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IGNITION DYNAMICS OF HIGH EXPLOSIVES

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Abstract

The laser ignition of the explosives HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine, $C_4H_8N_8O_8$), δ-phase HMX, PBX 9501 (95% HMX, 2.5% Estane, 2.5% BDNPA/BDNPF), TATB (1,3,5-triamino-2,4,6-trinitrobenzene, $C_6H_6N_6O_6$), and PBX 9502 (95% TATB, 5% Kel-F) and aged PBX 9502 has been conducted with the intent to compare the relative sensitivities of those explosives and to investigate the effect of beam profile, binder addition, and porosity. It has been found that there was little difference between a gaussian beam and a top hat profile on the laser ignition of HMX. We observe that the addition of binder in the amounts present in PBX 9501 resulted in longer ignition delays than that of HMX. In contrast to HMX, the addition of binder to TATB in PBX 9502 shows no measurable effect. Porosity effects were considered by comparing the ignition of granular HMX and pressed HMX pellets. Porosity appears to increase ignition delay due to an increased effective absorption scale and increased convective heat loss. This porosity effect also resulted in longer ignition delays for δ-phase HMX than for β-phase HMX. In order to simulate ignition in voids or cracks, the standard ignition experiment was modified to include a NaCl window placed at variable distances above the sample surface. When ignition experiments were performed at 29 W/cm^2 and 38 W/cm^2 a critical gap distance was observed of 6 ± 0.4 mm below which ignition was severely inhibited. This result underscores the importance of gas phase processes in ignition and illustrates that conditions can exist where simple ignition criteria such as surface temperature is inadequate.

Introduction

The two most common parameters measured in laser ignition experiments are the threshold ignition energy and the ignition delay. Threshold ignition energy is the minimal amount of incident radiative energy necessary to cause ignition of the sample under a given set of conditions. Ignition delay is the time from when the sample first begins to be irradiated until the sample ignites. Both of these measurements by definition depend upon the ability to accurately characterize the ignition event. But in the literature there are discrepancies in the practical definition of ignition and the best method to determine when ignition has occurred.

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The most widely used experimental method of determining when ignition has occurred is by using a photodiode and defining ignition as the appearance of "first light" from the HE sample. However, the light emission criterion used in experimental analysis cannot be applied to theoretical analysis as the ignition criterion because the detailed values of the physical parameters of the reaction kinetics are not known well enough[4]. Additionally, Boggs[1] states that it is not until ignition that equilibrium energy release may be assumed. Where ignition is defined as the point at which combustion may be sustained upon interruption of the external flux, known as go/no-go ignition and not first light or gasification.

Dimitriou[5] measured the surface emission in his experiments on the ignition of Ti+C and Zr+C. He used a pyrometer to view the thermal surface emission of the sample pellet and thus non-invasively determined the surface temperature during the ignition event. His resulting surface temperature trace had a steady rise in surface temperature up to the ignition event at which there was an abrupt change in slope. He defined ignition time as the temporal difference between the opening of the laser shutter and the cusp on the surface temperature rise right before the change in slope, although, other various ignition criteria could be assumed.

Many models define ignition in terms of a critical surface temperature. However, the majority of experiments have been conducted only with photodiodes without observation of the surface temperature. Consequently, there is a utility in monitoring "first light" as well as surface temperature in the same experiment to resolve these issues.

In measuring the ignition delay of a sample and the ignition threshold energy, the "go/no-go" ignition characterization has been traditionally used. When threshold ignition energy is measured in the experiments, a constant flux of radiative energy is incident upon the sample surface and then the irradiation of the sample is halted after sequentially varying time intervals. Ignition is said to have occurred when the sample remains ignited after removal of the external radiative energy. Unfortunately, this method for determining ignition is dependent upon the rate of de-radiation.

De Luca[6] states that extinction is assumed to occur when the instantaneous thermal state of the condensed phase can no longer be matched to the allowed values of the surface temperature and thermal gradient at the condensed side of the burning surface. This adverse condition may be induced by a sufficiently fast removal of an incident radiative source. Dik[7] concludes that a square pulse is the most unfavorable for "stable" ignition, which may be defined as a steady state relationship between mass conversion and pressure and an equilibrium energy release with gasification[1]. The sharp cutoff of the flux in a square pulse imposes rigorous conditions on the rate of rearrangement of the thermal field in the condensed phase. And so the position of the go/no-go boundary may be dependent upon differences in the rate of de-radiation between different experimental conditions. In our current experiments, the laser was allowed to run through completion of the data acquisition and go/no-go was not measured.

Summerfield[9] argued that ignition may be sensitive to the "particular intensity profile of the 'focal footprint' of the beam striking the propellant surface". This means that ignition results may vary between experiments due to different laser beam profiles. Experimenters have tried to reduce this problem by creating a uniform beam profile. Most have simply expanded a gaussian beam and then cutoff the outer 10% or so leaving an more nearly uniform profile with well-defined boundaries. Some have gone to further lengths by using a cylindrical beam integrator and other beam integrators[9,10]. Ideally the beam integrator should produce a one dimensional, top hat profile thus eliminating any problems with variations in energy intensity incident upon the HE surface. Due to the two different methods used for approximating a spatially uniform irradiance, expanding a gaussian beam and using beam integration, there is a need for a quantitative comparison between the two methods to determine what, if any, effect the different profiles have on ignition characteristics.

In this work, various aspects of laser ignition experiments and the characterization of certain properties of the explosives HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine, $C_4H_8N_8O_8$), PBX 9501 (95% HMX, 2.5% Estane, 2.5% BDNPA/BDNPF), TATB (1,3,5-triamino-2,4,6-trinitrobenzene, $C_6H_6N_6O_6$), and PBX 9502 (95% TATB, 5% Kel-F) and aged PBX 9502 are investigated. The effect of beam profile on the laser ignition results is investigated in this paper by comparing a 50% gaussian profile to a uniform, top-hat profile produced by a beam integrator. The use of a high-speed camera is implemented to capture gas phase ignition processes. Using the measured ignition delays, the relative sensitivities of the above mentioned explosives including δ-phase HMX are compared. The effect of binder addition on the laser ignition of HMX and TATB, the effect of aging upon PBX 9502 and the effect of porosity on the laser ignition of HMX are also investigated. Voids or cracks in a damaged HE can potentially be on the same scale as the gas phase flame of a burning explosive. Consequently, the ignition process may be affected. The standard ignition experiment has been modified to begin to study this issue. Specifically, a salt window has been placed at various distances above the pellet

surface. The gap above the sample surface created by the salt window has been intended to simulate macroscale incongruities present within a damaged sample or voids in between grain boundaries which may interrupt steady deflagration processes.

Experimental Setup and Procedure

In this work we studied the laser ignition of various HE of interest, HMX (Lot 920-32, density 1.78 g/cm^3), PBX 9501 (Lot HOL 89C 730-010, density 1.80 g/cm^3), TATB (Lot 12-11-81-0921-264, density 1.80 g/cm^3), PBX 9502 (Lot 890-022, density 1.80 g/cm^3) and aged PBX 9502 (lot 890-018). Most samples were in pressed pellet form with the exception of powdered HMX used for the porosity experiments and the δ-phase HMX. The powder HMX was the same stock powder used to create the pressed pellets. The δ-phase HMX was created by heating a pressed β-phase HMX pellet in an oven at 190°C for one hour. Heating expanded the pellets by approximately 12.8% by volume. The diagnostics included a photodiode, InSb IR surface detector, thermocouple, Molectron pyrometer, power meter, video camera, and high-speed video camera. Oscilloscopes were used to record the signals from all the experimental diagnostics and a PC was used to acquire the data from the oscilloscopes. The laser was a 9 - 11 μm variable wavelength, 180 Watt, CO_2 laser (Edinburgh Instruments Ltd. model PL-6). For all the experiments the laser was tuned to the 10P20 line at 10.6 μm. The beam profile of the laser was recorded using a laser beam analyzer (Spiricon model #300PC) and the spectral wavelength was determined using a CO_2 Spectrum Analyzer. The initial beam from the laser was TEM_{00}, but for some experiments a beam integrator (SPAWR model SI-061) was used to produce a top hat profile.

The photodiode (Thorlabs model DET110) detected the emission of visible light (spectral response 300 to 1100 nm) and had a response time of 10 nanoseconds. The photodiode therefore indicated the time of first light emission after the laser is turned on.

A single element InSb (Indium Antimony) infrared detector (EG&G Judson J10D-m205-R04m-60) with a peak wavelength of 5 μm was focused on the surface with a collection lens. This detector provided a non-invasive method for measuring the relative thermal emission of the HE sample surface.

A 25 μm S-type thermocouple placed on the sample surface provided a means of measuring the surface temperature during the ignition event. The thermocouple was stretched across the sample to ensure contact.

The laser power was measured before each experiment with a power meter (Laser Probe Inc. model RkT-1500W-C) which was placed at the very end of the laser path at the same position as the pellet. Before each power measurement, a black metal mask with a 1 cm diameter hole (pellet diameter is 1 cm) was placed over the power meter and the beam was centered through the hole. Thus, the power meter measurements accounted for any non-ideal absorption by mirrors and other optical devices and recorded the total power incident upon the pellet surface.

During the experiment a fast response Molectron pyrometer was used to dynamically measure the laser power. The signal from the Molectron also gave an accurate account of the rise and fall times of the laser output. As seen in figure 1, a NaCl window was used to pull off approximately 8% of the main beam and was used to direct that secondary beam into the Molectron.

The laser ignition experiments were visually recorded with a standard video camera at a frame speed of 60 Hz. A high-speed camera (Hadland Photonics Model #1568) was also used to view the ignition in the gas phase. The Hadland was placed level with the pellet and focused horizontally above the pellet surface. It took a sequence of eight pictures with varied delay separation and was triggered by the photodiode signal.

The ignition in a gap experiments used a slightly different setup as seen in figure 2. A crack or void in a piece of HE has been simulated by creating a gap above the surface of a pellet of HMX using a NaCl window. The incident CO_2 laser beam was transmitted through the salt window onto the pellet surface. Ignition was determined to have occurred if there was a visible flame present. The width of the gap was gradually decreased until ignition does not occur at a given laser flux. The tested laser irradiances were 29 W/cm^2 and 38 W/cm^2.

Results and Discussion

The experimental raw data included voltages as recorded by the oscilloscope of the InSb IR detector, the S-Type thermocouple, the photodiode, and the Molectron. The InSb traces are exemplified by figure 3 which shows steady heating up to an abrupt jump in the emission, which is taken to be the gas phase ignition. Even though the IR detector was focused onto the surface, it still had a large enough field of view to partially detect the gas phase. Before ignition, the detector picks up primarily surface emission because of the strong emission of the surface as compared to the much smaller gas phase emission. Once the gas phase ignites, however, its emission is significantly stronger than the surface due to the elevated temperatures of the gas phase and particulates such as carbon. Therefore surface temperatures as derived from IR emission measurements cannot be relied upon once ignition has occurred.

Figure 3 also shows the respective thermocouple trace in addition to the InSb trace. The general trend of the two different measurements compare well up to the point of ignition at which the thermocouple continues at a constant temperature and the InSb jumps. This apparent separation of the thermocouple and InSb trace is most likely due to the strong emission of the gas phase at ignition as mentioned previously. Therefore the InSb would jump, while the thermocouple, which is still on the surface possibly imbedded in the melt layer, would not immediately "see" the gas phase temperature rise.

In the experiments performed in this study, two methods for the characterization of the ignition event were implemented. The photodiode, viewing the gas phase, was used to detect the initial appearance of luminous emission at ignition and the InSb IR detector, viewing both the surface and gas phase was used to detect the jump in emission at ignition. These experiments were conducted at atmospheric pressures and therefore had only a strong luminous spike at ignition. From figure 4, it is seen that there is virtually no difference between the two methods of ignition characterization for HMX and TATB. This suggests a strong correlation between the runaway of gas phase exothermic reactions and gas phase luminous emission. Therefore it can be concluded that either method is sufficient for ignition delay measurements at atmospheric pressures.

The effect of beam profile on laser ignition was investigated in this study by using a clipped gaussian beam profile (50% of the beam) and a uniform, top-hat profile created from a Spawr beam integrator to ignite HMX and TATB. The results, seen in figure 5, clearly display that at the powers measured and at atmospheric pressure there is no difference in the ignition delay of the two different beam profiles. This suggests that either a clipped gaussian profile of 50% approximates a uniform profile adequetly so that there is no difference in the ignition for HMX and at these conditions. Furthermore, on some experiments the beam had drifted out of the TEM_{00} the profile represented more of a donut or TEM_{01} with the majority of the energy concentrated around the edges. Yet, even with these erratic profiles there was little difference in the ignition delays of the explosives. There is also some mixing of the particles ejected from all portions of the surface. And since HMX and TATB ignite in the gas phase, this mixing may also function to reduce the effect of thermal gradients present on the surface of the condensed phase. Therefore it is believed that the laser ignition of HMX and TATB is not strongly dependent upon beam profile, within reason.

One of the characteristics exposed by laser ignition is that of the sensitivity of explosives to thermal stimulus[11]. The effect of binder addition on the thermal sensitivity performance of explosives is of great importance to various applications. This effect can be realized by comparing the ignition delays of the pure explosive with that of the explosive/binder mix. Figure 6 compares the ignition delay of HMX and TATB based explosives. In comparing pristine PBX 9501 and HMX it appears that the addition of 5% Estane binder in PBX 9501 significantly increased the ignition delay over that of pure HMX. In comparing PBX 9502 and TATB there is virtually no difference in the ignition delays at the radiant fluxes measured. This would suggest that the addition of inert Estane binder decreased the sensitivity of HMX, while the addition of inert Kel-F binder to TATB had little effect. This difference in results between HMX and TATB may stem from the difference in the decomposition mechanisms of the two explosives. When HMX is ignited with a laser it first starts out in the condensed phase, then a thin melt layer is formed which then proceeds to gasify. TATB also starts out in the condensed phase but essentially sublimates directly to the gas phase where, like HMX, ignition occurs. When there are particles of Estane binder intermixed with those of HMX as in PBX 9501 and the material is heated, the melt layer formed should consist of both the binder and the HMX. If the liquid layer is assumed to be homogeneous, then when the laser irradiates the liquid layer, a portion of that energy would be consumed by the inert binder. This effect may possibly be similar to that of boiling point elevation of volatile/non-volatile mixtures. Along this line of reasoning, the addition of binder to TATB in PBX 9502 would not effect the ignition delay because, unlike HMX, TATB does not transition through a liquid layer, but instead sublimates from the condensed phase directly to the gas phase. However, the specific details of the ignition mechanisms of TATB and HMX and their binder interactions are not known well enough to make any concrete explanations.

There is also a concern about increased sensitivity of aged explosives. Figure 6 shows the results of the laser ignition of PBX 9502 and aged, 11 year old PBX 9502. In comparing the relative ignition delays, there appears to be no difference between the samples. This indicates that there is not a measurable sensitivity change in PBX 9502 over the lifetime of the samples tested. It should be noted that it is difficult to make accurate characterization of the TATB data due to the loose data grouping. This apparent lack of reproducibility is due in large part to the severe burning instability of TATB at atmospheric pressure. Further, pressurized experiments, are needed to more fully understand the ignition behavior of TATB.

All the pellets used in the experiments discussed in the paper were pressed to relatively high pressures and thus had little porosity. To determine if

pressures and thus had little porosity. To determine if there was any effect of porosity upon laser ignition experiments, a standard HMX pellet pressed to 95% TMD was compared with loose, course HMX powder. The powder was placed in a quartz holder of the same inner dimensions as the pellet. As seen in figure 7, there is a difference between the delays of the powder versus that of the pellet. The effect of porosity is to increase the ignition delay of HMX within the flux ranges tested. An effect of increased porosity is an increased effective absorption depth of the laser light and increased convective heat loss. As the particle density is reduced more space is introduced in between crystals of HMX. These voids, when present on the surface, allow the laser light to penetrate to a greater depth than for that of higher densities. Thus the effective absorption depth is increased. The increased size of the voids also allows greater air flow around the particles which results in an increased convective heat loss. Therefore for the powder, the laser energy was spread over a much larger volume than that of the pellet. This larger heated volume, with the flux being equal, could have resulted in a slower heating rate for the powder than for the pellet, which in turn might have resulted in the longer ignition delays.

More supporting evidence for this supposed effect of porosity comes from the laser ignition of δ-phase HMX. HMX in the δ-phase has long been established by drop tests and friction tests to be much more sensitive than β-phase HMX. Therefore one would expect that the laser ignition delays of δ-phase HMX pellets would be shorter than those of β-phase HMX. However, as seen in figure 7, δ-phase HMX delays are actually longer than those of β-phase HMX. The longer delays of δ-phase HMX may be due to the effect of the decreased density and increased porosity upon heating. However, to fully confirm this effect more experiments are being considered to compare equal porosity pellets of β-phase and δ-phase HMX.

One of the other diagnostics used in these laser ignition experiments was that of a high-speed camera to capture the ignition event. Boggs[1] described the ignition sequence of HMX as starting in the condensed phase and then gasifying. Once the HMX has gasified it forms a plume traveling off the surface of the pellet. As the molecules travel up this plume they exothermically react and at some distance above the surface, they finally ignite and then the initial flame spreads throughout the gas plume and "snaps-back" towards the surface. Figure 8 shows this "snap-back" event as caught by the high-speed camera. Ignition clearly occurs well off the surface and then the flame expands throughout the rest of the gas plume and towards the surface.

In performing the laser ignition experiments with a gap, it was found that at a constant flux there was a critical gap width of 6mm ± 0.4 mm. That critical gap width was the same for the two flux levels tested. When the gap was too small only a gas plume of pyrolysis gases was present which was deflected horizontally in all directions by the salt window. When the gap was large enough to allow ignition to occur (see figure 2) the plume and resultant flame was also deflected horizontally by the window. The difference between a critical gap and sub-critical gap is the distance in which the gas is allowed to travel, concentrated within the plume, without being disturbed. It may be theorized that a critical gap distance should exist below which ignition should not occur. The ignition process consists of thermal heating of the condensed phase to the gas phase, the rise off the surface of the gas phase energetic particles and finally ignition some distance off the surface within the gas phase. Ignition occurs off the surface because when the energetic material gasifies there is some inherent chemical reaction delay before the exothermic processes runaway and ignition occurs. If a barrier (the salt window) is placed within that gas plume at such a distance in which the gaseous particle reactions do not have either the time to go to completion or the necessary thermal energy to runaway before being disturbed by the impinging flow, ignition should not occur.

The fact that the same critical gap distance is not sensitive to the two different fluxes is somewhat puzzling, however. Without a gap, the ignition delays differ by a factor of 1.7 for the flux levels tested. More experimental work needs to be done to confirm a change in the gap distance with increasing laser flux. The fact that the critical gap distances for 29 W/cm^2 and 38 W/cm^2 were found to be the same may be explained by realizing that the two fluxes tested are relatively close although ignition times differ significantly. If experiments were to be conducted at higher fluxes, perhaps a change in the gap distance would be observed. However, the significance of these results remain clear and suggest that a simple thermal model of the ignition process is inadequate in certain circumstances. The results also underscore the importance of including gas phase processes in any modeling scenario of HMX.

Summary

Experimental methods using a photodiode and IR detector to determine time of ignition and top-hat and gaussian beam profiles were compared. A comparison of the various properties of HMX and TATB based explosives was also conducted with additional investigations into the effects of binder, porosity, and gaps above the sample surface on the laser

ignition of these explosives. It was found that there were no measurable differences between ignition delays measured with a photodiode and with an IR detector. There were significant deviations of the InSb IR detector from the thermocouple after ignition occurred. Top-hat or uniform beam profiles and 50% gaussian beam profiles were shown to have an insignificant effect upon ignition delay at atmospheric pressure. It is believed that the different profiles do not affect laser ignition experiments because HMX ignition and TATB ignition is not extremely sensitive to beam profile.

Binder added to HMX in the amounts present in PBX 9501 does have a significant effect upon the laser ignition results. That effect is to cause a longer ignition delay at comparable laser irradiances. The addition of binder to TATB in PBX 9502 does not have any observable effect. The difference in the effects of binder on the two explosives is believed to stem from differences in the ignition mechanisms of the two explosives. Aged PBX 9502 was also compared with pristine PBX 9502 and it was determined that aging had no effect upon the ignition delay for the conditions considered. The effect of porosity on laser ignition was investigated by comparing the ignition delays of loose, powder HMX and pressed HMX. It was found that porosity significantly increased the ignition delay of HMX and is believed to be due to the larger absorption depth caused by a decreased density and increased convective loss. This porosity effect may have also been a dominant factor in the ignition of δ-phase HMX, which should have had a shorter ignition delay than β-phase HMX, but instead had a longer ignition delay possibly due to the porosity. Future work that compares equal density samples of β-phase HMX and δ-phase HMX is necessary to confirm the dominance of this porosity effect.

The gas phase's effect on the laser ignition of explosives was investigated by using a high speed camera to capture the ignition in the gas phase and the "snap-back" of the flame to the surface. The gas phase's effect was also investigated in a novel experimental setup which uses a NaCl window to create a variable gap above the surface of the HE sample. It was found that there existed a critical gap distance of 6 mm ± 0.4 mm at irradiances of 29 W/cm^2 and 38 W/cm^2 below which ignition would not occur. More experiments at higher irradiances are necessary to confirm an dependence of the gap distance on irradiance, however the results underscore the importance of gas phase processes on the laser ignition of explosives.

Acknowledgements

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Figures

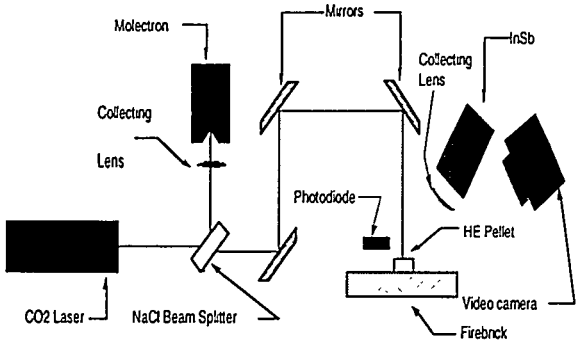
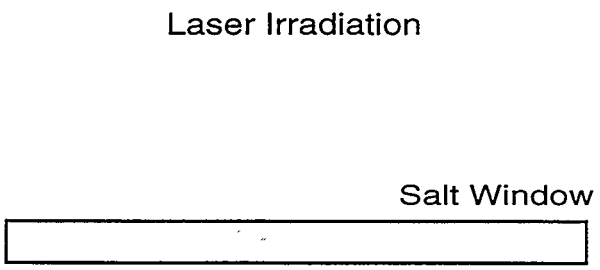
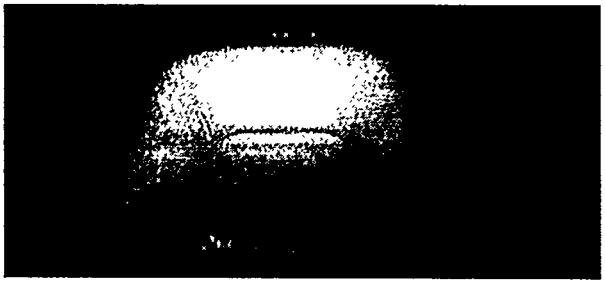


Figure 1: Experimental setup for the standard laser ignition experiment. Diagnostics shown include Molectron, InSb IR detector and photodiode.



(a)



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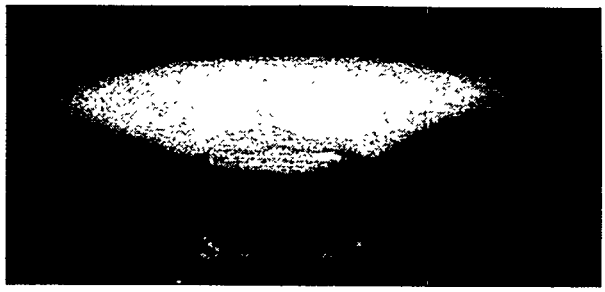


(c)

Figure 2: (a) Schematic of experimental setup using a salt window to create a variable gap above the surface of an HMX pellet.
(b), (c) Video camera pictures of an ignited HMX pellet within a gap.

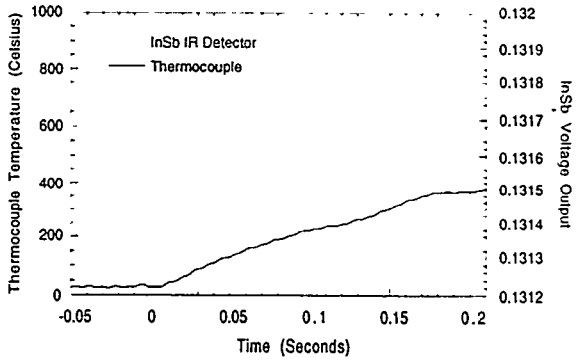


Figure 3: An example thermocouple and InSb IR detector trace. Laser was turned on at time 0 and ignition was assumed to occur at the jump in the InSb trace at approximately 0.186 seconds. Explosive was HMX which was exposed to an irradiance of 36 W/cm^2.

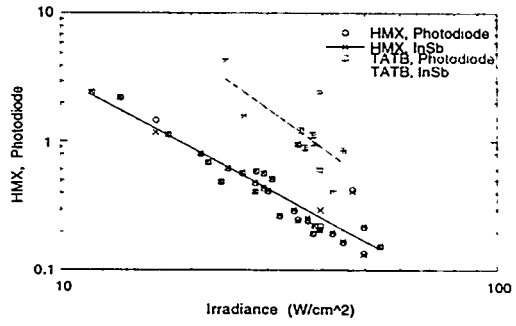


Figure 4: A comparison of the ignition delays of HMX and TATB as determined by a photodiode and an InSb IR detector. There is no observable difference in the two detection methods.

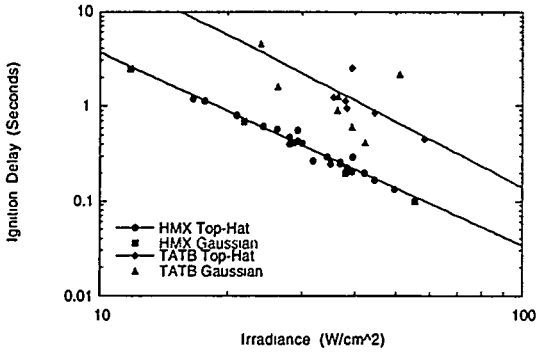


Figure 5: A comparison of the ignition delays of HMX and TATB with both a gaussian laser beam profile and a top hat beam profile. There appears to be no significant difference between the two tested beam types.

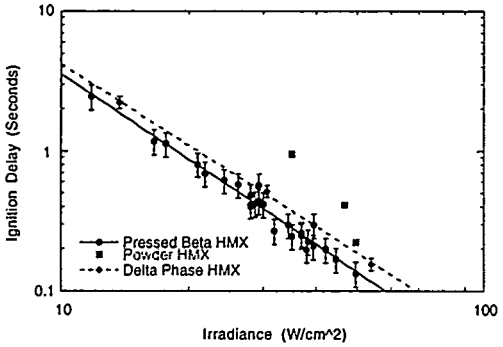


Figure 6: Ignition delays of HMX, PBX 9501, TATB, PBX 9502, and aged PBX 9502. The binder addition PBX 9501 has a significant effect while that of PBX 9502 has little effect. TATB ignition has only limited reproducibility due to its burning instability at atmospheric pressure. Aging has no observable effect upon PBX 9502 under these conditions.

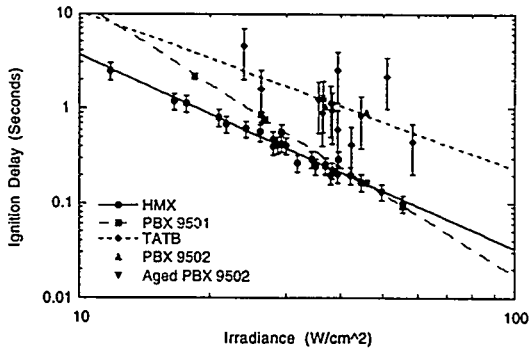
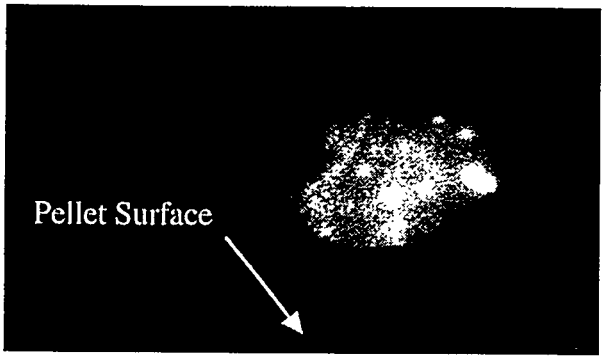
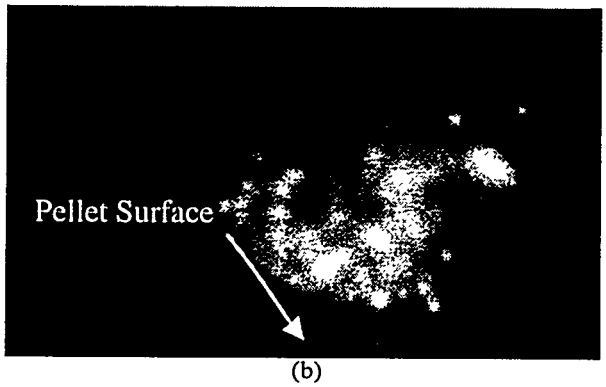


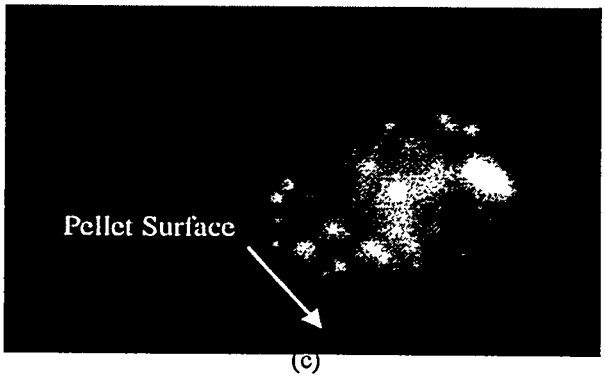
Figure 7: The effect of porosity is realized by comparing pressed HMX with powder HMX. Increased porosity causes the ignition delay to lengthen. δ-phase HMX, which is significantly more sensitive than β-phase HMX to impact, shows an increase in ignition delay. This may be due to a decrease in density. upon heating from β-phase to δ-phase more than the polymorph change.



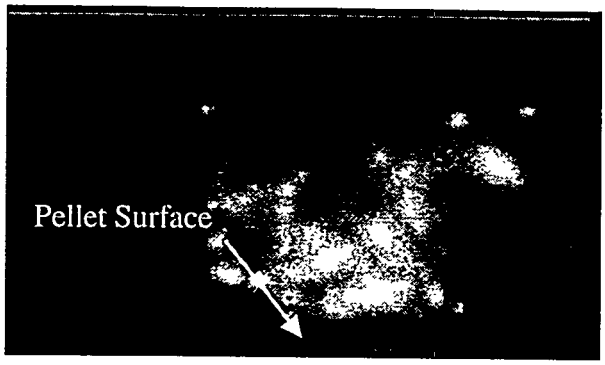
(a)



(b)



(c)



(d)

Figure 8: High speed camera photographs of the ignition of HMX at (a) 80 nsec. after first light (b) 0.300080 msec., (c) 0.600080 msec., (d) 0.900080 msec. Ignition off the sample surface in the gas phase is clearly shown, as is the subsequent spherical expansion and "snap-back" of the flame to the surface.