

Full length article

Laser ignition of energetic crystals of cyclotrimethylenetrinitramine (RDX) optically sensitized with gold nanoparticles and light-absorbing dye

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ABSTRACT

Laser ignition of energetic materials has shown promise in eliminating sensitive primary explosives to initiate a main explosive charge. To enhance the laser ignitability of energetic material cyclotrimethylenetrinitramine (RDX) with a near-infrared diode laser, the RDX was re-crystallized with three optical sensitizers: COOH-PEGylated gold nanorods (both solution and dry gold nanoparticle) and laser absorbing dye. Optical microscopy was undertaken on the crystals to investigate the uniformity of doping of the optical sensitizers. High-speed video was used to experimentally observe the enhanced laser ignitability of these RDX crystals. The solution gold nanoparticles enhanced doping uniformity resulting in it being an effective optical sensitizer. Compared to the other two optical sensitizers, the solution gold nanoparticles optical sensitizer significantly enhanced the laser ignitability of RDX by reducing the required power density to as little as 79 Wcm^{-2} to achieve ignition. At this laser power density, the ignition delay was approximately 68 ms.

1. Introduction

A primary explosive was traditionally used in the detonator because of its higher sensitivity. For high power in the main charge, secondary explosives are used as they are harder to initiate due to lower sensitivity. Since primary explosives are easy to ignite and demonstrate a very rapid transition from deflagration to detonation, they are very susceptible to accidental ignition through small stimuli (e.g., impact, friction, electrostatic discharge, and heat) [1], making them inherently unsafe for use. Examples of commonly used primary explosives include lead azide and lead styphnate [2]. Unlike cyclotrimethylenetrinitramine (RDX), a secondary explosive, which has a standard Figure of Insensitiveness (FoI) value of 80, lead azide and lead styphnate have the FoI value of 20 and 12 [3], respectively, hence they are very sensitive to accidental impact. Also most primary explosives are harmful to the environment. Lead has been extensively researched, and its environmental impact is well known and documented [4–6]. New regulations, including REACH, have increased the demand for environmentally friendly explosives to be used by progressively banning non-environmentally friendly substances. As research continues to look for alternative ‘green’ primary explosives [2,7–9], a growing number of alternative initiation systems look to remove the need for primary explosives in the explosives train.

The simplest and the safest technique to ignite energetic material is

through the direct laser irradiation of secondary explosives where the laser creates a hotspot on, or below, the irradiated material surface [10–12], and the hot spot grows until it transfers sufficient energy to the bulk of the material to cause an ignition. Using a high-power CO₂ laser operating at $10.6 \mu\text{m}$ [13], Ali et al investigated the ignitability of triaminotrinitrobenzene (TATB) and cyclotetramethylenetetranitramine (HMX) with a focus on the time for them to reach the ignition temperature. They found that to achieve an ignition, the explosive must reach a minimum surface temperature and a minimum energy density within the solid. Research undertaken by De et al [11] showed the energy required for initiation of non-primary explosives by a CO₂ laser operating at $10.6 \mu\text{m}$ was significantly less than using a xenon photoflash irradiation. Their work also showed exothermic decomposition is induced by the thermal heating occurring within the explosive because of the absorbed irradiation. Both studies indicate the need for optical sensitization of the materials.

The ability of energetic materials to absorb laser energy directly influences how easily thermal ignition will be achieved. At ultraviolet (UV)/visible wavelengths optical energy is generally strongly absorbed by the energetic materials, with weak absorption in the near infrared region [10]. However, lasers in UV/visible region are generally bulky and costly, making them not suitable for use as an initiator within a warhead or other portable devices [14]. Therefore, to use infrared lasers

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as the ignitors, research has been undertaken to improve the optical absorption of the energetic materials across the near infrared band. This can be achieved by the addition of inert additives as optical sensitizers to energetic materials. In the last two decades, the additives including carbon black [15–19], laser absorbing dye [20], and metallic particles [21–25] have been used as optical sensitizers for laser ignition of energetic materials. Ehrhardt et al [26] achieved successful ignition of RDX/nitrocellulose using a diode laser with 5 W power at 808 nm wavelength. For this typical low-sensitive explosive RDX, Xu et al [24] achieved laser ignition of RDX mixture with high active aluminium powder using a CO₂ laser with 10.5 W power at 10.6 μ m wavelength, while Fang et al [25] successfully ignited RDX crystals doped with gold-nanorods using a diode laser with 1 W power at 808 nm wavelength.

The research of this paper has investigated three optical sensitizers: COOH-polyethylene glycol (PEG)ylated AuNPs (both solution and dry powder), and laser absorbing dye to determine how they affect the absorption characteristics of re-crystallized RDX crystals. Optical microscopy was undertaken on the crystals to investigate the uniformity of doping of the optical sensitizers. High-speed video was used to experimentally observe the enhanced laser ignitability of the RDX crystals using a near infrared diode laser, in terms of required power density and corresponding ignition delay time.

2. Experimental

2.1. Sample materials and preparation

The energetic material used for optical sensitization was RDX. The solvent used to re-crystallize the RDX was analytical grade acetone (Sigma-Aldrich Ltd., UK). Three optical sensitizers were used, including laser absorbing chemical dye with an optical absorption band of around 811 nm and COOH-PEGylated AuNPs (NanoHybrids, USA) in the forms of dry powder and suspensions in deionized water at a concentration of 4 mg/ml, with the absorption band of around 808 nm.

Four samples (S1, S2, S3 and S4) were prepared using RDX re-crystallization mechanism based on evaporation, as shown in Fig. 1a–1d, respectively. Samples S1, S2 and S3 contain 0.5 wt% of the three sensitizers, respectively and sample S4 was pure RDX recrystallized without any additive. To obtain sample S1, 600 mg of RDX powder was weighed in a vial, and 12 ml of acetone was added to achieve critical saturation (~5 wt%) [27]. At room temperature, the vial was sealed with a seal cap and shaken every 10 min until the RDX powder was completely dissolved. To ensure the RDX was unsaturated, a further 2 ml of acetone was added. The vial was resealed, shaken, and left for 10 min. Three mg of an infrared dye Heptamethylcyanine (ADS800AT, America Dye Source, Ins.), having an absorption band at 811 nm, was mixed with the RDX-acetone solution in a 60 ml flask. The flask was sealed with a film containing one pinhole and left in a fume cupboard to evaporate naturally, resulting in completely dry crystals (Fig. 1a). RDX sensitized with dry powder AuNPs (sample S2) were prepared equivalently to sample S1, except that the chemical dye was replaced with 3 mg of the dry powder of AuNPs (Fig. 1b). RDX sensitized with AuNPs dispersions

in deionized water (sample S3) were prepared equivalently to sample S1, except that the chemical dye was replaced with 3 mg of the AuNPs suspended in 0.75 ml of deionized water (Fig. 1c). Pure RDX (sample S4) was prepared equivalently to sample S1, except that no optical sensitizer was added (Fig. 1d).

2.2. Sample characterization

Optical microscopy was carried out using a Leica DMLM microscope to investigate the optical sensitizers on the surface and within the RDX crystal samples. The microscope was capable of resolutions in the micrometer scale (1000 $\times$ magnification). The crystals were etched to determine visually whether the optical sensitizer was purely on the surface or had been incorporated inside the single crystal. To etch the RDX crystal, filter paper (Whatman grade 6) with a pore size of 3 μ m was placed on a flattened glass plate. Five drops of analytical grade acetone (Sigma-Aldrich Ltd., UK) were placed on the filter paper. Using plastic tweezers, the RDX crystal was rubbed on the acetone-soaked filter paper in figure of eight motions. Once the filter paper had dried, this process was repeated until the top layer of the crystal was removed, exposing the inner surface of the crystal. Once the top layer was removed, the crystal was stroked in three straight lines on a new acetone-soaked filter paper to ensure no doping material was deposited on the etched surface. To ensure the inner surface was exposed, optical microscopy images of the crystal were taken prior to etching commencing.

UV-Vis Spectroscopy analysis was carried out using an Evolution 220 UV-Vis Spectrophotometer device to investigate whether the dye, and AuNPs were incorporated within the RDX crystal, which were not visible through the optical microscopy analysis. To baseline the UV-VIS Spectrophotometer device, a 5 ml glass cuvette was filled with analytical grade acetone (Sigma-Aldrich Ltd., UK), and a baseline analysis was undertaken. At room temperature, using a 10 ml measuring conical flask, approximately 1 mg of un-etched sensitized RDX crystal was dissolved in acetone. This dissolved solution was transferred, via pipette, to the dried cuvette used in the baseline run. The analysis was performed from 350 to 1100 nm due to acetone's ability to absorb UV light between wavelengths from 220 to 340 nm [28]. This process was repeated for the etched crystals.

2.3. Laser ignition setup

Fig. 2 shows the experimental setup for the un-confined laser ignition by an 808 nm diode laser (JENOPTIK JOLD-45-CPXF). The laser was operated in CW mode with adjustable power of up to 45 W. Individual crystals were placed on an aluminum plate, with the upper surface of the sample exposed for laser irradiation. The laser beam spot size was 2.5 mm in diameter, which was measured by firing the laser for 0.1 s at 5 W onto laser absorbing paper and measuring the size of the marked spot with a Vernier microscope. The laser beam was focused onto the sample using a lens of 50 mm in both diameter and focal length. A pilot laser beam was used to align the sample to the igniting laser beam. For analysis of the enhanced ignitability of the crystal sample, a high-speed

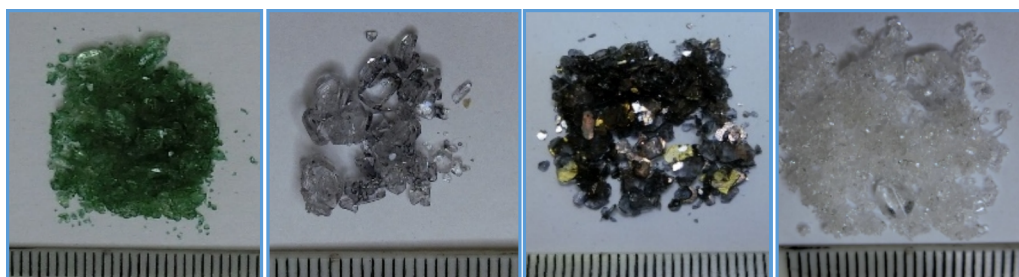


Fig. 1. Photos of re-crystallized RDX (the graduations are in millimetres) sensitized with a) dye, b) dry powder of AuNPs, and c) AuNPs dispersed in deionised water, and d) without sensitization.

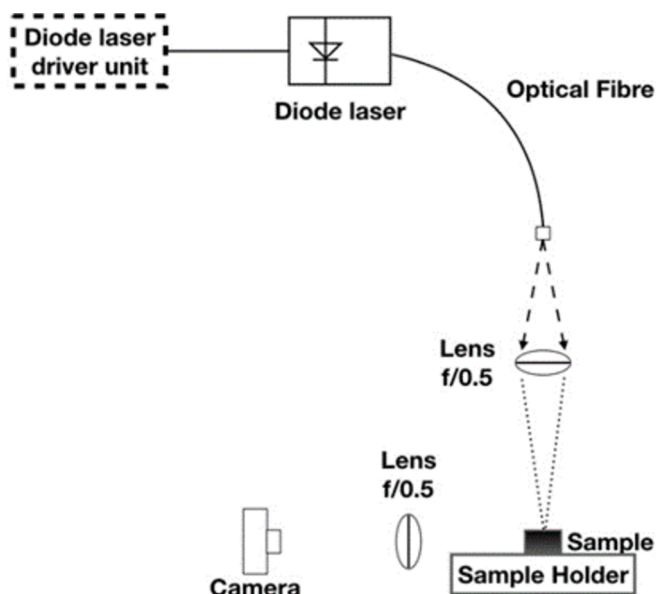


Fig. 2. Laser ignition setup.

camera (Casio EX-ZR800) of up to 1000 frames per second was fitted on an optical rail with the image magnified through a lens with the same specification as the above to record the side view of the sample. VLC Media Player v3.0.3 was used for analyzing each video frame.

3. Results and Discussion

3.1. Optical microscopy analysis

Optical microscopy analysis was undertaken on all crystal samples to visualize the effects of dye and AuNPs doping. The optical microscopy images of un-etched and etched samples S1, S2 and S3 are shown in Fig. 3. The dye and AuNPs can be seen coated on the surfaces of the RDX crystal before their etching, as shown in Fig. 3 a, c, e, respectively. Overall, the level of surface coating of sensitizer was quite high and consistent between the individual crystals for samples S1 and S3. For sample S1 in Fig. 3a, it appears that during the crystallization, as the acetone evaporated, the dye only resulting in little or no aggregation of the dye with RDX crystals in acetone. Conversely, for sample S3 in

Fig. 3e, the solution of AuNPs appears to be incorporated into the surfaces of the crystals, which suggests as the acetone evaporated during the crystallization, there was aggregation of the AuNPs with RDX crystals in acetone, and this led to more uniform coating than that of the dye coating on sample S1, as shown in Fig. 3a.

As highlighted in Fig. 3c, sample S2 also had surface deposits of optical sensitizer, which were not incorporated into the surface. This suggests that as the acetone evaporated during crystallisation, some of the AuNPs were deposited on the outer surface of the RDX crystals. It was noted that the side of crystal which was touching the bottom of the flask used for the crystallisation had a reduced coating of dye and AuNPs. Visual inspection of the flask revealed that optical sensitizer remained on the flask surface, suggesting that some of the sensitizer was detached from the crystal surfaced during crystal removal.

Etching the surface of RDX crystal sensitized with dye revealed the sensitizer was only deposited on the surface of the crystal, as no dye sensitizer was visible on the inner surface under microscope, even at 1000 x magnification. This provides further evidence that during sample preparation there was no aggregation of the laser absorbing dye with RDX crystals in acetone.

The post-etch results obtained for the RDX crystals sensitized with AuNPs were mixed. Some of the crystals tended to reveal that there was not AuNPs optical sensitizer on the inner surface, while others reveal that their inner surfaces had limited amounts of AuNPs present, as shown in pre- and post-etched sample surface images in Fig. 4. These AuNPs on the very limited locations on the inner surface may exist as impurity during the recrystallization that has not had an opportunity to reject the AuNPs from the individual crystal lattice [29]. This indicates that there was aggregation of the solution AuNPs with RDX in acetone during crystallisation. Limitations in microscope resolution meant nano-scale details of the RDX crystals could not be visually seen or analyzed. Further research needs to be undertaken to fully understand the level of doping within the crystals. It has been previously proven that an increased surface area of AuNPs and RDX interfaces when AuNPs are doped inside of the RDX crystals results in a more efficient heat transfer from AuNPs to their surrounding RDX, and no direct heat dissipation into the air [21].

3.2. UV-VIS spectroscopy analysis

UV-Vis spectroscopy was undertaken on all samples to determine the absorbance of light across the spectrum of wavelengths to verify the visual observations of dye and AuNPs doping. Additionally, the

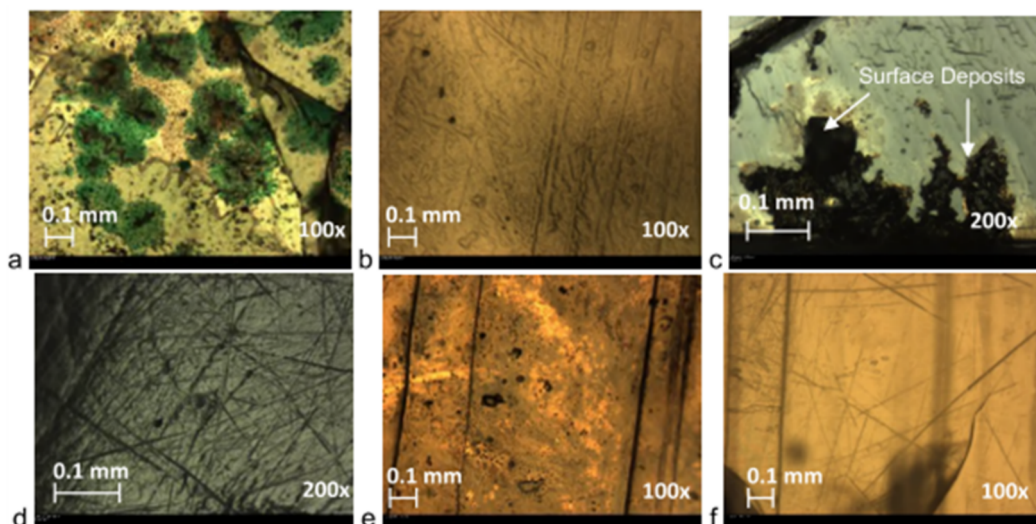


Fig. 3. Optical microscopy images of RDX crystal surfaces of: a) sample S1, b) etched S1, c) sample S2, d) etched S2, e) sample S3, f) etched S3.

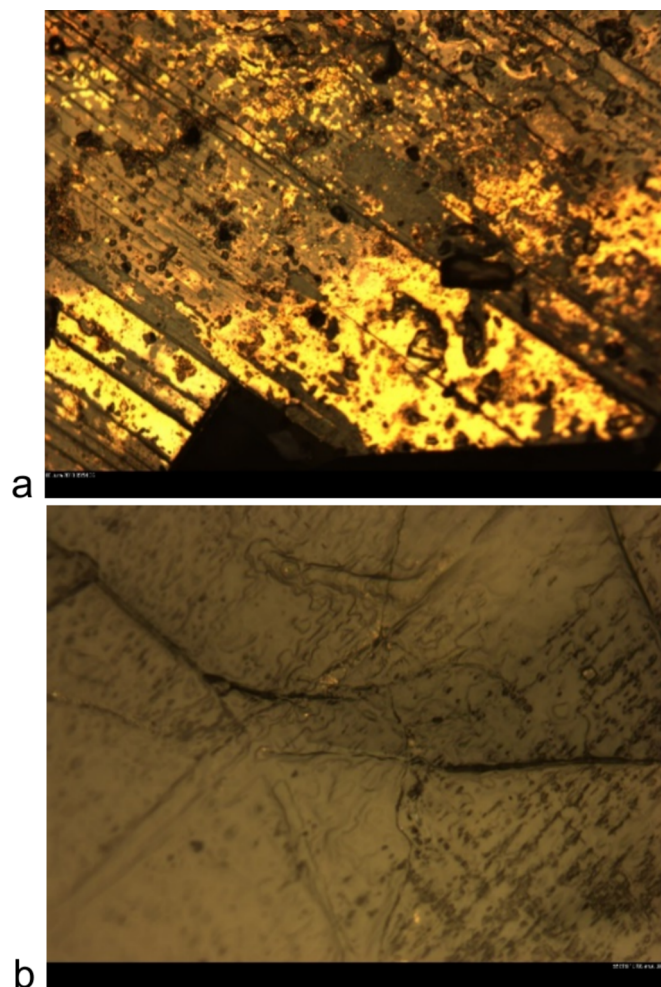


Fig. 4. Optical microscopy images of sample S3 crystal surfaces: a) before etching (at 100x magnification), b) after etching (at 200x magnification), the small yellow patches show the AuNPs present on the etched crystal surface.

absorbance of light would determine whether doping was achieved throughout the crystal for optical sensitizer particles, which may have been too small to see during optical microscopy. Fig. 5 shows the

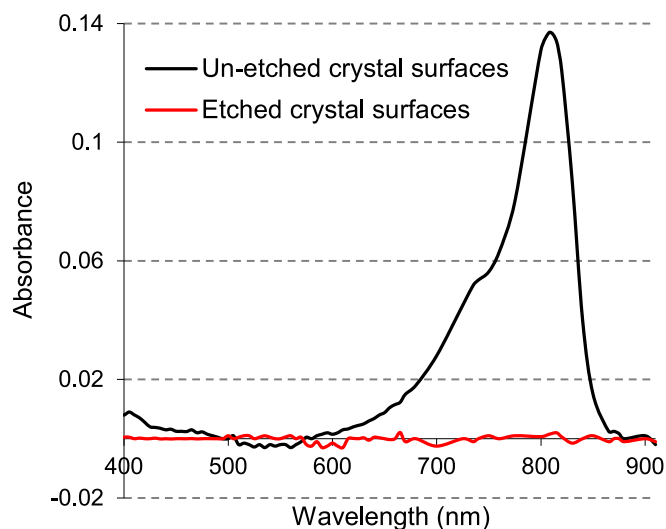


Fig. 5. Absorption spectra of RDX crystals sensitized with dye for their etched and un-etched crystal surfaces, respectively.

absorption spectra of sample S1 with and without being etched. It shows that the non-etched crystal had an absorbance peak at 808 nm, in line with the manufacture specifications of the dye. For the etched sample S1, no absorbance peak was obtained, meaning the dye had only been coated on the crystal surface, which is consistent with its optical microscopy analysis. For samples S2 and S3, no light absorption was observed for both their non-etched and etched crystals (Therefore, the spectral data was not recorded). This is believed to have occurred because the acetone had not suspended the AuNPs sufficiently to be present for the light absorption. Because of limited samples, and the destructive nature of this test, other suspension solutions were not investigated.

3.3. Laser ignitability

3.3.1. Laser ignition threshold

Laser ignition tests were undertaken over a range of laser power densities, on the three sensitized RDX samples and a pure RDX sample to determine their laser ignitability. At each power density, seven repeated ignition attempts for samples S1 and S3 were undertaken, and five ignition attempts for sample S2. Most of produced sample crystals were too small for the test and therefore only limited number of samples were available for ignition test. The ignition success rate (%) was defined as the number of successful ignitions out of the number of attempts at the same laser power density with laser irradiation duration of 300 ms. It was defined that a successful ignition has a success rate of 50 % or above. Table 1 shows the success rates at various power densities for the three samples. With an ignition success rate greater than 50 %, the laser ignition thresholds were approximately 100 Wcm^{-2} (or 30 Jcm^{-2}) for S3, 142 Wcm^{-2} (or 42.6 Jcm^{-2}) for S1 and 203 Wcm^{-2} (or 60.9 Jcm^{-2}) for S2. For sample S4 (pure RDX), no ignition was achieved even using the highest power density available of 914.4 Wcm^{-2} (or 274.3 Jcm^{-2}). Under optical microscopy analysis, it was seen, as shown in Fig. 6, that the laser induced heat was not able to cause ignition on the crystal.

As expected, sample S3 was the most optically sensitive RDX crystal, as AuNPs greatly enhanced the laser ignitability of explosives when compared to sample S1 using dye. However, sample S2 required higher power densities to achieve ignition when compared sample S1. This may be attributed to the less coated area by AuNPs on S2 than that by dye on S1 and therefore, a less heated surface area was generated on S2.

3.3.2. Laser ignition maps

To evaluate the influence of applied laser power density on the ignition delay time, laser ignition tests were performed on all three samples. With each frame of the high-speed video equating to 1 ms, the ignition delay time was measured as the time between the first frame in which the laser hit the sample and the commencement of the ignited burn of the sample. Fig. 7 plots, with trend lines, the measured ignition delay times at a variety of laser powers densities. This plot is commonly referred to as an 'Ignition Map'. For all three samples, the ignition map shows the ignition delay sharply decreases as the powers densities changes from 100 Wcm^{-2} to 400 Wcm^{-2} and trends towards a saturated ignition delay at higher powers densities. At the highest available power density (914.4 Wcm^{-2}), the ignition delay time was recorded as 1 ms across all three samples. It is noted that this high-speed camera only allows up to 1000 frames per second, and thus the measure is limited to

Table 1

The success rates of laser ignition for optically sensitized RDX crystal samples at various laser power densities.

Sample type	Ignition success rate (%) at the laser power densities (Wcm^{-2}):				
	60.7	101.6	142.2	203.2	304.8
S1	0	0	57	100	100
S2	0	0	0	60	100
S3	14	71	100	100	100

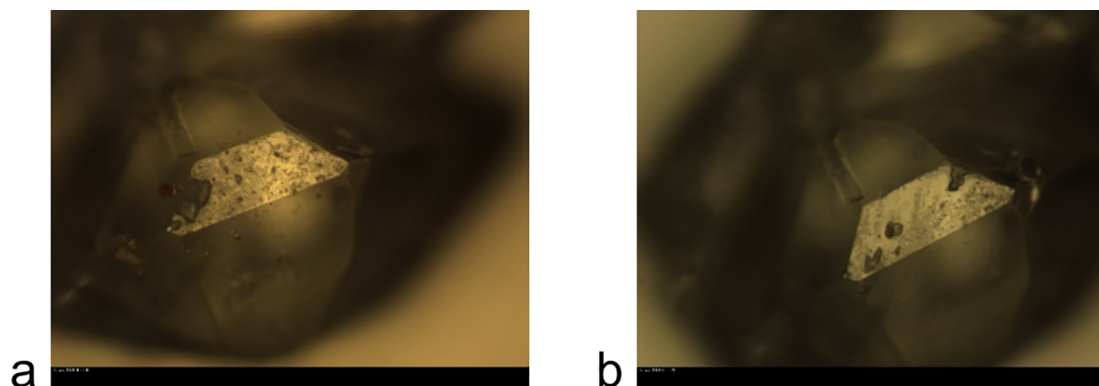


Fig. 6. Optical microscopy images (with 100x magnification) of a pure RDX crystal surface: a) before and b) after laser ablation.

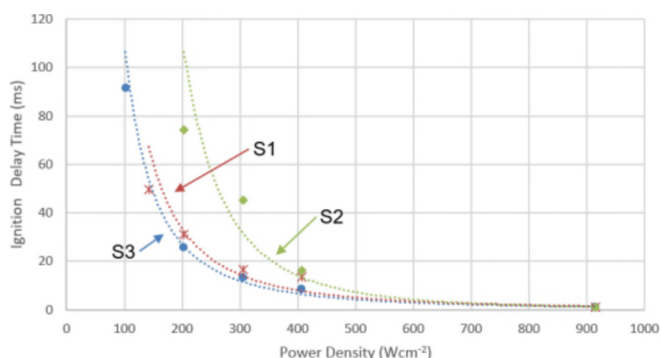


Fig. 7. Ignition delay time vs. laser power density for each optically sensitized RDX crystal. The relative standard deviation was approximately 25%.

1 ms. With the comparison of ignition delays at low success ignition power density for the three samples, e.g. 203.2 Wcm^{-2} , sample S3 shows faster ignition ($\sim 23 \text{ ms}$) than S1 ($\sim 29 \text{ ms}$) and S2 (75 ms).

3.3.3. Effect of laser spot size

A larger laser beam size 4.2 mm in its diameter, which was slightly bigger than the RDX crystal, was used for laser ignition tests of samples S3. Five repeated ignition attempts were undertaken at laser power density of 79 Wcm^{-2} . The achieved ignition delay time in average was around 68 ms and the ignition success rate was 80% . Both results were improved from the previous results using smaller laser spot size (2.5 mm in diameter) which achieved delay time of 91 ms and success rate of 71% at the laser power density of 101.6 Wcm^{-2} . This may indicate that the threshold laser power density and ignition delay time were reduced with larger laser beam spot on the crystals. However, more experimental ignition testing, and accurate measurement of laser spot size are required to address such effects in good details and accuracy in our future research.

4. Conclusions

This study has experimentally confirmed the effectiveness of AuNPs, which have been functionalized with a COOH-PEGylated group, as an optical sensitizer for RDX crystals (S3) when using an 808 nm CW laser as the ignition source. These RDX crystals were proven to have superior optical sensitization to both dye-coated (S1) and dry powder COOH-PEGylated AuNPs (S2) coated RDX crystals. Using a 2.5 mm (diameter) beam, and with an ignition success rate greater than 50% , the threshold laser power density for sample S3 was reduced to approximately 100 Wcm^{-2} , whereas ignition could not be achieved with pure RDX crystals using the highest power density of 914.4 Wcm^{-2} . At the threshold laser power density, the ignition delay time was

approximately 91 ms and sharply decreased as the power densities was increased to 400 Wcm^{-2} and tended to saturation beyond this power density. A larger laser beam spot improves the ignition performance of the optically sensitized RDX crystals in terms of threshold laser power density, ignition delay and success rate.

CRedit authorship contribution statement

Xiao Fang: Writing – review & editing, Writing – original draft, Validation, Supervision, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Andrew Wells:** Writing – original draft, Validation, Methodology, Investigation, Formal analysis, Data curation.

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 new data were created or analysed in this study. Data sharing is not applicable to this article.

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