

Full length article

Optical sensitising of insensitive energetic material for laser ignition

Xiao Fang^{*}, Alexander J Walton

Centre for Defence Chemistry, Cranfield University, Defence Academy of the United Kingdom, Shrivenham, SN6 8LA, UK

ARTICLE INFO

Keywords:

Optical sensitising
Explosives
Laser ignition
FOX-7
Carbon black

ABSTRACT

An experimental investigation into optical sensitisation for laser ignition of an insensitive explosive 1,1-Diamino-2,2-dinitroethene (FOX-7) has been carried out, by using a near-infrared diode laser at a wavelength of 808 nm. In this study carbon black as the optical sensitizer was mixed at 5 wt% with the explosive using two different mixing techniques, tumble mix and ground mix. The mixture samples were characterised by microscopy to examine the dispersion of carbon black within the mixtures and analyse effects of the mixing techniques on their laser ignitability. Laser ignition maps were developed for both mixing techniques and varying sample densities also examined to determine the density effect on laser ignition with various laser parameters of beam width, laser duration and laser power. The results have shown that ground mixing method provides more uniform dispersion of carbon black in the mixture samples, and therefore allows a lower laser ignition threshold than that of tumble mixing method.

1. Introduction

Safety is an increasingly important driver in the development of explosive initiation systems. Laser's ability to directly initiate insensitive secondary explosives allows the removal of sensitive primary explosives and increases resistance to accidental initiation. It is environmentally friendly as current primaries include toxic heavy metals. Laser initiation systems are also not susceptible to electromagnetic interference and can produce multiple initiation sources due to their use of optical fibres. Laser ignition has been studied world-wide and in as early as 60s' by using lasers, e.g. Nd:YAG and ruby lasers, at various wavelengths such as 355 nm, 532 nm, 1064 nm and 694 nm respectively [1–3]. The research showed that bare Cyclotrimethylenetrinitramine (RDX) was unable to be initiated with a Nd:Glass laser at 1060 nm, however lasers at ultraviolet wavelengths were able to initiate unconfined explosive, which indicated the greater ignition efficiency of UV lasers of higher photon energy that may allow direct breaking of molecular bonds. In his research [4], Paisley also suggested that there are two separate mechanisms for the optical ignitions at UV and NIR wavelengths, being photo dissociation and thermal decomposition, respectively, and the addition of graphite (5–10 %) does not decrease the laser initiation power threshold. This was further explored by Östmark utilising tuneable CO₂ laser at 900 nm –1100 nm to initiate RDX [5]. The research showed that the laser absorption in the material followed the Lambert-Beer law and hence the absorption depth of the material irradiated is controlled mainly by the

material's absorption at the wavelength. Therefore, optical absorption of a specific material is significant for its laser ignition, which was also shown by the work on the Laser Ignition in Guns, Howitzers and Tanks (LIGHT) [6]. More recently, laser ignition method was experimentally investigated by using energetic nano-aluminium (n-Al) and polyvinylidene fluoride (PVDF) particles as optical sensitizer and sustained ignition of ammonium perchlorate (AP)/hydroxyl-terminated polybutadiene (HTPB) composite propellants was achieved with relatively low laser energy levels (less than 5 J/cm²) [7]. Pentaerythritol tetranitrate (PETN) and RDX were studied for their ignition with aluminium nano-powder as an optical sensitizer by a pulsed neodymium laser [8]. Also using a diode laser of low power, insensitive gun propellants based on RDX and nitrocellulose were investigated for laser power effects on their ignition and combustion characteristics [9].

1,1-Diamino-2,2-dinitroethene (DADNE) commonly referred to as FOX-7 is a modern example of insensitive secondary explosive. It is less sensitive than RDX but exceeds its performance; yet requires a greater stimulus to initiate. Thus, it is a widely researched material for use in insensitive munition applications [10–12]. Therefore, optical sensitisation of the material by mixing it with a laser absorption additive to increase its optical absorption would reliably achieve its initiation by the laser. Whilst metallic powders and other materials have been utilised as the additives, it is carbon black that has been proven to be an effective additive and the most widely studied especially in secondary explosives and pyrotechnics [13,14]. This paper has investigated the direct ignition

^{*} Corresponding author.

E-mail address: x.fang@cranfield.ac.uk (X. Fang).

<https://doi.org/10.1016/j.optlastec.2022.108767>

Received 21 April 2022; Received in revised form 26 September 2022; Accepted 3 October 2022

Available online 21 October 2022

0030-3992/© 2022 The Author(s). Published by Elsevier Ltd. This is an open access article under the CC BY license (<http://creativecommons.org/licenses/by/4.0/>).

of FOX-7, optically sensitized with carbon black, utilising a near-infrared (NIR) diode laser at a wavelength of 808 nm. Following our previous research [15–17], it has specifically examined the nature of the dispersion of carbon black within its mixture with the explosive and analysed two different mixing techniques. Laser ignition maps have been developed for both mixing techniques and varying sample densities examined to determine their effect on laser ignition with various laser parameters of beam width, laser duration and laser power.

2. Experimental

2.1. Sample preparation and characterisation

FOX-7 used in this project was in its powder form and examined utilising Scanning Electron Microscopy (SEM) shown in Fig. 1. The particles are comprised of flat plates a few microns thick with 20–40 μm width and up to 80 μm length. This large flat plate structure would lead to the formation of large voids in loose powder and light pressings, with particle fracture likely under higher pressing loads. FOX-7 displays high optical absorbance in line with the amino and nitro groups of its molecule at wavelengths of 2.94–3.12 μm and 6.06–7.40 μm [10,18]. Its lack of absorption in the near infrared (NIR) range defines the requirement for the addition of an optical sensitizer to absorb the laser energy at NIR for laser ignition of FOX-7.

The carbon black used for this study is a black aciniform, as can be seen in Fig. 2 where the material agglomerates to form larger clusters of up to 20 μm with the majority in 0.1–10 μm size. Such large particles of the material may cause undesirable uniformity when mixing with the explosives. The previous work at Cranfield [16,17] shows that the required mixing ratio of carbon black to increase absorbance varies considerably and addition of 5 wt% carbon black for mixing with FOX-7 obtained a good result for the laser ignition studies. Therefore, it was decided to utilise this ratio in this study. It should be noted that an optimum ratio may exist outside this value. The performance and sensitivity of an explosive mixture are affected by the addition of carbon black that reduces the explosive volume and can act as a grit sensitising the explosive to friction and grit.

To mix carbon black with Fox-7, the materials were weighed on a balance and then transferred to a glass vial, and an initial mixing was conducted by carefully rolling and inverting the glass vial producing an even mix of the carbon black and FOX-7. This sample type is referred to as Tumble Mix. A portion of this mixture was then transferred to a mortar and water was added to the mortar to a level just covering the surface of the mixture. Grinding with a pestle was conducted by hand for about 20 min until the mix no longer contained any grains that could be felt with the pestle. This was then dried in an oven under vacuum at 100° Celsius for two hours to remove the water. This mixture is referred to as Ground Mix.

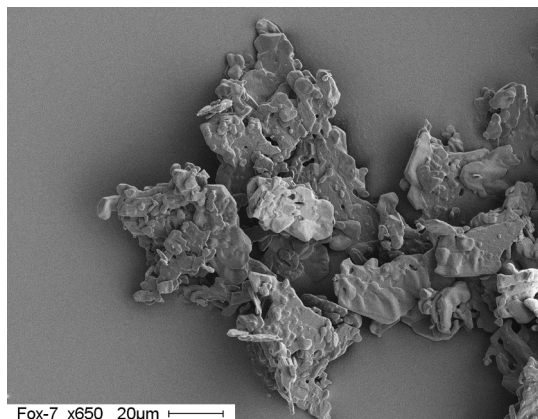


Fig. 1. SEM image of FOX-7 particles.

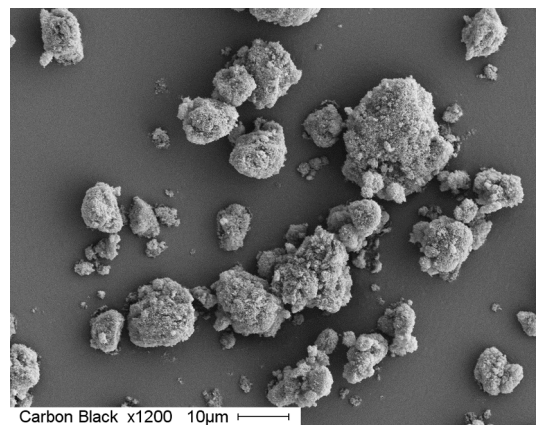


Fig. 2. SEM image of carbon black.

When mixed with carbon black utilising the tumble mix technique, the morphology of the FOX-7 particles remained the same as shown in Fig. 3. When the mixture of FOX-7 and carbon black was ground the morphology was considerably altered, as shown in Fig. 4 where the overall size of the particles has been reduced and they no longer exhibit the flat plate shape.

For each ignition experiment the mixture sample powder was weighed and then placed into individual cells within an aluminium sample holder; this holder allowed for ten samples to be prepared at a time. The powder was then pressed to a consistent volume using a Perspex hand press; this ensured a uniform surface and density between samples. The sample holder and the press are shown in Fig. 5. Each cell has 3 mm diameter and 2 mm depth and the used press (the protruding part) has 1.4 mm height and around 2.99 mm diameter. The pellet has around 3 mm diameter and 0.6 mm height. The examples of the pressed samples with Ground Mix and Tumble Mix were shown in Fig. 6 and Fig. 7 respectively for their optical microscopic images. The ground mixture appeared as a homogenous dark olive colour. The tumble mixture had the appearance of fine sand of black and yellow particles and agglomerations of both carbon black and FOX-7 leading to large variance in appearance across the sample. Sample density was calculated from a mass to volume ratio. For the investigation of density effects, a set of sample densities were obtained by pressing various masses into a fixed volume within the cells of the sample holder, as listed in Table 1, where the sample pellets have the same dimension of 3 mm diameter and 0.6 mm thickness.

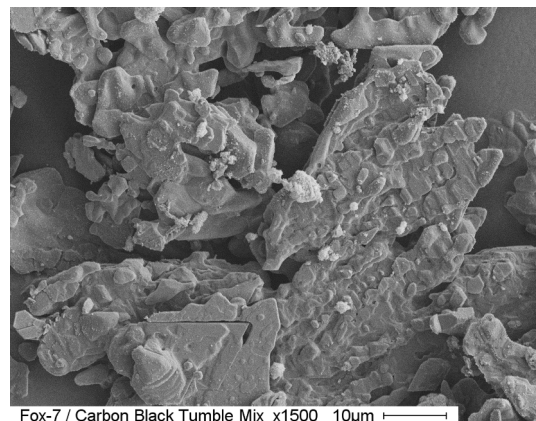


Fig. 3. FOX-7/carbon black tumble mix.

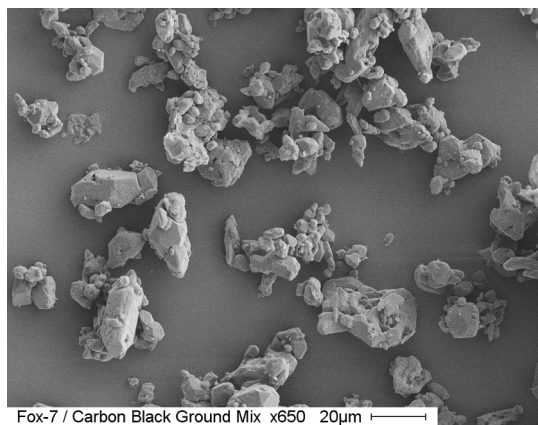


Fig. 4. FOX-7/carbon black ground mix.

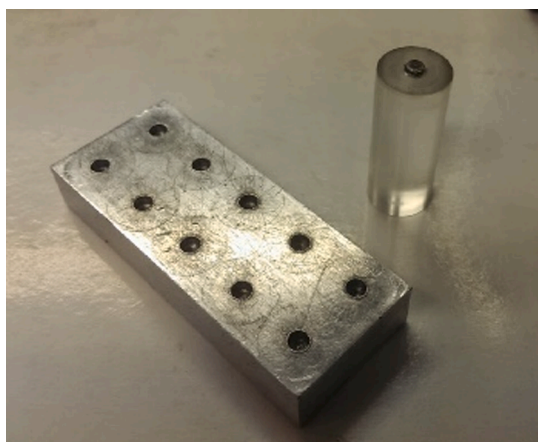


Fig. 5. Sample holder and press tool.

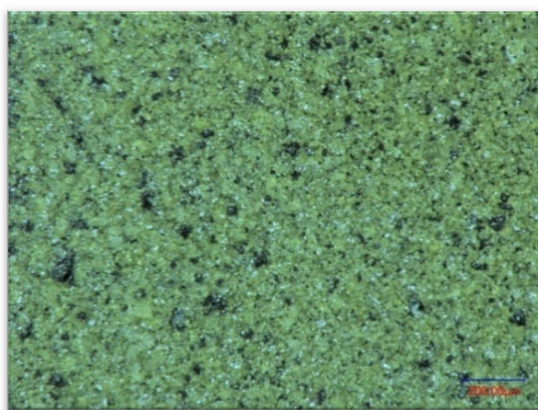


Fig. 6. Ground mix (200x zoom).

2.2. Experimental set up

2.2.1. Ignition testing

As shown in Fig. 8, ignition testing was carried out with the laser focussed on to the sample surface by a convex lens of 50 mm diameter 50 mm focal length. Two photo diodes were used to detect both the laser pulse and its ignited flame from the sample. A successful ignition event happens when the laser induces a flame (or visible burning) on the explosive. A laser filter centred at 800 nm was used to filter out the laser and enable the detection of the flame. A diode laser at the wavelength of

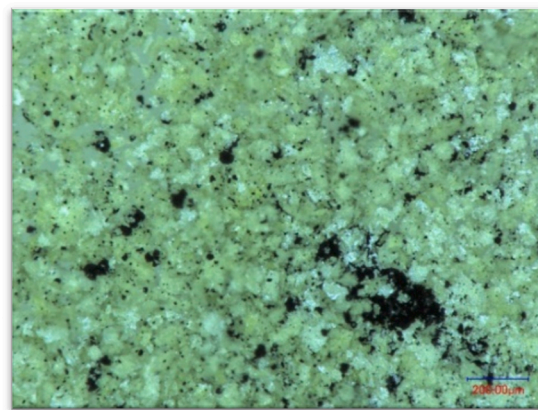


Fig. 7. Tumble mix (200x zoom).

Table 1
Sample densities examined.

Mass (mg)	3	4	5	6	7
Density (g.cm^{-3})	0.71	0.94	1.18	1.42	1.65

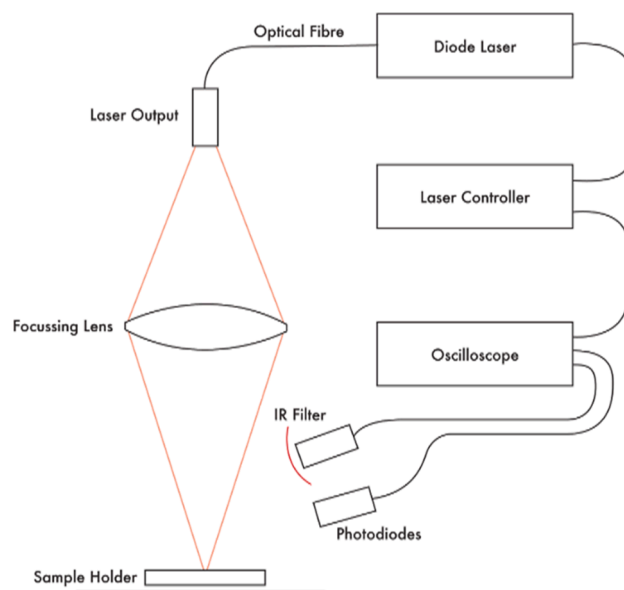


Fig. 8. Laser ignition experimental set up.

808 nm (JOLD 45 CPXF 1L Jenoptik) was used for all experimentation. This laser delivers a diverging beam through a 0.4 mm fibre core at a power of up to 45 W in continuous wave (CW) output. To control the power and timings of the laser a laser diode controller (LDC 1000, Laser Electronics) was used, and it also provided a trigger signal to a digital oscilloscope (Agilent Technologies DSO1024A). The diode laser system has a built-in pilot laser to provide a visible red beam in line with the 808 nm laser output for assisting with its alignment. To vary laser beam size on the sample surface, the sample holder was placed on a translator stage and moved up away from the focal plane of the lasers to obtain increased laser beam sizes.

2.2.2. Ignition map development

The ignition map was developed by measuring ignition delay time versus laser power. As shown in Fig. 9 for the oscilloscope traces of the igniting laser and the ignited flame, the ignition delay is the time taken between the commencements of the laser pulse and the flame, and the

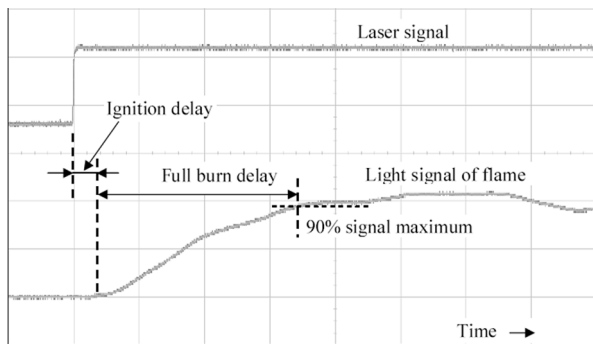


Fig. 9. Oscilloscope traces of the light signals from the laser and the ignited flame, defining the ignition delay time and the full burn delay time.

full burn delay is the time taken from the commencement of the flame to its 90 % signal maximum. The commencement of the ignited flame is measured at the time point after which the flame signal rises. The onset of the laser pulse was clearly defined in all tests, and this is used as the base reference for the ignition delay and full burn delay times.

These two values allow the creation of ignition maps by varying the power and comparing the delays. These measurements were carried out in the ignition tests under various laser powers, and the ignition threshold was determined when it achieved successful ignitions in over 5 out of 10 repeated tests. Once the threshold had been determined, experimentation was conducted above the thresholds. Unlike Bruceton method [19], the repeated ignition test method may directly indicate the ignition reproducibility of the samples produced with tumble and ground mixing techniques. Data in ignition maps is represented by mean values of the successful repeated tests at a laser power and the error is calculated by equation (1), with the experiment's standard deviation σ , being divided by the average value *Avg*.

$$\text{Error} = \frac{\sigma}{\text{Avg}} \times 100 \quad (1)$$

2.2.3. Absorption testing

To determine the laser absorption and diffuse reflectance of the two mixture samples using different preparation techniques, the experimental setup was used, as shown in Fig. 10. Additional optical shielding (not shown to simplify the diagram) was erected to reduce the light reflection and scattering from the surrounding background affecting the result. Sample material was weighed and placed inside a washer on a glass slide to ensure an equal density and sample depth between samples. Measurements were taken with barium sulphate as a reference since it has a high reflectance and is assumed to have negligible absorbance.

Using equation (2) it was possible to calculate the total intensity I_o from the results of the barium sulphate reflected and transmitted signals and using this value a relative value for the absorption of the explosive samples was obtained. This provided a coarse relative level for diffuse reflectance and absorption for the materials tested and more accurate analysis should utilise integrating sphere equipment.

$$I_o = I_t + I_r + I_a \quad (2)$$

I_o : Incident Irradiance.

I_t : Transmitted Irradiance.

I_r : Reflected Irradiance.

I_a : Absorbed Irradiance.

3. Results and discussion

3.1. Microscopy

SEM was used to assess the structure of the FOX-7 particles and

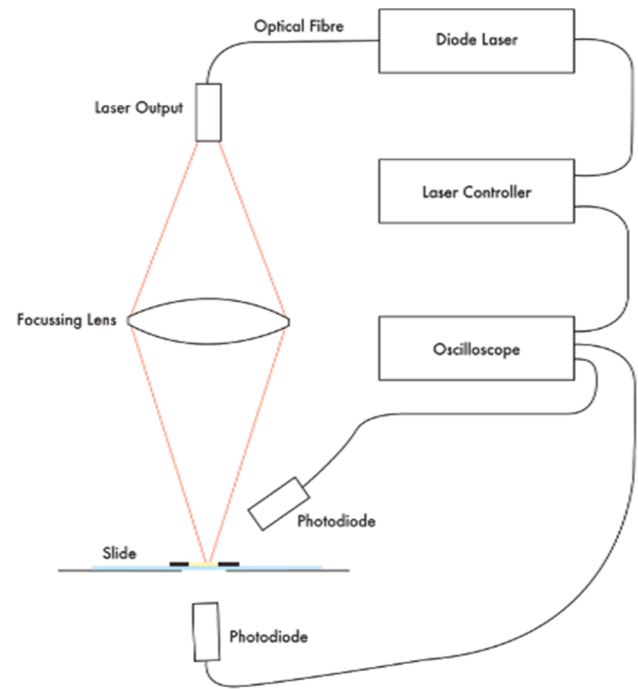


Fig. 10. Experimental set up for laser absorption measurement.

distribution of carbon black with the two mixing techniques. In the tumble mixture (see Fig. 3) the FOX-7 particles exhibited large flat plates. These plates were in the order of a few microns thick and approximately 20–50 μm across the other axes. Whilst the bulk densities were not directly measured it was noted when preparing samples of set densities for experiments that tumble mix had a significantly larger volume at each mass. It is likely that this plate like structure was the primary cause of the lower bulk density of the tumble mix, as packing of the large flat structures would lead to the formation of large voids within the material. It is recommended that future work investigating laser interaction with FOX-7 utilises a commercial grade of FOX-7 with its more spherical particle morphology. This is likely to lead to an improved density and the rheological improvements with this morphology compared to large flat plates may improve mixing between the explosive and absorbing particles. Also, in the tumble mixture carbon black particles which appear a lighter colour due to their rough surface, and a number of carbon black particles can be seen adhered to the FOX-7 particle, the rough surface of the larger particle appears to hold the carbon black particles. This fact could be utilised to design a FOX-7 particle shape to increase the trapping of carbon black powders in simple binary mixes, however it is more likely that including absorbing particles in a rubbery coating would improve the dispersion and also improve the sensitivity.

In the ground mixture (see Fig. 4), FOX-7 particles no longer exhibit a plate like structure and are significantly smaller with most particles being less than 20 μm across in any axis. Additionally, there is an increased range of sizes visible in the samples with a high proportion of smaller particles. This size distribution explains in part the higher bulk density of the ground mix. The particle shapes also vary significantly with some rounded and some rhombic sharp-edged particles evident within the samples. The appearance of some particles suggests that partial recrystallisation may have occurred, alongside mechanical breakup of the material under grinding. Water was chosen to wet the mixture for safety and is noted for being a poor solvent for FOX-7, some recrystallisation may have occurred with the pressure induced from grinding.

Optical microscopy was used for identification of FOX-7 and carbon black through their colour and allowed samples to be prepared with

similar physical properties. The images of both ground and tumble mixtures in Fig. 6 and Fig. 7 in above section enabled a larger area to be observed for the analysis of dispersion. Utilising ImageJ, a Java-based open-source image-processing software developed by the National Institutes for Health [20], particle analysis was conducted to determine the difference in the distributions of carbon particles between the two mixing techniques utilised. The image such as the one in Fig. 6 was first processed to isolate the carbon particles, as shown in Fig. 11, and then the particle analysis module within the software run, producing images such as Fig. 12 and data on the carbon particles. This allowed the number and size of the carbon particles to be summed and averaged. Ten sample pictures were taken for each mixing technique allowing a broad comparison to be made. It was established that the quantitative results obtained from the standard software suite were enough to make an assessment on the carbon particles between the two mixing techniques.

As summarised in Table 2, ImageJ particle analysis shows that tumble mix results in significantly fewer particles on average and that these particles are larger and cover less surface area of the sample. Additionally, it was seen that the tumble mix results had a larger variation in both the number of particles and the area they covered, it was anticipated that this would result in greater variation in absorption and ignition testing. The fewer particles and smaller area of carbon black within tumble mix would lead to greater areas without carbon black.

3.2. Optical absorption

The powder of barium sulphate was used as a reference sample for calibration of the total intensity, and it was assumed that this material had an absorbance of zero at the wavelength used. Equation (2) was used to establish I_0 from a summation of the outputs of both sensors in Fig. 10 of above section; this was then used to reference the results of the FOX-7 and carbon black mixtures. The results from testing tumble and ground mix are shown in Fig. 13. Neither of these mixtures allowed the transmission of light, thus it is only barium sulphate that has values recorded for I_t . FOX-7 was also tested and found to absorb slightly at the subject wavelength.

As predicted from the ImageJ analysis there was a significant difference in the results from the two mixing techniques as can be seen in Table 3, including the results of pure FOX-7 without mixing with any additive. The results also show that there is a greater variation in the results from the tumble mix than either the ground mix or the barium sulphate; this is to be expected from the results of the optical analysis. Tumble mix showed an error of 5 % for the samples I_r , ground mix 2 % and barium sulphate 0.7 %. Ground mix reflected 56.9 % I_{0Ref} indicating that 43.1 % I_{0Ref} was being absorbed by the material; this is to be expected as there is a greater area of carbon within the samples tested. Ground mix had 133 % greater carbon area compared with tumble mix,

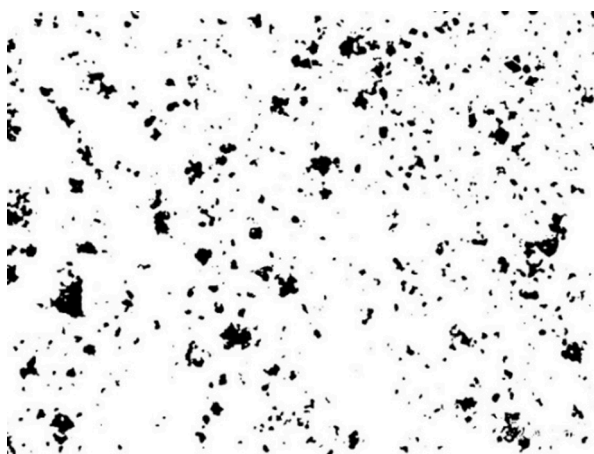


Fig. 11. Processed image showing carbon distribution.

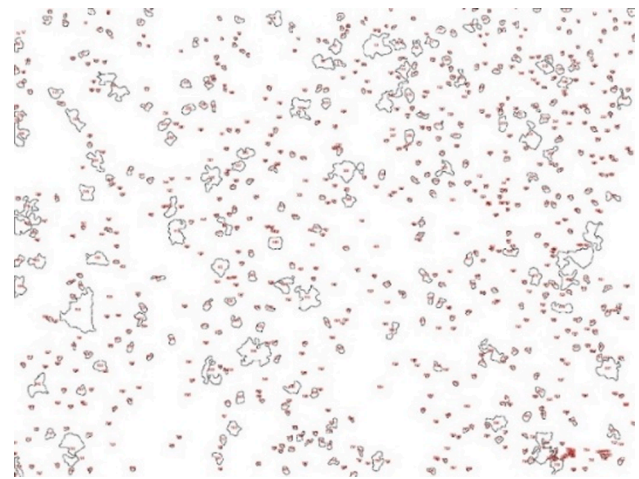


Fig. 12. Processed image showing particle assignment.

Table 2

ImageJ particle analysis summary.

	Count	Total Area mm ²	Average Size μm	% Area	Feret μm
TM Average	252	0.147	0.60439	6.429	24.8
GM	895.4	0.1974	0.22125	8.6067	17.2
Average					

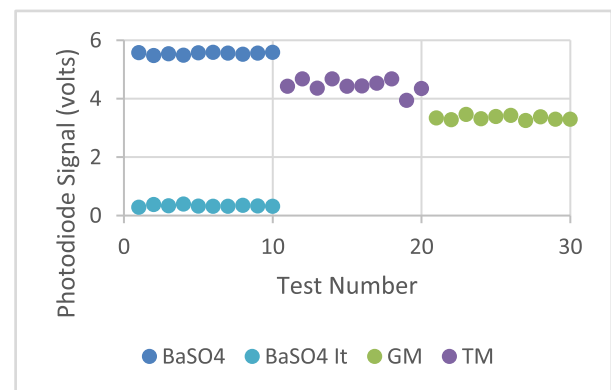


Fig. 13. Absorption testing I_r results.

Table 3

Absorption testing results (% I_{0Ref}).

	FOX-7	FOX-7/CB Tumble mix	FOX-7/CB Ground mix
Ia	9.2	24.3	43.1
Ir	85.6	75.7	56.9
It	5.2	0	0

however the absorption comparison is 177 %, and this suggests that the altered grain shape could also affect the absorption of the material.

The experimental method used for this analysis utilised a modified set up of the ignition testing rig and as such is limited to a simple comparison between the materials. Future work should consider the use of an integrating sphere equipment so as to get more accurate results. It is also recommended that alternate mixing techniques are investigated such as the use of Resonant Acoustic Mixing (RAM); this could improve mixing between the two materials without the physical impact to particle shape. It is also necessary in further research to understand how the porosity and percentage (%) of carbon black affect the optical

absorption.

3.3. Ignition delay

Initial investigations were conducted utilising a 1.0 mm beam width and a density of 0.64 g.cm^{-3} utilising a 'Go – No Go' assessment to identify the threshold power which was found to be 17 W for the ground mix. A range of laser powers were then utilised at this fixed beam width to determine a characteristic ignition map for ground mix and tumble mix. Fig. 14 shows the ignition delay from the threshold power of 17 W up to a maximum of 40 W. As expected, the ignition delay reduces as power is increased. Additionally, as power was increased a general trend of reduced error in results was also seen. The main values for this ignition map are listed in Table 4.

Characteristic ignition map for tumble mix is shown in Fig. 15. Tumble mix was seen to be far less consistent in ignition tests with more failures throughout the power ranges utilised. The threshold value for tumble mix was found to be 20 W, higher than that of ground mix. Of note is that the ignition delay at 20 W of successful tests is approaching the value at the highest power of 40 W, showing that tumble mix does not follow the expected trend. Additionally, the largest delays were seen at a higher power of 35 W. It is clearly shown that the uniformity of the mixture is an important factor for relatively small laser beam used in laser ignition. The main values for this ignition map are listed in Table 5.

Overall tumble mix produced larger errors compared with ground mix. Further analysis was conducted at 40 W, and it was seen that the range and deviation was significantly larger with the tumble mix in Fig. 16 than the ground mix at Fig. 17. These variations in ignition delays as shown in the two figures also indicate that the ignition energy (the products of laser power and the pulse duration) used for ground mix samples is more consistent than for tumble mix samples. Less variation within the dispersion of carbon black within ground mix is attributed as the primary reason for this improved performance.

However, resultant ignition testing for the pure FOX-7 without being mixed with carbon black found that even at the maximum 45 W laser power no reaction or ignition was observed, although it has low optical absorption as shown in Table 3. Therefore, its laser ignition threshold is higher than 45 W. The addition of carbon black made its threshold significantly lower.

3.4. Effect of beam size

Testing for the effect of laser beam size was conducted across a number of beam widths at a density of 0.64 g.cm^{-3} and a power of 25 W in order to determine if the improved dispersion of ground mix had any effect. As shown in Fig. 18, the average ignition delays of both ground and tumble mix increase in a linear trend with the beam width. Full burn delay follows the same trend and is shown in Fig. 19. There is no significant difference seen between the average values of the two mixtures, other than at 1.5 mm beam width, which was not expected following the

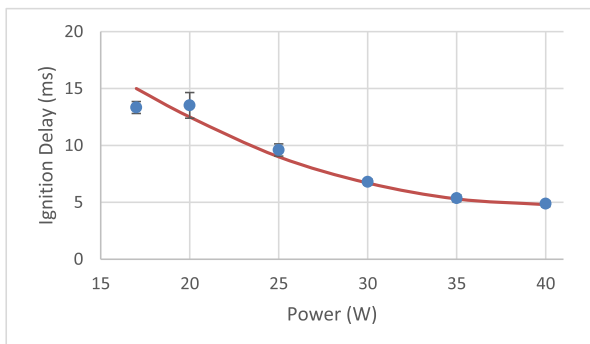


Fig. 14. Ignition map for ground mix.

Table 4

Ground mix ignition delay parameters (1.0 mm beam width) based on 10 shot tests.

Parameter	Value
Minimum Ignition Delay	4.9 ms @40 W
Threshold Ignition Power	17 W
Maximum margin of error	16.7 %
Minimum margin of error	7.4 %
Average margin of error	10.6 %

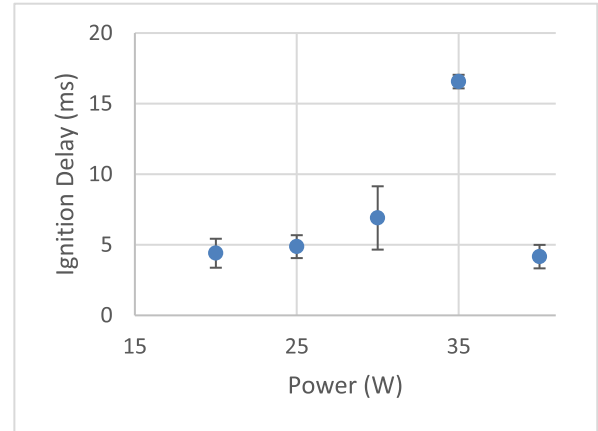


Fig. 15. Ignition map for tumble mix.

Table 5

Tumble mix ignition delay parameters (1.0 mm beam width) based on 10 shot tests.

Parameter	Value
Minimum Ignition Delay	4.2 ms@40 W
Threshold Ignition Power	20 W
Maximum margin of error	64 %
Minimum margin of error	5.8 %
Average margin of error	38.2 %

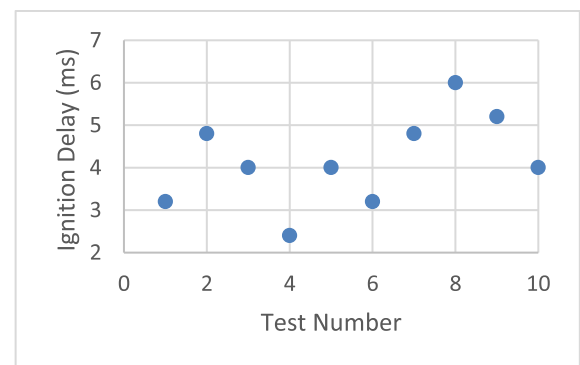


Fig. 16. Tumble mix ignition delay at 40 W and 1.0 mm beam width.

results from the ignition delay testing. Also, the density of laser power (P) is related to its beam width d as $I = P/(\frac{1}{4}\pi d^2)$ which indicating the effect of laser power density on ignition delay. The laser power densities for the used beam widths are listed in Table 6.

The error in the ignition delay was not seen to either increase with decreasing beam width, nor to differ significantly between either ground or tumble mix, as can be seen in Fig. 20. Analysis of the results in conjunction with the ignition delay results above suggest that the laser

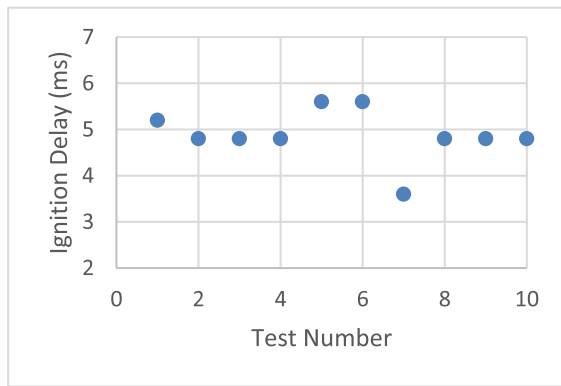


Fig. 17. Ground mix ignition delay at 40 W and 1.0 mm beam width.

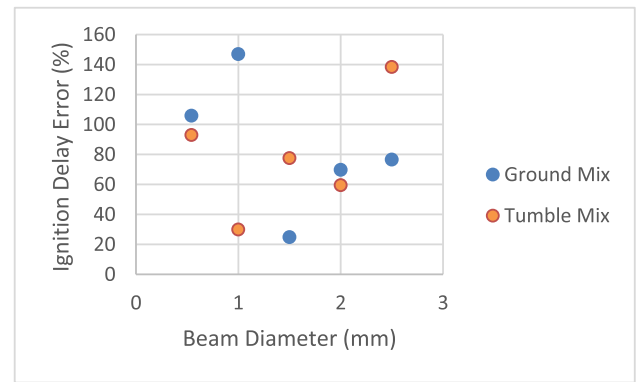


Fig. 20. Ignition delay error vs beam size.

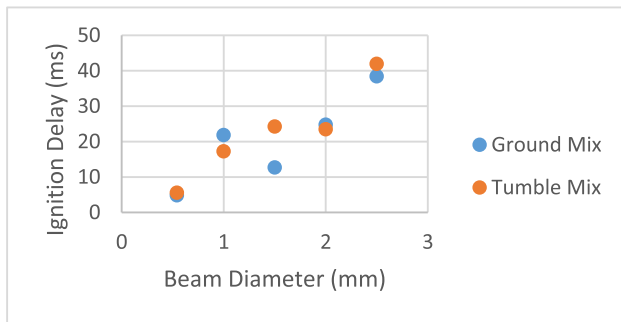


Fig. 18. Ignition delay vs beam size.

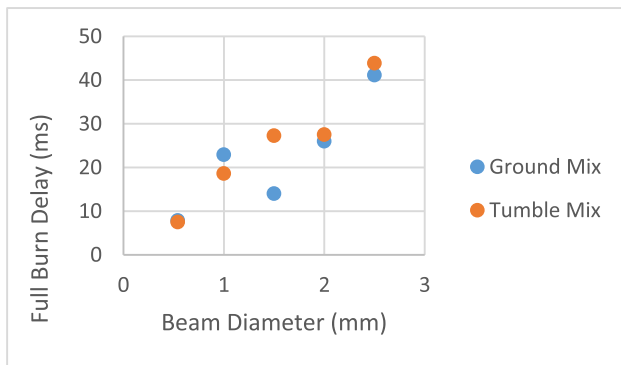


Fig. 19. Full burn delay vs beam Size.

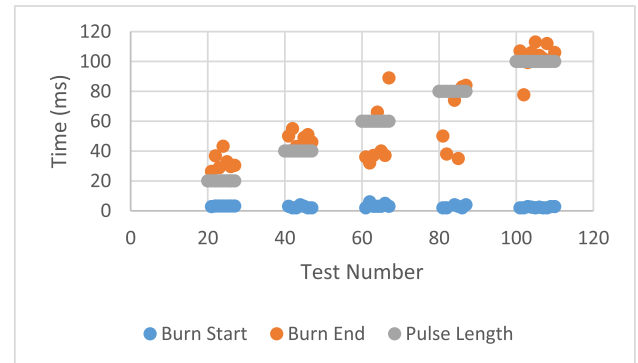


Fig. 21. Burn length for sample density 0.71 g.cm^{-3} .

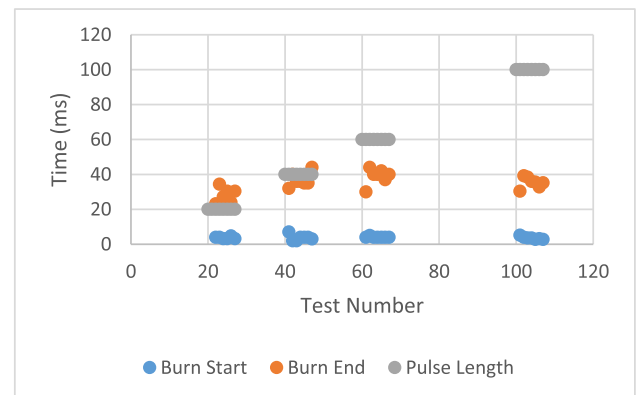


Fig. 22. Burn length for sample density 0.94 g.cm^{-3} .

Table 6

Laser power densities for the used beam widths at the power of 25 W.

Beam width (mm)	0.5	1.0	1.5	2.0	2.5
Power density (kW/cm^2)	12.74	3.18	1.42	0.8	0.51

power of 25 W used for this testing is too close to the threshold power for these materials, and therefore the errors could be associated with approaching this level of power rather than changes in the beam width.

3.5. Pulse duration

Further experiments were conducted using the samples with densities of 0.71 g.cm^{-3} and 0.94 g.cm^{-3} respectively to investigate the effect of the laser pulse length on full burn start and end time. The results are shown in Fig. 21 and Fig. 22, where the times of burn start, burn end and laser

pulse length are marked for each test. At 0.71 g.cm^{-3} it was evident that the flame output closely followed the length of the laser pulse or did not exceed the length of laser input by a significant margin. Also observed was the fact that the reaction did not progress outside of the cylinder illuminated by the laser within the material, and that at shorter pulse length the consumed material did not reach the lower surface of the sample holder.

The results for both densities show that the ignited burning of FOX-7 was not self-sustainable, and that the flame output would only last a short period of time following the cessation of the laser or ended before the end of the laser duration. There was sample material remaining after the burning ends, which indicates that the sample was not fully burnt and the combustion was not sustainable. It is suggested that heat is being drawn away from the reaction by the significant mass and relative low temperature of the sample holder and heat transport properties also

strongly depends on internal porosity of the samples. There is a significant difference in the outputs from the two densities tested, which could be attributed to the loose powder versus compacted material. The shorter pulse lengths for 0.71 g.cm^{-3} sample again support the fact that the reaction is not sustainable as the reaction has proceeded to a high output level prior to the laser pulse ending, yet the flame output ends in line with the laser. At 0.94 g.cm^{-3} this is not as evident however the 20 ms pulse shows that the reaction in this higher density does not progress to its full burn condition of a 40 ms burn.

4. Conclusions

Ignition was achieved with FOX-7 and carbon black ground mixtures at a lower laser power threshold of 17 W, with an average ignition delay of 13.33 ms at this power level. Across most of the experiments conducted this ignition was followed closely by a sharp rise to a high-level flame output. As laser power is increased the ignition delay and full burn delay decreases in a predictable manner, with the ignition delay reducing to 4.88 ms at the highest laser power of 40 W. However, tumble mixed samples of FOX-7 and carbon black had a significantly higher power threshold at 20 W with the limited results not displaying a uniform relationship between power and ignition delay. This was in part due to its inconsistent nature.

Dispersion of carbon black particles was assessed qualitatively, and it was found to be more consistent with ground mix samples, and that large areas of low carbon black particle density were common within tumble mixed samples. This was also supported by data extracted from quantitative analysis of optical microscopy that showed ground mixed samples had a greater number of carbon black particles and covered a greater area of the sample surface in comparison to tumble mix. Predictably this led to a significant difference in the absorption of laser light between the two FOX-7 and carbon black mixtures, and additionally both mixtures showed an improvement over pure FOX-7. Ground mix was shown to absorb 43.1 % of incident laser power in comparison to 24.3 % for tumble mixed. Ground mix also displayed less variation in the results achieved in absorption testing.

Testing across various beam widths showed that ignition delay increased with a reduction in power density, highlighting that whilst there is a threshold power, there is also threshold energy for laser ignition. Experiments conducted across various densities showed that there were two behaviours evident between low density loose powders and compressed higher densities. Thermal conductivity improves as the powder densities increase and this was seen to increase the rate at which FOX-7/carbon black burns. It was found that a density of 0.94 g.cm^{-3} to 1.17 g.cm^{-3} produced the quickest burn.

Sustainability was not witnessed in the tests conducted, with significant volumes of the sample material remaining after ignition. The volume of sample illuminated by the laser was the only material to react. Thermal conduction to the sample holder is suggested as the primary cause of this; however, the effect of pressure was not examined as all experiments were conducted unconfined. Reduction in laser pulse length at various densities showed that the burn length, whilst exceeding the shorter pulses to some extent, followed the laser pulse length reduction.

Overall FOX-7 with 5 % carbon black can be ignited with the use of a diode laser at 808 nm with a minimum power of 17 W. Ground mixing method shows more uniform dispersion of carbon black in the mixture samples, and therefore allows a lower laser ignition threshold than that of tumble mixing method. However, under the conditions tested the ignition reaction was not sustainable.

CRedit authorship contribution statement

Xiao Fang: Conceptualization, Validation, Resources, Writing – original draft, Writing – review & editing, Visualization, Supervision, Project administration. **Alexander J Walton:** Methodology, Software, Formal analysis, Investigation, Data curation, Writing – original draft, Visualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

Acknowledgement

The authors would like to thank Dr James Padfield and Dr Jonathan Painter for their technical supports in sample preparation and characterisation.

References

- [1] A.A. Brish, A. Galeyev, The mechanism of initiation of condensed explosives by laser radiation, *Fiz. Goreniya i Vzryva* 4 (1969) 475–480.
- [2] A.M. Renlund, P.L. Stanton, W.M. Trott, Laser initiation of secondary explosives, 9th Int. Symp. Detonation (1989) 1118–1127.
- [3] M. D. Bowden, M. Cheeseman, S. L. Knowles, R. C. Drake, Laser initiation of energetic materials: a historical overview. *Proc. SPIE* 6662 (2007) 666208–666208–12.
- [4] D. L. Paisley, Prompt detonation of secondary explosives by laser, 9th Int. Symp. Detonation (1989) 11110–11117.
- [5] H. Ostmark, M. Carlson, K. Ekvall, Laser ignition of explosives: Effects of laser wavelength on the threshold ignition energy, *J. Energ. Mater.* 12 (1994) 63–83.
- [6] A.W. Barrows, B.E. Forch, R.A. Beyer, J.E. Newberry, Laser Ignition in Guns, *The LIGHT Program, Howitzers and Tanks*, 1993.
- [7] K. E. Uhlenhake, M. Gomez, D. N. Collard, M. Örnek, S. F. Son, Laser ignition of solid propellants using energetic nAl-PVDF optical sensitizers, *AIAA SCITECH 2022 Forum*.
- [8] B.P. Aduiev, D.R. Nurmukhametov, I. Y. Liskov, A.V. Tupitsyn, G.M. Belokurov, Laser pulse initiation of RDX-Al and PETN-Al composites explosion, *Combust. Flame* 216 (2020) 468–471.
- [9] L. Courty, P. Gillard, J. Ehrhardt, B. Baschung, Experimental determination of ignition and combustion characteristics of insensitive gun propellants based on RDX and nitrocellulose, *Combust. Flame* 229 (2021) 111402.
- [10] I. J. Lochert, FOX-7 - A New Insensitive Explosive (2001).
- [11] Euroco | FOX-7. at <http://www.euroco.com/content/explosives/defence-security/high-explosives/insensitive-explosives/fox-7/>.
- [12] H. Bergman, A. Pettersson, H. Östmark, FOX-7, an IM ingredient candidate - Where are we today? *Insensitive Munitions Energ. Mater. Technol. Symp.*, 2009.
- [13] N.K. Bourne, On the laser ignition and initiation of explosives, *Proc. R. Soc. A Math. Phys. Eng. Sci.* 457 (2010) (2001) 1401–1426.
- [14] S.G. Goveas, R.C. Drake, J.E. Field, A study of the laser ignition of HMX/Carbon Black compositions, 30th Annu. Conf. ICT, 1999.
- [15] S.R. Ahmad, A.E. Contini, X. Fang, Laser ignition of PolyPZ-Q (An optically sensitized energetic polyphosphazene) and its formulations with HNS, *Annu. Conf. ICT*, 1991.
- [16] X. Fang, W.G. McLuckie, Laser ignitability of insensitive secondary explosive 1,1-diamino-2,2-dinitroethene (FOX-7), *J. Hazard. Mater.* 285 (2015) 375–382.
- [17] X. Fang, S. Ahmad, Laser ignition of an optically sensitised secondary explosive by a diode laser, *Central European, J. Energ. Mater.* 13 (1) (2016) 103–115.
- [18] N.V. Latypov, J. Bergman, A. Langlet, U. Wellmar, U. Bemm, Synthesis and reactions of 1,1-diamino-2,2-dinitroethylene, *Tetrahedron* 54 (38) (1998) 11525–11536.
- [19] R. Wild, E. Collani, Modelling of Explosives Sensitivity Part 1: The Bruceton Method, *Economic Quality Control* 17 (2002) 113–122.
- [20] T.J. Collins, ImageJ for microscopy, *Biotechniques* 43 (1S) (2007) S25–S30.



DSPACE

<https://dspace.org/>

Optical sensitising of insensitive energetic material for laser ignition

Fang, Xiao; Walton, Alexander J.

2022-10-21

Elsevier

<https://doi.org/10.1016/j.optlastec.2022.108767>

Fang X, Walton AJ. (2023) Optical sensitising of insensitive energetic material for laser ignition. Optics and Laser Technology, Volume 158, Part A, February 2023, Article number 108767