In the current work, we investigate HMX with three different compositions: 1) 95 wt.% HMX with 5 wt% HTPB binder, 2) 85 wt.% HMX with 15% binder, and 3) 91 wt.% HMX with 1 wt.% nitrocellulose (NC) and 8 wt.% K10 binder.

The PBX 9501 model[7] assumes the binder is reactive with kinetics based on a BFNPA/F nitroplasticizer. For the HMX with HTPB binders, we assume that the binders are either inert and nonreactive, or that they react with the PBX 9501 binder kinetics. For the HMX with 1 wt% nitrocellulose and 8 wt.% K10 binder, we assume that 0.5% of the binder reacts and the remaining binder is nonreactive or inert.

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Tables 2 and 3 give the initial compositions used in the model for the HMX/HTPB and HMX/NC/K10 formulations, respectively. We assume 0.5 wt.% moisture in all formulations. We assume the moisture is associated with the HMX in all compositions. The densities of the 95:5 and 85:15 HMX:HTPB formulation were 1320 kg/m^3 (72%TMD) and 1610 kg/m^3 (93%TMD), respectively. A temperature was imposed on each of these experiments by either immersing the apparatus in a heated bath, or by induction heating.

Table 1. PBX 9501 model[7].

$H_2O_s \rightarrow H_2O$ (1)
$NP \rightarrow NVR + 4\ NO_2$ (2)
$Estane + NO_2 \rightarrow 7.1\ Gas + 8.1\ C$ (3)
$HMX + NO_2 \rightarrow 11.5\ Gas + C$ (4)
$HMX \rightarrow 10\ Gas + 1.6\ C$ (5)

Table 2. Initial compositions for HMX/HTPB

HMX:HTPB	95:5	95:5	85:15	85:15
Binder reactivity	*r*	*nr*	*r*	*nr*
H_2O_s	0.5	0.5	0.5	0.5
HMX	94.5	94.5	84.5	84.5
binder-*r*	5	0	15	0
binder-*nr*	0	5	0	15

**r* and *nr* correspond to reactive and non-reactive

Table 3. Initial compositions for HMX/NC/K10

HMX/NC/K10	91/1/8
H_2O_s	0.5
HMX	90.5
binder-*r*	0.5
binder-*nr*	8.5

**r* and *nr* correspond to reactive and non-reactive

HMX with HTPB binders

Figure 8.A presents the measured (symbols) and predicted (lines) ignition times for the two HMX:HTPB formulations. The blue solid circles represent the HMX:HTPB 95:5 measured ignition times. The blue solid and dashed lines represent predictions wherein the binder is assumed to be nonreactive or reactive, respectively. The orange open circles represent the HMX:HTPB 85:15 measured ignition times. The solid and dashed orange lines represent the predictions whereing the binder is assumed to be nonreactive or reactive, respectively.

In Figure 8.A, the measured ignition time with a boundary temperature of 230 °C does not seem to follow the trend of the other two points. This could be caused by a leak in the system, or inaccurate estimates of the internal gas ullage or volume. For the HMX/HTPB runs, we assumed that there was no ullage. Additional ullage would cause the predicted ignition times to increase.

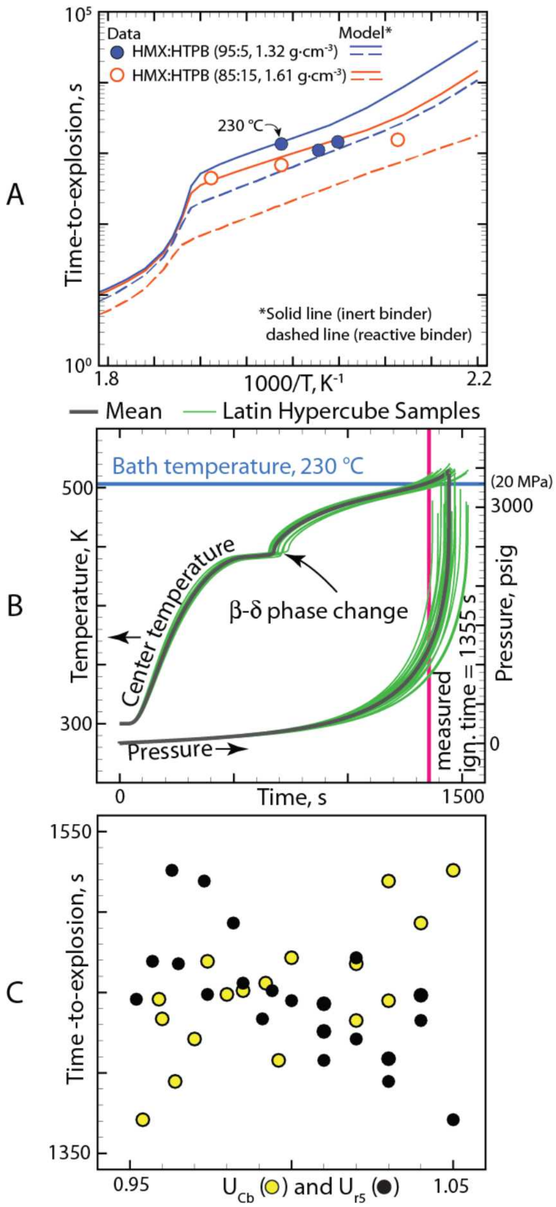

Fig. 8 A) Ignition times for HMX:HTPB runs, B) uncertainty in HMX/HTPB predicted temperature and pressure, and C) correlation of ignition time with specific heat (C_b) rate of reaction 5 (r_5).

Figure 8.B presents the uncertainty in the predicted center temperature and pressure. The uncertainty was determined by running 21 Latin Hypercube Sample (LHS) calculations as discussed in Ref. 5. Most of the dispersion in the predicted ignition time was attributed to specific heat and the rate of reaction 5. Figure 8.C shows the correlation between 21 LHS predictions of ignition time as a function of the uncertainy multipliers for the specific heat and the rate of reaction 5. As the rate of reaction 5 increases, the ignition time decreases. As the specific heat increases, the ignition time increases.

Figure 9.A presents a colored contour plot of the temperature within the 3-cm diameter 95:5 HMX:HTPB explosive at 1444 s when the imposed boundary temperature was 230 °C. Ignition occurs in the mid-radial position. A line plot of the temperature at ignition is given in Fig. 9.B.

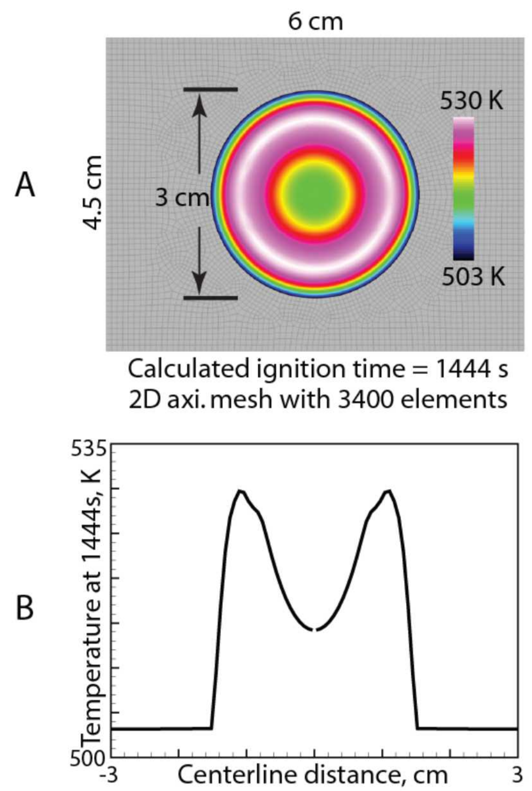

Fig. 9. Temperature A) contours and B) profile at ignition for 95:5 HMX:HTPB with 230 °C boundary temperature.

HMX with NC:K10 binder

More care was given to determing the gas ullage and binder reactivity for the HMX:NC:K10 simulations. We started by simulating the ignition time for ODTX experments performed at LLNL of an explosive with a similar composition 91:9 HMX:oil, polymer, nitrocellulose[8]. We determined the maximum ullage in the ODTX experiment to be about 0.07 cm^3. This estimate was based on available gas space around the copper gasket as well as volume gain by thermal expansion of the aluminum confinement.

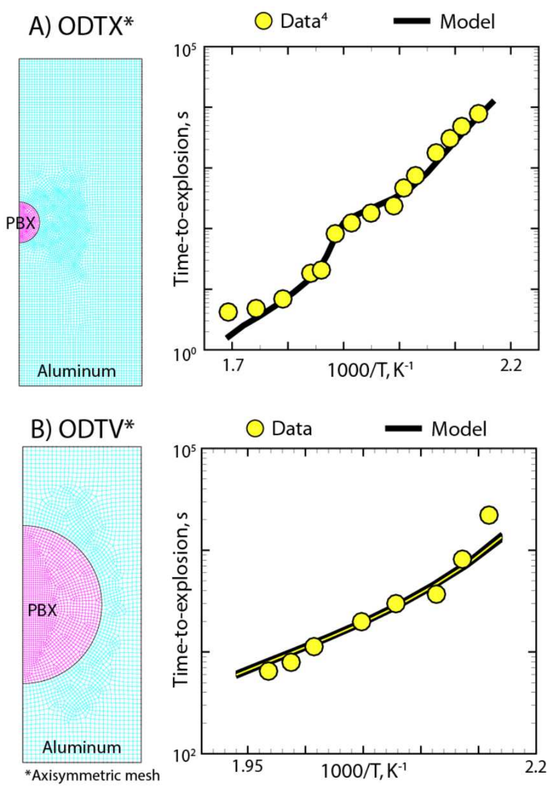

Fig. 10. Measured (circles) and predicted (line) ignition times for 91:9 HMX:binder in A) ODTX and B) ODTV apparatus. Mesh is shown on left.

We also estimated the ullage in the ODTV experiments. We believe there is no more than a 1 mm gap between the explosive and the confining aluminum giving approximately 3 cm^3 of gas space. We also believe there is approximately 1 cm^3 of gas space around the thermocouple wells.

The reactivity of the binder was determined by running several ODTX simulations with different amount of reactive binder. We chose 0.5% of the binder to be reactive as shown previously in Table 3.

Figure 10 shows a comparison of measured and predicted ignition times in the ODTX and ODTV experiments. The axisymmetric mesh for each experiment is also shown in Fig. 10. In the ODTX experiment, two anvils are preheated to a given temperature prior to confinement. At the start of the experiment, the two preheated anvils are brought together to deform a copper gasket to provide confinement. In these simulations, the anvils were assumed to be at the boundary temperature at the start of the experiment. In contrast, the ODTV experiments impose the boundary temperature at the edge of the room temperature aluminum at the start of the experiment.

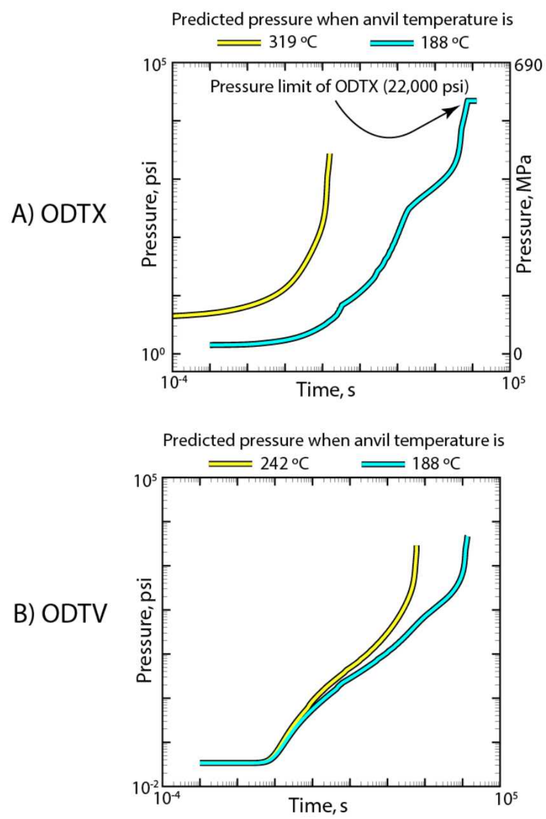

Fig. 11. Predicted pressure histories from A) ODTX and B) ODTV experiments.

The model predictions for the ODTV experiments has a yellow line outlined in what appears to be a black background. The yellow line is the mean predicted ignition time. The thicker black line represent 21 LHS sample runs using the same uncertainty as in Ref. 5. The dispersion in predicted ignition times was small. In other words, the uncertainy in the predictions is of the same order as the thickness of the prediction lines.

The model matches both the measured ODTX and the ODTV data. A few of the experimental points give longer ignition times. For example the hotter temperatures in the ODTX are slightly longer. In contrast, the cooler boundary temperature in the ODTV give longer ignition times. A likely explanation might be that the experiment leaked resulting in longer ignition times.

Figure 11 shows the predicted pressure for various temperature boundary conditions in both the ODTX and ODTV experimental apparatus. Predictions for both experiments show pressures approaching the limit of the confining aluminum. In fact, Fig. 5.A shows that the pressure limit of the ODTX apparatus was exceeded for the lower boundary temperature simulation.

Summary and Conclusions

We have developed a new Cook-Off apparatus that is similar to the ODTX experiment. However, the ODTV experiment can accommodate an order of magnitude larger explosive charge, and can be run without a dedicated load frame for each experiment. Our ODTV experimental apparatus is robust enough that we have significantly increased the working pressure of the vessel.

We have performed several experiments in the ODTV experiment. In the current work, we described three different HMX formulations that included 95:5 HMX:HTPB, 85:15 HMX:HTPB, and 91:1:9 HMX:NC:K10. We have applied a constant temperature boundary condition by using a constant temperature bath as well as using inductive heating. The HMX:NC:K10 experiments are the first to use inductive heating for our boundary temperatures. We believe this method of heating will be beneficial to investigate various heating conditions.

In previous work with HMX based explosives with variable amounts of HTPB binders, we have shown a strong correlation between the number of fragments and PDV velocity[2]. The more violent experiments occur at lower temperatures and produce higher PDV velocities and correspondingly more fragments. In the current paper, we show the same trend using a different formulation of HMX and binder. Our work shows that PDV velocity can be used as a violence metric for Cook-Off of HMX-based explosives with various quantities and types of binders.

We have simulated our Cook-Off experiments by modifying a previously developed model for PBX 9501. The model was modified by specifying different amounts of HMX and binder to match our explosives of interest. This model is pressure dependent and successfully simulated both the HMX:HTPB compositions as well as the HMX:NC:K10 compositions. For the HTPB simulations, we either assumed that the binder was nonreactive or reactive with the same binder kinetics as used in the PBX 9501 model. The measured ignition times were between the two predicted ignition times.

For the HMX:NC:K10 predictions, we tried to determine the excess gas volume more precisely and estimated the reactively of the binder by predicting ODTX experiments from Tarver and Tran's data[4]. Simulations were run in the ODTV experiment with the same reactivity parameters that were used for the ODTX simulations. The agreement between the experiments performed at separate laboratories was good.

We also performed several LHS sensitivity analysis and found the the model was most sensitive to the the specific heat and to the rate of the 5th reaction, which was the pressure dependent decomposition of HMX into equilibrium products.

The work discussed in the current paper shows the importance of confinement during Cook-Off of HMX based explosives. We have found that ignition time and pressurization depend strongly on the available volume for gas pressurization. We have also found that the reactivity of the binder in HMX based explosives is needed to accurately predict pressurization and eventual ignition.

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Acknowledgements

Sandia National Laboratories is a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.