Laser Ignitibility of Insensitive Secondary Explosive 1,1-Diamino-2,2-dinitroethene (FOX-7)

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Abstract: An experimental investigation into laser ignitibility of insensitive secondary explosives, 1,1-Diamino-2,2-dinitroethene (FOX-7) has been carried out, using a diode laser of continuous wave at the laser wavelength of 974 nm. The direct optical ignition of an insensitive explosive will add more safety features to insensitive munitions (IM) or explosive devices. In this study, effects of laser parameters on the ignitibility were analysed in terms of laser ignition threshold, the times to initiate the ignition and full combustion, and burning sustainability. The results have shown that carbon black (CB) as an optical sensitizer is compatible with FOX-7, and significantly enhances laser ignitibility of the explosive when a small amount of CB is uniformly doped in FOX-7. The delay times for ignition and subsequent development of sustainable burning of the material are mainly determined by ignition laser power although the other laser parameters have effects. The minimum laser power required to ignite the optically sensitized FOX-7 was found below 10 W and a fast ignition was initiated in as short as 70 μs by a laser power of 40 W. Also the effect of the mixture uniformity of FOX-7/CB on laser ignition performance was evaluated in this study.

Keywords: Insensitive explosive, laser ignition, optical sensitizer, FOX-7, Carbon black

1 Introduction

For the development of explosive driven devices and systems, safety requirement in their entire life cycle from manufacturing and storage to the operation is the most important issue. Accidental or premature initiation of explosives causes disastrous consequences to both life and property due to a wide range of operational environments that have potential stimuli such as electromagnetic interference (EMI) from lightening or a radar system, cook-off of sensitive explosives in a hot environment, and impact in moving carriers, etc. For example, a conventional, currently used initiator usually contains metal-wires buried in sensitive primary explosives and/or pyrotechnics. Such a device suffers from corrosion & surface degradation of the metal components, impossible maintenance, external EMI, and high sensitivity of primary explosive to external stimuli. However, these advert drawbacks can be well overcome by directly igniting intrinsically insensitive secondary explosives using optical power from a laser, where undesirable primary explosives and metal components are not present. The use of laser for initiation also allows optical shutters being applied to provide extra safety controls.

Research in ignition/initiation of energetic materials by laser radiation has been undertaken world-widely for the last decades [1-10]. This has not only included experimental investigation into ignition behaviours but also included theoretical studies and modelling to understand the underlying mechanisms of laser ignition [4,7,8,11,12]. Some works [13-15] have studied the interaction of laser radiation with energetic material and shown that laser-induced hot spots play an important role in ignition mechanism. These researches have indicated the effects on hot spot formation and its ignition of energetic materials which are caused by material electromagnetic absorption properties and microstructural features (e.g. defect inclusions, void distribution & spacing, grain morphology) in addition to laser parameters (e.g. wavelength, etc.). Also the effects of laser beam dimension and laser power density on laser ignition were studied even back in 80’ [16,17]. Various materials have been used in these studies, such as pyrotechnics (e.g. gun powder), primary explosive (e.g. lead azide) and secondary explosives (e.g. HMX, PETN, RDX), and they can indeed be ignited or initiated by lasers in the UV/VIS wavelength range due to their UV/VIS absorption bands. For the ignition by lasers at red and near infrared (NIR) wavelengths, the materials were optically sensitized by mixing with light absorbing materials, mostly carbon black. This sensitisation has made diode laser the most suitable laser source, because of its commercial availability at NIR, miniature size, low cost, and ease of system integration.

As a relatively new secondary explosive, 1,1-Diamino-2,2-dinitroethene (FOX-7) has not be used in the study of laser ignition although it is of much lower sensitivity than those commonly used and particularly considered as a low vulnerability ammunition (LOVA) materials [18-21]. Some of its safety properties are shown in Table 1 in comparison with RDX and HMX. It is indicative of much lower sensitivities to friction and impact for FOX-7 than the other two, while they all have similar ignition temperatures. Such insensitivities are specifically suitable for the applications where the explosives are carried in a fast moving body and undergo accelerating (decelerating) forces. Therefore, using FOX-7 as a suitable explosive candidate allows Insensitive Munitions (IM) or devices to be achieved. However, insensitive explosives, whilst being characteristically less sensitive to accidental stimulus, are also less sensitive to conventional ignition mechanisms. In many cases their ignition requires the addition of more sensitive primary explosives which have an adverse effect on the sensitivity of the munitions or devices. The use of laser power for direct ignition of insensitive explosives will eliminate the adverse use of sensitive materials while keeping its immunity to potential stimuli.

Table 1 Comparison of material properties

	FOX-7	RDX	HMX
Ignition temperature (°C)	226	223	250
Impact sensitivity ($h_{50\%}$, cm)	126	80	56
Friction sensitivity (N)	>350	220	175
Drop weight (cm)	70	38	26

Since the introduction of insensitive energetic material FOX-7, very little has been published or reported on its ignitability using laser radiation. In this study laser ignitibility of FOX-7, doped with carbon black for optical sensitization, was experimentally investigated using optical power at 974 nm from a diode laser of continuous wave. The essential ignition performance in terms of ignition maps (ignition delay and full burn delay versus applied laser power), ignition threshold laser power, and the combustion sustainability were studied when various laser parameters were applied.

2 Experimental

2.1 Sample preparation and characterisation

FOX-7 used in this study is a yellow crystalline material in its powder form and has the chemical formula, $C_2H_4N_4O_4$, and structure as shown in Figure 1. In order to optically sensitise FOX-7, its powder (~1g) was mixed with a small amount of carbon black (CB) in 5 wt% (by mass) content. As commonly used in laser ignition of secondary explosives [5,7], a small amount of CB (1% to 5%) could increase the optical absorption of explosive mixture to ~90% which is optically sensitive enough to the laser ignition. The concentration used in this work may provide high sensitization without affecting the material properties and performance. This mixing was simply done by adding 95 wt% FOX-7 and 5 wt% CB together in a glass vial and tumble mixing (rolling the vial) by hand for 15 minutes. The sample of FOX-7/CB mixture was examined by Scanning Electron Microscopy (SEM) and the images in x100 and x1000 magnifications are shown in Figure 2. The FOX-7 particles in the images appear to be comprised of thin crystalline flakes of only a couple of microns thickness and are almost rectangular in nature. The crystals measured approximately 30 - 40 µm in width and up to 70 - 80 µm in length. The CB particles appear to be of relatively uniform size and distribution, measuring approximately 5-10 µm round. Optical microscopy was also used to examine the sample mixture and a photo-image is shown in Figure 3. It can be seen that this tumble mixing technique produced a reasonably uniform mixture. However, inhomogeneity in the distribution of carbon particles and the density in the sample material did exist, as indicated in the figure where large ‘clumps’ of either CB or the explosive are present. This may affect the ignition characteristics of the material, particularly when a relatively small beam was used.

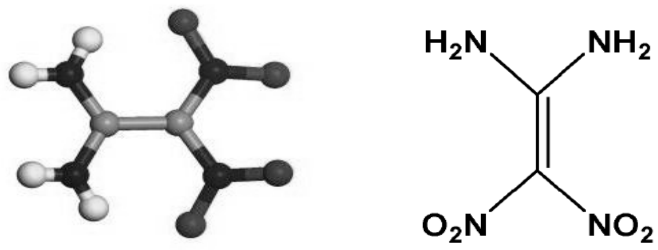


Figure 1 FOX-7 molecular structure

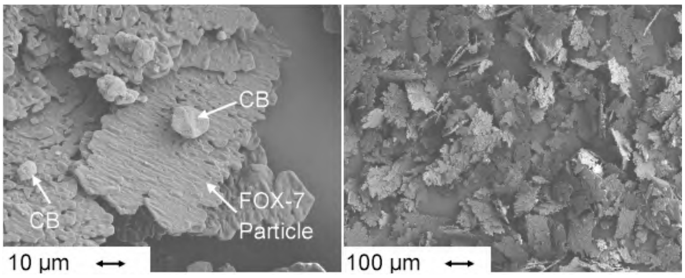


Figure 2 SEM images of FOX-7/CB mixture sample

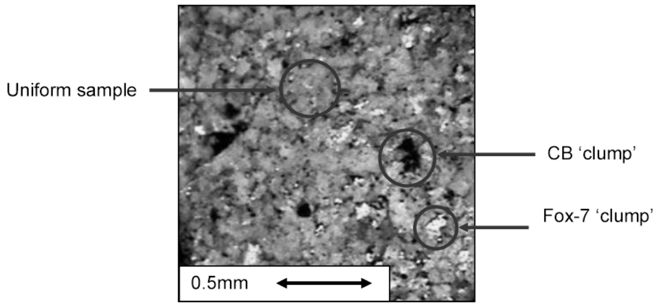
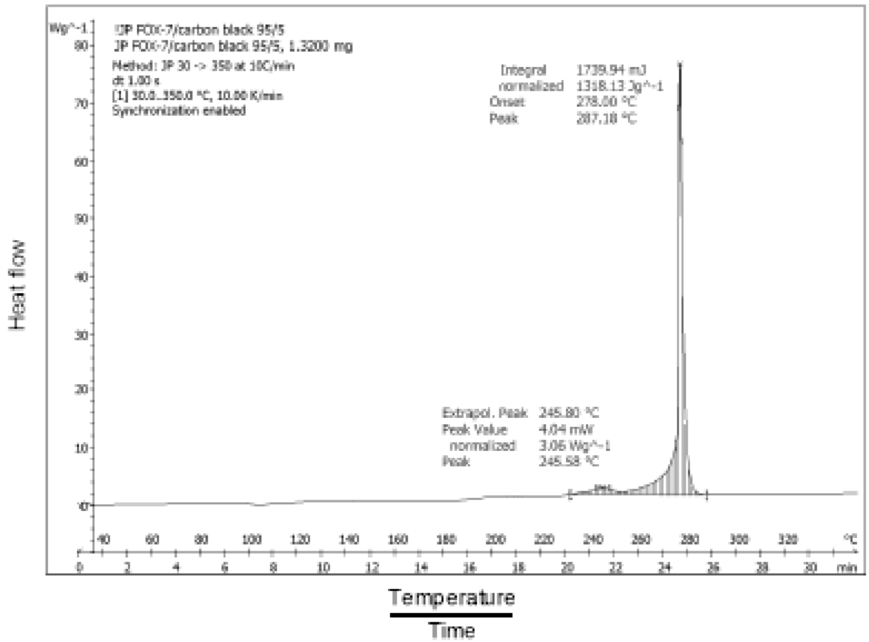


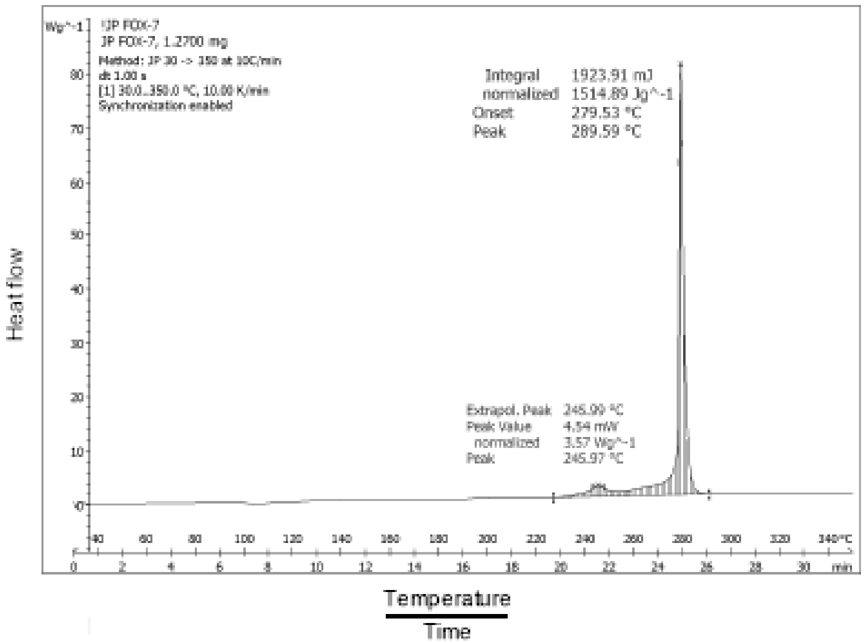
Figure 3 Optical microscopy of FOX-7/CB mixture sample

When laser radiation is incident on the sample surface, the small CB particles, as shown in Figure 2, which strongly absorb at all range of optical wavelengths are preferentially heated up by the laser to form hot spots [13] at their particle sites. The heat will then be transferred to the surrounding FOX-7 particles to raise the temperature of the explosive to its ignition point. Therefore, in spite of the explosive's poor optical absorption at visible and infrared region including the laser wavelength used, the inclusion of CB particles in the explosive greatly enhances its optical ignitibility. As main hot spot sources in laser ignition, CB particles have micro-sizes compared with mm-sizes of incident laser beam and the enhancement effect is contributed by all CB particles in the sample surface within the laser beam. Inhomogeneity in their density causes the contributions from different number of particles irradiated the laser beam and affects the reproducibility of ignition.

Activation energy for FOX-7 has been reported as 54 - 56 kcalmol^{-1} which shows that it is more thermally stable when compared to RDX at 40 kcalmol^{-1} and HMX at 35 kcalmol^{-1} [20,21]. This may render it less susceptible to laser ignition given the thermal nature of laser ignition process. To evaluate the compatibility of FOX-7 with CB, Differential Scanning Calorimetry (DSC) was conducted on the mixture sample and also the pure FOX-7 at a heating rate of 10 ^{o}C min^{-1}. The result, as given in Figure 4, shows two exothermal peaks at 245.58 °C and 287.18 °C for the mixture, and similarly 245.97 °C and 289.59 °C for the pure material. It shows that doping of a small amount of CB in FOX-7 didn't affect its exothermal peaks noticeably. This is in relatively good agreement with those reported in literatures [20,21] where exothermal peaks in the range 214 - 238 °C and 250 – 281 °C were identified for FOX-7 and were indicative of a two-stage decomposition. Therefore, FOX-7 has a good compatibility with CB and the inert material CB did not evidently affect thermal property of the explosive material. Also for safety assessment of the FOX-7/CB mixture, hammer testing on the samples was conducted and no reaction was observed. This result ensured the sample material had not been rendered unstable or sensitised to shock by the addition of CB.



(a)



(b)

Figure 4 DSC results for a) FOX-7/CB mixture and b) pure FOX-7

2.2 Laser ignition experiments

Laser ignition experiments were carried out using a lab-based set up as shown in Figure 5. The laser used was a fibre coupled laser diode (IPG PLD60-A-974) that operates at a wavelength of 974 nm in continuous wave mode. A laser diode controller (ThorLabs ITC4020) was used to control laser power output and pulse length. The laser output from the fibre exit of a diameter of 100 μm was focused with a lens (50mm diameter and 25mm focal length) onto the sample surface of explosive in the sample holder to ignite the explosive samples. The sample holder was consisted of 10 round holes of 3mm diameter and 2mm depth, and each sample of 7 mg was then lightly pressed into one of the holes using a pressing tool to ensure a flat surface and a consistent filling density (~0.5 gcm^{-3}). The sample holder was placed on a height adjustable platform so that laser spot sizes on the sample surface along the focusing beam were chosen for laser ignition. Two photodiodes were used to detect the incident laser and the light from the ignited combustion flame respectively. A light filter which filters out the laser was placed on a photodiode to collect the combustion signature. These photodiodes were connected to a digitizing oscilloscope (Agilent Technologies DSO5054A) in order to measure and record the temporal history of the ignited flame.

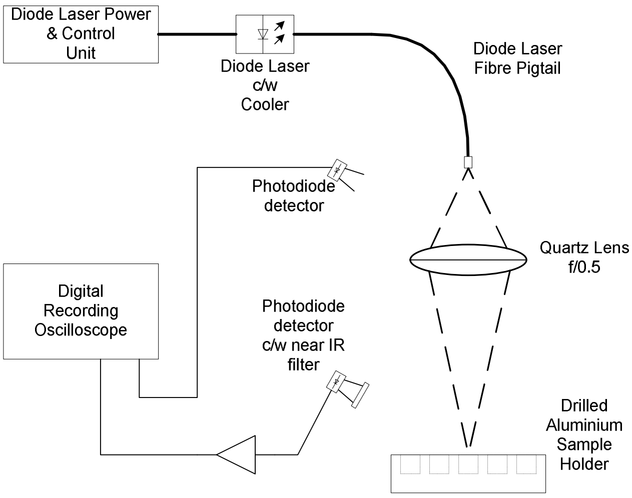


Figure 5 Experimental setup for laser ignition

In each of ignition tests oscilloscope traces of the light signals from the ignited flame and the igniting laser were recorded and temporal characteristics of the flame were measured in terms of ignition delay and full burn delay as shown in Figure 6. The ignition delay is the time taken between the commencements of the laser pulse and the flame, and the full burn delay is the time taken from the commencement of the flame to its 90% signal maximum. These measurements were carried out under various laser parameters including laser power, igniting beam size and laser irradiation duration. Also ignition tests (go/no-go testing) were conducted to determine ignition thresholds that are defined as the powers at which successful ignitions are achieved in over 50% of the tests. Ignitions with the laser powers near the threshold can be very inconsistent and as such the threshold power should be considered as an indication. Once the threshold had been determined, experimentation was conducted above the thresholds.

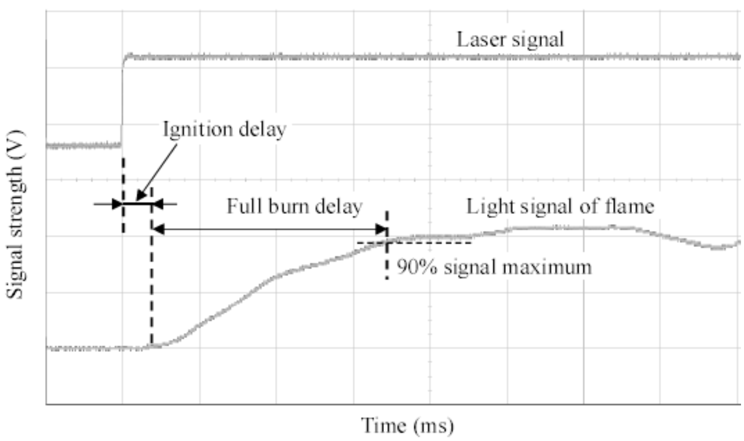


Figure 6 Oscilloscope traces of light signals from laser and the ignited flame

3 Results and Discussion

Laser ignition tests were carried out to evaluate the ignitibility under various laser parameters including laser power, laser beam size and laser duration. The results in the measurements of ignition delay, ignition threshold, full burn delay and the sample burning duration were obtained and analysed in following sections.

3.1 Ignition Delay

Ignition delays were investigated across a range of laser power settings and beam diameters in order to develop characteristic ignition maps for the material. The diameters of laser spots on the sample surface were 0.3 mm, 1 mm, 1.5 mm, 2 mm and 2.5 mm respectively. In order to firstly determine the threshold ignition powers, a series of 'go / no-go' tests were conducted at powers of 6 W to 10 W for 0.3mm beam and at a power of 8 W to 12 W for the other beam sizes. The laser power varied with 1W increments. For 0.3mm beam spot, 90% successful ignition was achieved at ~ 10 W and 50% at 9 W. Samples which did not ignite were observed to undergo decomposition, producing a grey smoke with no ignition. Therefore, the threshold power at this beam spot was approximately 10 W. The rates of successful ignition at 10 W for all the beam diameters are listed in Table 2. It is indicated that a smaller beam size or higher power density achieves more reproducible ignition and a laser power of 10 W was required for successful ignitions over 50% rate.

Table 2 Rates of successful ignition at laser power of 10 W

Beam widths (mm)	0.3	1	1.5	2	2.5
Success rate (%)	90	70	70	60	55

The ignition delays were measured at these beam spot sizes and laser powers from 10 W to 40 W. The ignition map (delay time versus laser power) for the laser beam spot of 0.3 mm was developed and presented with a trend line and error bars (~42%) in Figure 7. It shows how fast an ignition takes place at various laser powers. In the figure, each data point of delay time was an average of 10 repeated ignitions at a laser power and the error bars were given by their relative standard deviations averaged over the powers. It can be seen that at lower powers the ignition delay decreases sharply as the power increases and approaches to a nearly saturation level at medium laser powers (e.g. 40 W). The error bars also show larger delay deviations and thus poorer reliability at lower powers. The delay time was measured as ~ 80±33 ms (with 3 'no-go's not counted) at the threshold ignition power 10 W and ~ 3.8±1.2 ms at 40 W. The variance or error in ignition delay was mainly attributed to the effects of inhomogeneity in both the optical properties and packing density of the samples, as previously shown in Figure 3, particularly when using a relatively small beam width (e.g. 0.3 mm). This ignition map also shows that the ignition delay is controllable by igniting laser power, which would be a useful function in real applications.

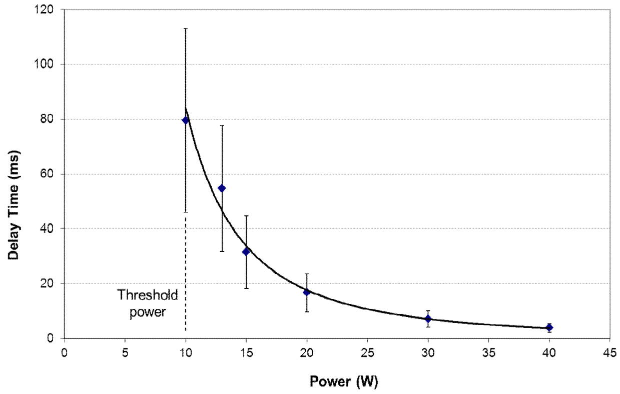


Figure 7 Ignition map for a 0.3mm beam width

Similarly the ignition maps for all the beam widths were developed and presented together in Figure 8 to allow a comparative analysis. From this graph it is clear that the larger the beam width the longer the ignition delay time in most of cases. This is attributed to the reduced power density at a larger beam width. Based on heat transfer theory for laser ignition [11] the temperature increase induced until ignition commences in explosive materials is directly proportional to the igniting laser power density and square root of delay time, as shown in equation 1 below.

$$\Delta T = \frac{2I_0}{k}\sqrt{\frac{K\tau_p}{\pi}} \qquad (1)$$

Where I_o is laser power density, τ_p is the ignition delay, *K* thermal diffusivity, *k* thermal conductivity of the material. Therefore, the lower power density requires a longer ignition time to increase the temperature of hot spots in the explosive to its ignition temperature. However, this equation doesn't take into account the effect of the radial heat dissipation from the exothermic reaction zone. Duginov's theoretical calculation [22] shows that due to this effect for a narrow beam, the threshold ignition energy density depends inversely on laser beam radius, as expressed below:

$$W_{th} \sim c\frac{\Delta T}{r} \qquad (2)$$

where *c* is a constant, ΔT is the difference between initial temperature and ignition temperature, and *r* is the beam radius. The ignition delay at 0.3 mm beam width was not shorter than the others in spite of its highest power density. The reason is that although pre-ignition reactions occurred much earlier than the ignition, as shown in Figure 9, the reactions did not progress to the full burn condition and thus they were discounted in the measurement. These may be due to the too small laser beam spot to induce sufficient number of hot spots on CB particle sites within the laser irradiated sample surface. It may also be attributed to an inefficient heat transfer in the sample of low packing density and radial heat dissipation [22] causing the increase of ignition threshold, as predicted by the equation (2). At 40 W the pre-ignition reaction occurred as early as 0.02 ms after commencement of laser radiation, which is expected for high power densities at this beam width. The ignition delays at 40 W for all the beam widths, and the threshold ignition powers with over 50% success rate of ignition are listed in table 3. This indicates the differences in ignition delay and the same threshold ignition power for various beam sizes. A minimum average ignition delay time of 70μs was achieved at a maximum laser power of 40 W and with 1.0mm beam width.

In order to evaluate the ignitibility enhancement of doping CB into the explosive, a series of laser ignition tests were also carried out for pure FOX-7 samples using the same settings of ignition parameters as those used for the CB-doped FOX-7. It was found that for the laser power of up to 40 W which was the maximum available in this study, the pure FOX-7 samples were not ignited at all. The reaction observed in such tests was the smoke due to slow heating up of the samples by the laser. The incapability of ignition on pure explosives was also found in Ahmad's research [5] in ignition of HMX using a high power diode laser. Without optically sensitizing, the explosive requires high ignition laser power to ignite. Below such threshold, the temperature of laser irradiated explosive may only increase to a saturated level below its ignition point because of the insufficient heating rate, as could be similar to McGrane's research results on high explosives such as TATB, HMX, etc [23]. It is evident that the doping of CB has played a dominating role in successful ignition by dramatically absorbing laser energy and providing hot spots sources for the explosive, consequently reduced the laser ignition threshold down to around a 10W level in this research.

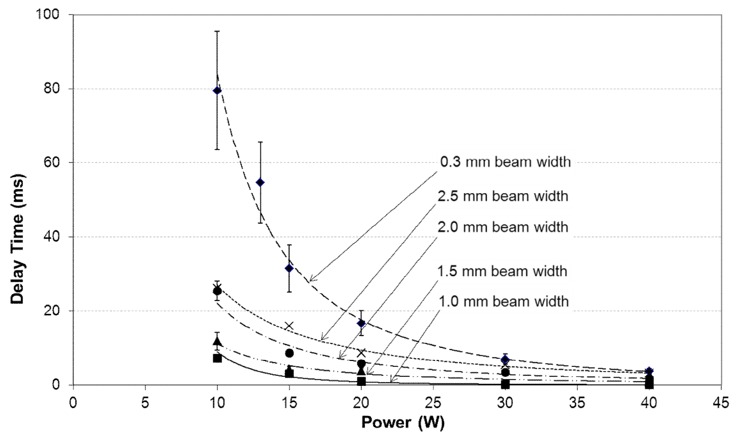


Figure 8 Ignition maps for various beam widths

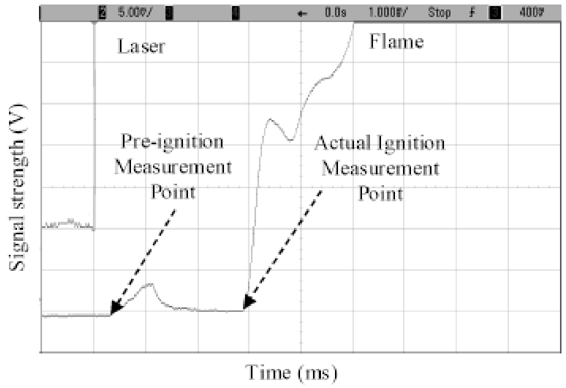


Figure 9 Oscilloscope trace of pre-ignition reaction and ignited flame

Table 3 Ignition delay at laser power of 40 W and threshold power

Parameters	Laser beam widths (mm)				
	0.3	1.0	1.5	2.0	2.5
Minimum Ignition Delay (ms)	2.38	0.07	0.32	0.22	2.53
Threshold power (W)	10	10	10	10	10

3.2 Full Burn Delay

In laser ignition, the times to reach a full burn condition from the start of ignition were also measured at various laser power and beam widths. These are presented in Figure 10, where the error bars are 29%, 17%, 23%, 23% and 19% for the beam widths from 0.3 to 2.5 mm respectively. It can be seen that the full burn delays decrease steeply with the increases of laser power at lower powers, and progress to saturation at higher powers. This is particularly evident for the experimentation conducted with a beam diameter of 0.3 mm. It is also shown that the difference in full burn delay for the different beam widths is minimal at the laser power of 40 W, but increases as the power is reduced. Due to the same effects found and explained for ignition delay, the full burn delay for laser beam of 0.3 mm was also longer than those for larger beam sizes. For a comparison of the effect, the critical energy densities for the full burn at laser powers of 40 W and 10 W are listed in Table 4 for all the beam sizes and plotted in Figure 11. The critical energy density was calculated based on the square pulse with the duration of the full delay time. It can be shown that the dependence of the critical energy density on beam size is significantly evident when a smaller beam (< 1 mm) is used. Also for a small beam (e.g. 0.3 mm), the critical energy density is much higher at a lower laser power (10 W) than that at a higher power (40 W), because of more radial heat dissipation from the slower heating by a lower power laser. This result well explained the longer delay time found for 0.3 mm beam. Overall the full burn time characteristics follow very closely the ignition delay characteristics. This parameter is a measure of how quick the burning of the explosive develops once it is ignited. It is of particular importance in the applications where explosives are confined to generate a sufficiently fast pressure rise for initiation or detonation.

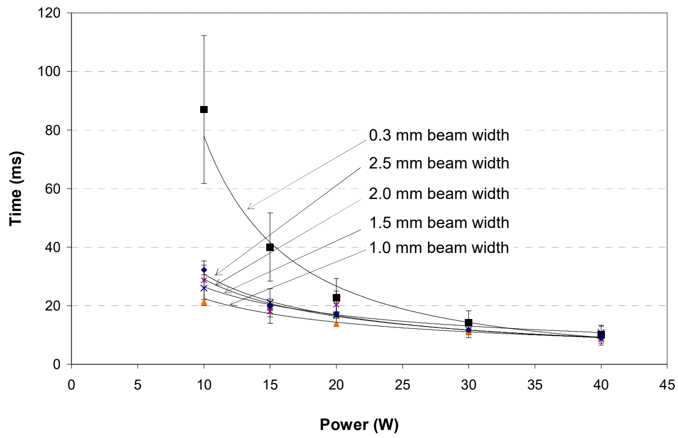


Figure10 Full burn delay vs laser power for various beam widths

Table 4 The critical energy densities for the laser beam diameters at 10 W and 40 W laser powers

d, mm	0.3	1	1.5	2	2.5
W_{10}, kJ/m^2	12243.5	270.1	162.5	91.4	65.6
W_{40}, kJ/m^2	5718.3	463.7	244.6	108.3	74.2

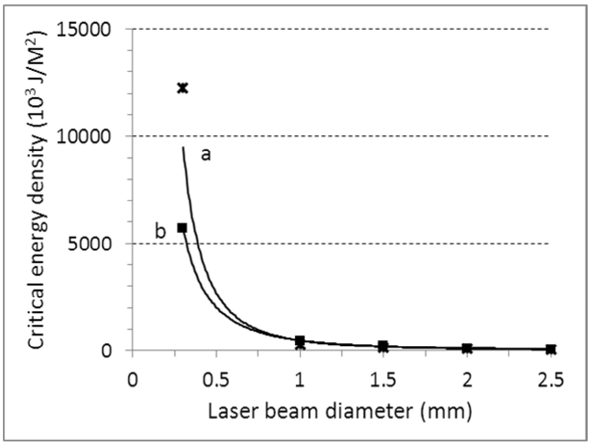


Figure 11 Dependence of critical energy density on beam size at laser powers: a) 10 W and b) 40 W

3.2 Sustainability

The ability of FOX-7 to undergo a sustainable combustion reaction was initially investigated by varying the laser pulse length (starting at 5ms with 5ms increments) at various incident laser powers at the beam width of 0.3mm, and determining the minimum pulse length required for a sustainable combustion to take place. In this study, a sustainable combustion reaction means that the laser induced combustion still continues after laser radiation stops at the end of its pulse length. From these measurements, the threshold ignition energy for a sustainable combustion is obtained by the product of the minimum pulse length required and the laser power used. In order to characterise the sustainability of the reaction, the resulting threshold ignition energies were graphed across the range of laser powers and shown in Figure 12. From the graph it can be seen that the required laser energy for a sustainable combustion decreases significantly with the laser power, and there tends to be a minimum ignition energy plateau that occurs at approximately over 40 W. When laser power is increased beyond this point the minimum ignition threshold energy will remain constant at approximately 0.1 J. Also minimum laser power of 10 W and energy of around 0.8 J are required to achieve a sustainable ignition. This ignition behaviour is expected as the increased laser power, i.e. energy input rate, speeds up the interaction of laser with the explosive material which also imply the accelerating of the formation of laser induced hotspots [13,14], and reduces heat dissipation. Therefore, the required ignition energy is reduced when a higher laser power is used.

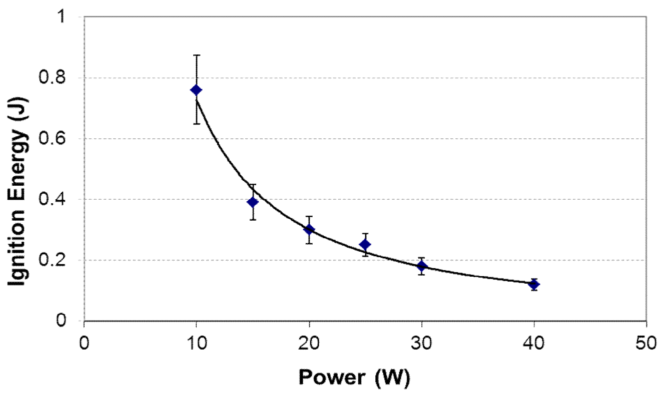


Figure 12 Threshold ignition energy for sustainable combustion at 0.3mm beam width

Subsequent experimentation investigated the ignition sustainability at larger beam diameters ranging from 1.0 to 2.5 mm at the laser power of 20 W and 40 W. In the ignition tests, the laser pulse length was changed in 5ms increments starting at 5ms, and the minimum pulse lengths to achieve sustainable combustion at these various power densities were obtained. The results are plotted as a function of power density against threshold pulse length in Figure 13. It can be seen that once the power density drops below 0.8 $kWcm^{-2}$ the required pulse length increases significantly, and for any higher power densities, the required pulse length was 5 ms or below. Therefore, in order to achieve sustainable ignition the power density is an affecting parameter. Although the above data shows the thresholds for sustainable ignition, it was found that a proportion of the sample remained un-burnt at the completion at laser power of 10 ~ 15 W for all the beam diameters (0.3 mm to 2.5 mm) used in this study. This poor sustainability is due to very low sample density and associated poor thermal conductivity which allows the heat produced during the slower reaction induced by the lower laser power to diffuse to the environment. Therefore, the minimum required laser power and energy to induce a sustainable combustion with the sample being fully burnt could be used to quantitatively evaluate the sustainability. Further research may consider the use of pellets to increase thermal conduction and also the use of confinement to increase the rate of reaction.

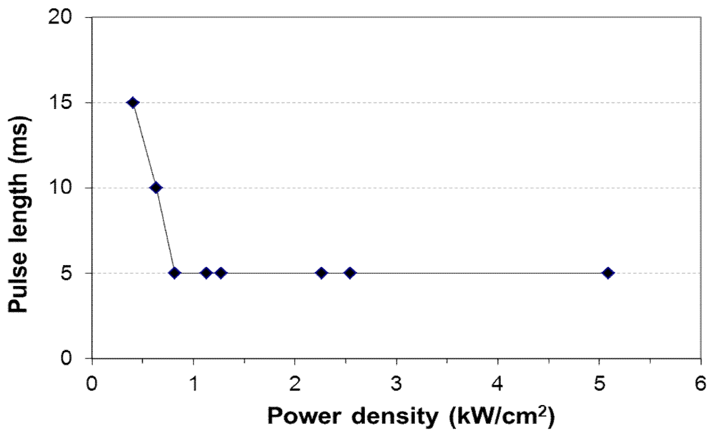


Figure 13 Threshold power densities for sustainable ignition

4 Conclusions

Carbon black (CB), as a dopant for optical sensitization, is compatible with insensitive secondary explosive FOX-7 due to its chemical neutrality. Relatively uniform mixture of a small amount (5 wt%) of CB with FOX-7 greatly enhances laser ignitibility of the explosive. Ignition delay and full burn delay decreases significantly and monotonically with the increase of laser power at a low power level, and slowly at a high power level to reach nearly constant minimum delay times. The threshold ignition power was found to be 10 W for all laser beam widths investigated, which may suggest that the ignition power threshold is independent of the power density under the experimental conditions of this study. However, laser beam width, or power density does affect ignition delay, and a smaller beam width allows shorter ignition delay unless it is too small (e.g. 0.3 mm in this study) which may have evident effect from radial heat dissipation. The effects of inhomogeneity in both the optical properties and material density of the samples on reproducibility of ignition become more evident when a relatively small laser beam is used. The minimum delay times of ignition and full burn can be achieved as 0.07 ms and 9 ms respectively at a laser power of 40 W. Sustainable combustion in laser ignition of FOX-7 can be achieved when the minimum power and energy required for ignition are met. The threshold ignition energy to achieve sustainable ignition was dependent on the applied power, and higher powers requiring shorter pulses and hence less energy. A further research may be considered on the effect of sample uniformity and density on laser ignition to achieve highly reliable and further enhanced ignitibility for the insensitive secondary explosive.

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