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14. ABSTRACT

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a. REPORT	b. ABSTRACT	c. THIS PAGE			Dana Dlott
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RPPR Final Report
as of 11-Jul-2023

Agency Code: 21XD

Proposal Number: 73041WS **Agreement Number: W911NF-19-2-0037**
INVESTIGATOR(S):

Name: Ph.D. Dana D. Dlott Ph.D.
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Phone Number: 2173332187
Principal: Y

Organization: **University of Illinois - Urbana - Champaign (UIUC)**
Address: c/o Office of Sponsored Programs, Champaign, IL 618207406
Country: USA
DUNS Number: 041544081 EIN: 376000511
Report Date: 30-Jun-2023 Date Received: 11-Jul-2023
Final Report for Period Beginning 01-Dec-2018 and Ending 31-Mar-2023
Title: Detonation on a tabletop
Begin Performance Period: 01-Dec-2018 **End Performance Period:** 31-Mar-2023
Report Term: 0-Other
Submitted By: Ph.D. Dana Dlott Email: dlott@illinois.edu
Phone: (217) 333-2187

STEM Degrees: 1 **STEM Participants:** 2

Major Goals: Detonation on a tabletop refers to experimental studies in the Dlott group at the University of Illinois that involve using a pulsed laser to launch small hypervelocity flyer plates at tiny charges of various explosives while diagnostics with high time and space resolution probe the explosive response. There were four main thrust areas with corresponding publications listed below. These were: (1) Experimental advances; (2) Hot spots and detonations in plastic-bonded explosives (PBX); (3) Shocked reactive materials. The high points were: (1) Improved methods for temperature measurements; (2) Multimaterial simulations of laser-launched flyer plate launch and impact; (3) Tabletop detonations produced in nitromethane (NM) and NM with amine catalyst and visualization of novel cellular structures; (4) Hot spots dynamics in several PBX; (5) Hot spot dynamics visualized in single and poly crystals of HMX; (5) Fast energy release in reactive materials.

Accomplishments: Please see summary document uploaded.

Training Opportunities: Nothing to Report

Results Dissemination: The results of our project were disseminated by a combination of publications in archival journals, presentations at universities and other research organizations, and reports filed with ARO marked for unlimited distribution.

A number of organizations expressed interest in some of the techniques we developed to produce and study hypervelocity impacts.

Lawrence Livermore National Laboratory has developed a temperature measurement system based on our 32-channel pyrometer. Sandia National Laboratory developed a laser flyer launcher based on our design. Johns Hopkins University also developed a similar flyer launcher.

Honors and Awards: Nothing to Report

Protocol Activity Status:

Technology Transfer: Nothing to Report

PARTICIPANTS:

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Participant Type: Postdoctoral (scholar, fellow or other postdoctoral position)
Participant: Meysam Akhtar
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
National Academy Member: N

Participant Type: Postdoctoral (scholar, fellow or other postdoctoral position)
Participant: Siva Kumar Valluri
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
National Academy Member: N

ARTICLES:

Publication Type: Journal Article Peer Reviewed: Y **Publication Status:** 1-Published
Journal: Applied Physics Letters
Publication Identifier Type: DOI Publication Identifier: 10.1063/1.5092984
Volume: 114 Issue: 19 First Page #: 194101
Date Submitted: 7/24/19 12:00AM Date Published: 5/1/19 5:00AM
Publication Location:
Article Title: Dynamic absorption in optical pyrometry of hot spots in plastic-bonded triaminotrinitrobenzene
Authors: Will P. Bassett, Belinda P. Johnson, Dana D. Dlott
Keywords: Optical pyrometry, shock initiation, TATB
Abstract: We have measured emission spectra from a plastic-bonded formulation of triamino trinitrobenzene (TATB) shocked by 2-4 km/s impacts with Al flyer plates. These spectra show significant deviations from graybody behavior. To extract reliable temperatures via optical pyrometry, we fit the spectra to a combination of graybody and either a Gaussian absorption or emission spectrum. We found that the absorption needed to fit the data corresponds well with the known absorption of the yellow TATB, and that the absorption model gives temperatures and emissivities in line with other explosives. By contrast, emission gives temperatures too low and emissivities that decrease as more material reacts. We conclude the nonthermal part of the spectrum is dominated by the absorption of unreacted TATB, and accurate pyrometry of TATB must use either our graybody plus absorption model or must limit the spectral range of observation to above 700 nm.
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Publication Type: Journal Article Peer Reviewed: Y **Publication Status:** 1-Published
Journal: Journal of Applied Physics
Publication Identifier Type: DOI Publication Identifier: 10.1063/1.5098476
Volume: 125 Issue: 21 First Page #: 215904
Date Submitted: 7/24/19 12:00AM Date Published: 6/1/19 5:00AM
Publication Location:
Article Title: Hot-spot generation and growth in shocked plastic-bonded explosives studied by optical pyrometry
Authors: Will P. Bassett, Belinda P. Johnson, Lawrence Salvati, Dana D. Dlott
Keywords: shock compression, plastic-bonded explosives, hot spots, PETN
Abstract: The aggregate behavior of hot spots in shocked plastic-bonded explosives (PBX) was studied by nanosecond optical pyrometry. The averaged thermal emission spectra from at least 25 tiny (50 ?g) explosive charges of a PETN (pentaerythritol tetranitrate) PBX, at several impact velocities from 1.5 to 4.5 km/s, was used to determine average temperatures and emissivities. Individual spectra were analyzed to determine the distribution of hot spot temperatures in individual charges with unique microstructures. Understanding shocks in tiny charges with different microstructures is needed to understand shocks in large PBX charges which sample many microstructures as they propagate. The initial hot spot density was several percent, and the average initial hot spot temperature of 4000K was, surprisingly, independent of impact velocity. With underdriven shocks, the initial hot spot temperatures clustered around 4000K, but with overdriven shocks, there were both hotter and colder hot spots. Th
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Publication Type: Journal Article Peer Reviewed: Y **Publication Status:** 1-Published
Journal: AIP Confer. Proc.
Publication Identifier Type: DOI Publication Identifier: https://doi.org/10.1063/12.0001019
Volume: 2272 Issue: First Page #: 060009
Date Submitted: 5/10/22 12:00AM Date Published: 11/4/20 6:00AM
Publication Location:
Article Title: Shock compression microscopy: Shocked materials with high time and space resolution
Authors: Dana D. Dlott, Meysam Akhtar, Will P. Bassett, Mithun Bhowmick, Belinda P. Johnson, Sergey Matveev
Keywords: shock compression, energetic materials
Abstract: The tabletop shock compression microscope is described, along with some recent applications to problems such as the temperature of shocked TATB (1,3,5 triamino 2,4,6 tri nitrobenzene), hot spots in HMX (cyclotetramethylene-tetranitramine) and detonation on a tabletop in nitromethane (NM).
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Publication Identifier Type: DOI Publication Identifier: 10.1063/12.0000960
Volume: 2272 Issue: First Page #: 030036
Date Submitted: 8/13/21 12:00AM Date Published:
Publication Location:
Article Title: Shock compression dynamics of PETN/TATB explosive charges
Authors: Wei Zhang, Will P. Bassett, Meysam Akhtar, Lawrence Salvati III and Dana D. Dlott
Keywords: shock initiation, TATB, optical pyrometry
Abstract: We developed a convenient method to mass produce arrays of 1 mm diameter double-layer plastic-bonded explosive (PBX) charges for tabletop studies of shocked insensitive explosives. In the double layer, a pentaerithritol tetranitrate (PETN) booster charge 100 ?m long is initiated by a laser-launched flyer plate. The booster sends a shock into a 40 ?m long 1,3,5 triamino-2,4,6 trinitrobenzene (TATB) charge. The TATB is initiated more efficiently by the booster charge than by the flyer plate alone. The temperature of the shocked TATB was measured with 2 ns time resolution using optical pyrometry. We could monitor the decomposition of the shocked TATB via the disappearance of its blue-green absorption, and we could monitor the volume explosion by the resulting fast adiabatic cooling.
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Volume: 2272 Issue: First Page #: 030024
Date Submitted: 5/10/22 12:00AM Date Published: 11/4/20 6:00AM
Publication Location:
Article Title: Imaging the Reactive Flow Structure in Shocked Nitromethane and Nitromethane with Additives
Authors: Erin J. Nissen, Mithun Bhowmick and Dana D. Dlott
Keywords: shock to detonation, nitromethane, cellular structure
Abstract: We used table-top laser driven flyer plates to generate planar shock waves in tiny cuvettes to produce detonations in liquid nitromethane (NM), and NM with sensitizing, desensitizing and inert additives. The detonations were probed by a combination of high-speed video and photon Doppler velocimetry (PDV). The thermal emission revealed cellular structures in the detonation front, whose size was dependent on the type of additive. Since there is a close relationship between these structures and the reaction zone, this technique may prove useful in determining how the reaction zone length is affected by additives. We considered the possibility that these structures arose from similar structures in the initiating shock using Al or steel lids on the entry face of the cuvettes with different roughnesses, which should introduce similar structures in the initiating shock, we showed that the structures we observe are a property of the reactive flow in NM itself.
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Publication Identifier Type: DOI Publication Identifier: https://doi.org/10.1063/12.0001021
Volume: 2272 Issue: First Page #: 030027
Date Submitted: 5/10/22 12:00AM Date Published: 11/4/20 6:00AM
Publication Location:
Article Title: Probing Shock-Initiation of Plastic-Bonded Explosives With a Tabletop Microscope
Authors: Lawrence Salvati III, Belinda P. Johnson, William P. Bassett, Dana D. Dlott
Keywords: shock initiation, PETN, shock to detonation
Abstract: In this study we investigate the shock-to-detonation transition (SDT) in a plastic-bonded explosive (PBX) consisting of pentaerithritol tetranitrate (PETN) with poly-dimethyl siloxane (PDMS) binder. The study uses a tabletop shock compression microscope with laser-driven flyer plates and arrays of tiny PBX charges. The PBX was shocked by 4 ns duration 0.5-4.5 km/s impacts and probed using photon Doppler velocimetry (PDV) and optical pyrometry. The PBX was efficiently initiated and hot spots were produced, but the entire PBX was not ignited and detonation was not achieved. Possible methods to achieve detonation over short run distances <0.25 mm were discussed.
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Volume: 45 Issue: 2 First Page #: 338
Date Submitted: 7/24/20 12:00AM Date Published: 11/1/19 5:00AM
Publication Location:
Article Title: Hot Spot Chemistry in Several Polymer-Bound Explosives under Nanosecond Shock Conditions
Authors: Will P. Bassett, Belinda P. Johnson, Dana D. Dlott
Keywords: Plastic explosives, pyrometry, hot spots
Abstract: Initial hot spot temperatures and temperature evolutions for 4 polymer-bound explosives under shock compression by laser-driven flyer plates at speeds from 1.5– 4.5 kms?? 1 are presented. A new averaging routine allows for improved signal to noise in shock compressed impactor experiments and yields temperature dynamics which are more accurate than has been previously available. The PBX formulations studied here consist of either pentaerythritol tetranitrate (PETN), 1,3,5-trinitro-1,3,5-triazinane (RDX), 2,4,6-trinitrotoluene (TNT), or 1,3,5-triamino-2,4,6-trinitrobenzene (TATB) in a 80/20 wt.% mixture with a silicone elastomer binder. The temperature dynamics demonstrate a unique shock strength dependence for each base explosive. The initial hot spot temperature and its evolution in time are shown to be indicative of chemistry occurring within the reaction zone of the four explosives. The number density of hot spots is qualitatively inferred from the spatially- averaged emissivity and
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Journal: Propellants, Explosives and Pyrotechnics
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Volume: 45 Issue: First Page #: 223
Date Submitted: 7/24/20 12:00AM Date Published: 1/24/20 6:00AM
Publication Location:
Article Title: Shock initiation microscopy with high time and space resolution
Authors: Will P. Bassett, Belinda P. Johnson, Lawrence Salvati III, Dana D. Dlott
Keywords: Shock initiation, pyrometry, microscopy, plastic-bonded explosives
Abstract: We describe studies of shock initiation and shock-to-detonation transitions in energetic materials using a tabletop shock compression microscope with nanosecond time resolution and micrometer spatial resolution. Planar input shocks with durations of 4-20 ns are produced using 0-4.5 km/s laser-launched flyer plates. Emphasis is on measurements of temperature, velocities, pressure and microstructure using photon Doppler velocimetry (PDV), optical pyrometry and high-speed videography. Techniques are discussed for fabricating disposable shock target arrays of tiny plastic-bonded explosives (PBX), liquid and powder explosives, and single-crystal explosives for high-throughput studies. Optical temperature measurements of shocked triaminotrinitrobenzene (TATB) are discussed. Since TATB is yellow, we developed methods to correct for the blue absorption to obtain more accurate temperatures. Hot spots in shocked polymer-encased HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine) crystals are
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Journal: Applied Physics Letters
Publication Identifier Type: DOI Publication Identifier: 10.1063/1.5145216
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Date Submitted: 8/26/20 12:00AM Date Published: 3/1/20 6:00PM
Publication Location:
Article Title: Shock initiation and hot spots in plastic-bonded 1,3,5-triamino-2,4,6-trinitrobenzene &;#x28;TATB)
Authors: Wei Zhang, Lawrence Salvati, Meysam Akhtar, Dana D. Dlott
Keywords: Shock initiation, TATB, hot spots
Abstract: TATB (1,3,5-triamino-2,4,6-trinitrobenzene) is a powerful explosive whose dynamical behavior is difficult to study because TATB is so insensitive to initiation by shock waves. We used a tabletop microscope equipped with 0–4.5 km/s laser-launched flyer plates to study shock initiation of TATB, which was fabricated in the form of an array of hundreds of plastic-bonded explosive minicharges (X-TATB¼80% TATBþ20% Sylgard 182 polymer). The 4 ns shocks from the flyer plates were not effective in initiating TATB, but we also developed a twolayer array where flyers first initiated a plastic-bonded PETN (pentaerythritol tetranitrate) charge (X-PETN¼80% PETNþ20% Sylgard), which drove an initiating 25 ns shock into the X-TATB. Thermal emission from shocked X-TATB was used to measure time-dependent temperature profiles with a resolution of 2 ns and to produce high-speed (5 ns) videos. In X-TATB, flyer plates produced 2500–3500 K hot spots and combustion at 2500 K. With X-PETN initiators, X-TATB had
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Journal: The Journal of Physical Chemistry A
Publication Identifier Type: DOI Publication Identifier: 10.1021/acs.jpca.0c02788
Volume: 124 Issue: 23 First Page #: 4646
Date Submitted: 7/24/20 12:00AM Date Published: 5/1/20 5:00AM
Publication Location:
Article Title: Observing Hot Spot Formation in Individual Explosive Crystals Under Shock Compression
Authors: Belinda P. Johnson, Xuan Zhou, Hoya Ihara, Dana D. Dlott
Keywords: HMX, hot spots, single crystal
Abstract: The formation of hot spots in dynamically compressed, plasticbonded explosives is known to be the primary mechanism by which these materials ignite and initiate, but hot spots are small, fleeting, and hard to observe. Using a microscope equipped with laser-launched, miniflyer plates, we have studied hot spots in small grains of cyclotetramethylene-tetranitramine (HMX) embedded in a polyurethane binder, shocked to about 20 GPa. A nanosecond video with 4 ?m spatial resolution is used to observe hot spot formation and growth, while nanosecond optical pyrometry measured temperature. Using individual ?200 ?m nominally single crystals of HMX (HMX-SC), we observed hot spots forming preferentially on corners or edges. These hot spots are about 4000 K. When there are multiple hot spots, the flame propagated along crystal edges, and the crystal is mostly combusted after about 300 ns. Using polycrystalline grains (HMX-PC), 6000 K hot spots are created near internal defects or crystal junctions. H
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Journal: Combustion and Flame
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Volume: 225 Issue: First Page #: 5
Date Submitted: 8/13/21 12:00AM Date Published:
Publication Location:
Article Title: Shock-induced chemical kinetics of nitromethane detonation
Authors: Erin K. Nissen, Mithun Bhowmick and Dana D. Dlott
Keywords: shock to detonation, cellular structure, nitromethane
Abstract: Using a combination of high-speed diagnostics, optical pyrometry, velocimetry and video photography, we examined the spatiotemporal reaction kinetics of a prototypical high-energy explosive, liquid nitromethane (NM), as we drive this high explosive into detonation. The detonations were initiated by powerful shock waves whose durations (4 ns) were shorter than the characteristic time associated with the reaction (7 ns). Simple optical spectroscopy alone cannot characterize the kinetics with high time resolution because the reactants and products are flowing at a high velocity of ~6 km/s (6 ?m/ns). Every detection volume behind the shock front contains material initiated over a range of times. High spatial resolution can be obtained by probing interfaces where the shock enters or breaks out of the NM, and we show in addition that we can obtain high spatial resolution via cellular patterns imprinted in the luminous shock front by the two-stage explosion. These spatiotemporal patterns
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Acknowledged Federal Support: **Y**

Publication Type: Journal Article Peer Reviewed: Y **Publication Status:** 1-Published
Journal: Applied Physics Letters
Publication Identifier Type: DOI Publication Identifier: 10.1063/5.0043586
Volume: 118 Issue: 10 First Page #: 101902
Date Submitted: 8/13/21 12:00AM Date Published: 3/2/21 12:00AM
Publication Location:
Article Title: Fast energy release from reactive materials under shock compression
Authors: Sergey Matveev, Dana D. Dlott, Siva Kumar Valluri, Mehnaz Mursalat, Edward L. Dreizin
Keywords: reactive nanomaterials, shock initiation
Abstract: Metal-based reactive nanomaterials (RNMs) can produce two to three times more energy than conventional organic explosives, but the exothermic reactions, which ordinarily require diffusive mixing of separated fuel and oxidizer components, are too slow and do not generate enough gas to produce detonations. Here we studied shock initiation of 4Al/Bi2O3 and 4Al/BiF3 RNMs produced by arrested reactive milling. Initiated by a 3 km/s impact, which approximates a powerful detonation, fast energy release produced 3200 K temperatures. In the fluoride, a rapid volume increase was also observed. The shock-induced energy release was orders of magnitude faster than when the RNM was heated. Although these RNM powders by themselves likely cannot produce detonations, our results suggest that used as additives in detonating systems, they might significantly boost the energy of chemical explosives.
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Journal: Journal of Applied Physics
Publication Identifier Type: DOI Publication Identifier: 10.1063/5.0049817
Volume: 129 Issue: 19 First Page #: 195901
Date Submitted: 8/13/21 12:00AM Date Published: 5/1/21 5:00AM
Publication Location:
Article Title: Laser-driven flyer plate impact: Computational studies guided by experiments
Authors: Svjetlana Stekovic, H. Keo Springer, Mithun Bhowmick, Dana D. Dlott, D. Scott Stewart
Keywords: flyer plate, laser launch, high velocity impact, ALE3D
Abstract: We present a computational approach using a multi-material, arbitrary Lagrangian-Eulerian code termed ALE3D to model the nanosecond/micrometer dynamics of the launch of 0.5-4.5 km/s laser-driven metal flyer plates and the impact with stationary targets of Pyrex and fused silica glasses, and Lexan and Plexiglas polymers, producing pressures in the target in the 5-20 GPa range. The simulations are compared to experimental results where the flyer velocity profile and the velocity profile imparted to the target material were measured with high-speed velocimetry. The experimental flyer launch by a high-intensity pulsed laser is modeled by depositing heat into a thin vaporizable layer under the flyer plate. This model produces a flyer plate that has not been exposed to the laser pulse, allowing us to compare the properties of the real flyer to a simulated ideal flyer. Simulations of target impact are generally in good agreement with experiment except at the highest impact velocities wher
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Journal: The Journal of Physical Chemistry B
Publication Identifier Type: DOI Publication Identifier: 10.1021/acs.jpcb.1c04427
Volume: 125 Issue: 29 First Page #: 8185
Date Submitted: 8/13/21 12:00AM Date Published: 7/1/21 5:00AM
Publication Location:
Article Title: Ethylenediamine Catalyzes Nitromethane Shock-to-Detonation in Two Distinct Ways
Authors: Erin J. Nissen, Mithun Bhowmick, Dana D. Dlott
Keywords: detonation, shock to detonation transition, nitromethane, amine sensitized
Abstract: Adding amines to liquid nitromethane (NM) is known to lower the threshold for the shock-to-detonation transition because amines catalyze proton transfer reactions that are the initial steps in the energy release process. We studied NM with 1 wt.% ethylenediamine (NM/EDA) with 4 ns input shocks using time and space resolved diagnostics: photon Doppler velocimetry (PDV), optical pyrometry and nanosecond video imaging. The 4 ns shocks are fast enough to time-resolve the reaction kinetics and the shock-to-detonation transition. We find that it is possible to shock ignite the NM/EDA without producing a detonation, so there is more to amine sensitization of the shock-to-detonation process than simply lowering the barrier to initial reactions. We find that although 1 wt.% EDA has little effect on the ambient properties of NM, it dramatically alters the Hugoniot. The shock speed in NM/EDA is reduced, indicating that shocked NM/EDA is significantly more compressible than NM. Higher compre
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Publication Identifier Type: DOI Publication Identifier: 10.1039/D2CP00418F
Volume: 24 Issue: First Page #: 10653
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Publication Location:
Article Title: Laser pulses into bullets: tabletop shock experiments
Authors: Dana D. Dlott
Keywords: energetic materials, hypervelocity, flyer plates, microscopy
Abstract: This article discusses tabletop high-throughput laser experiments on shock waves in solids and liquids, where the more usual laser pump pulse is replaced by a 0.5 mm diameter laser-launched bullet, a thin metal disk called a flyer plate. The hypervelocity flyer (up to 6 km s┐1 or Mach 18) can have kinetic energy (B1 J) to briefly produce extreme conditions of temperature and pressure, thousands of K and tens of GPa (1 GPa = 10 000 bar) in a small volume with a rise time o2 ns. The experiments are performed using a ‘‘shock compression microscope’’, a microscope fitted with the laser flyer launcher plus an optical velocimeter, a high-speed laser interferometer that measures the motion of the flyer plate or the sample material after impact. This makes it possible to generate extreme conditions at the push of a button in an intrinsically safe environment, and probe with any of the diagnostics used in microscope experiments, such as high-speed video, optical emission, nonlinear coherent spe
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Date Submitted: 5/10/22 12:00AM Date Published: 2/16/22 6:00AM
Publication Location:
Article Title: High throughput tabletop shock techniques and measurements
Authors: Fabing Li, Dana D. Dlott
Keywords: flyer plates, optical emission, high throughput
Abstract: Although shock experiments are traditionally performed in large facilities, tabletop experiments that provide convenient high-throughput shock testing have been growing in importance. Here, we describe tabletop experiments using a shock compression microscope that features a pulsed 0–6 km/s laser flyer plate launcher and a photon Doppler velocimeter. We also describe methods to mass-produce flyer plates and targets to achieve high throughput. We explain how to condition a laser beam to launch flyers that provide reproducible short-rise time impacts with minimal tilt, and we present a number of applications including measuring shock propagation in nanoporous media, a simple way to describe shock wave energy absorption, the use of photoemissive probes such as organic dyes or quantum dots to study shocked inhomogeneous media, the development of an apparatus to measure optical absorption in shocked media, methods to study and measure the temperature of shocked energetic materials in the fo
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Publication Location:
Article Title: Comparing the shock sensitivity of insensitive energetic materials
Authors: Meysam Akhtar, Dana D. Dlott
Keywords: energetic materials, shock, insensitive
Abstract: We present a tabletop method to study the shock sensitivity of plastic-bonded explosives that are considered shock insensitive using high dynamic range optical emission spectroscopy with laser-launched km/s flyer plate impacts (2, 3, and 4 km/s), which measures the spectral radiance (the emission spectrum vs a calibrated intensity standard) every 0.8 ns in the nanosecond and microsecond regimes. The explosives were TATB (1,3,5 trinitro, 2,4,6 triamino benzene), FOX-7 (1,1-diamino-2,2-dinitroethylene), LLM-105 (2,6-diamino-3,5-dinitropyrazine-1-oxide), and NTO (nitrotriazolone), all with 20% Sylgard 182 binder. Time resolving emission from shocked explosives produces a unique fingerprint at each shock pressure, and the emission temporal profile can be used to understand each material's shock response and the underlying mechanisms within the framework of the well-known shock ignition and growth models. Ignition was characterized by the emission intensity during nanosecond hot spot igniti
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Article Title: measurements of high explosives
Authors: Dhanalakshmi Sellan, Xuan Zhou, Lawrence Salvati, Siva Kumar Valluri, Dana D. Dlott
Keywords: high explosives, spectroscopy, plastic-bonded explosives, nitromethane
Abstract: In operando studies of high explosives involve dynamic extreme conditions produced as a shock wave travels through the explosive to produce a detonation. Here we describe a method to safely produce detonations and dynamic extreme conditions in high explosives and in inert solids and liquids on a tabletop, in a high-throughput format. This method uses a shock compression microscope, a microscope with a pulsed laser that can launch a hypervelocity flyer plate along with a velocimeter, an optical pyrometer, and a nanosecond camera that together can measure pressures, densities and temperatures with high time and space resolution (2 ns and 2 ?m). We discuss how a detonation builds up in liquid nitromethane and show that we can produce and study detonations in sample volumes close to the theoretical minimum. We then discuss how a detonation builds up from a shock in a plastic-bonded explosive (PBX) based on HMX (1,3,5,7-Tetranitro-1,3,5,7-tetrazocane), where the initial steps are hot s
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Authors: Erin J. Nissen
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Authors: Belinda P. Johnson
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Partners

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I certify that the information in the report is complete and accurate:
Signature: Dana D. Dlott
Signature Date: 7/11/23 2:39PM

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"Detonation on a Tabletop"

Executive summary

Detonation on a tabletop refers to experimental studies in the Dlott group at the University of Illinois that involve using a pulsed laser to launch small hypervelocity flyer plates at tiny charges of various explosives while diagnostics with high time and space resolution probe the explosive response. There were four main thrust areas with corresponding publications listed below. These were: (1) Experimental advances; (2) Hot spots and detonations in plastic-bonded explosives (PBX); (3) Shocked reactive materials. The high points were: (1) Improved methods for temperature measurements; (2) Multimaterial simulations of laser-launched flyer plate launch and impact; (3) Tabletop detonations produced in nitromethane (NM) and NM with amine catalyst and visualization of novel cellular structures; (4) Hot spots dynamics in several PBX; (5) Hot spot dynamics visualized in single and poly crystals of HMX; (5) Fast energy release in reactive materials.

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Experimental advances

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Dana D. Dlott, Meysam Akhtar, Will P. Bassett, Mithun Bhowmick, Belinda P. Johnson, Sergey Matveev, Erin J. Nissen, Lawrence Salvati III, Svjetlana Stekovic, Wei Zhang and Xuan Zhou, Shock compression microscopy: Shocked materials with high time and space resolution. AIP Confer. Proc. AIP Confer. Proc. 2272, 060009 (2020). https://doi.org/10.1063/12.0001019.

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"Theory and modeling of laser-driven flyer plate experiments to study explosives under a microscope", PhD thesis of Svjetlana Stekovic, July 2021.

Nitromethane detonation

Erin J. Nissen, Mithun Bhowmick and Dana D. Dlott, Imaging the Reactive Flow Structure in Shocked Nitromethane and Nitromethane with Additives, AIP Confer. Proc. 2272. 030024 (2020). https://doi.org/10.1063/12.0000910.

Shock-induced chemical kinetics of nitromethane detonation, Erin K. Nissen, Mithun Bhowmick and Dana D. Dlott, Combust. Flame (submitted)Shock-induced kinetics and cellular structures of liquid nitromethane detonation, Erin K. Nissen, Mithun Bhowmick and Dana D. Dlott, Combust. Flame. 225, 5-12 (2021). 10.1016/j.combustflame.2020.10.046.

"Ethylenediamine Catalyzes Nitromethane Shock-to-Detonation In Two Distinct Processes," Erin J. Nissen, Mithun Bhowmick, and Dana D. Dlott, J Phys Chem B, 125 pp. 8185-8192 (2021). DOI: 10.1021/acs.jpcb.1c04427.

"Shock compressing liquids on a tabletop microscope with special focus on detonating nitromethane", PhD thesis of Erin J. Nissen, June 2021.

Hot spots

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Observing Hot Spot Formation in Individual Explosive Crystals Under Shock Compression, Belinda P. Johnson, Xuan Zhou, Hoya Ihara, and Dana D. Dlott. J. Phys. Chem. A. 124, 4646-4653 (2020).

"Tracking hot spot dynamics in solid state explosives with shock compression microscopy", PhD thesis of Belinda P. Johnson, July 2021.

Shocked reactive materials

"Fast Energy Release From Reactive Materials Under Shock Compression", Sergey Matveev, Dana D. Dlott, Siva Kumar Valluri, Mehnaz Mursalat and Edward L. Dreizin, Appl. Phys. Lett. 118, 101902 (2021) DOI: 10.1063/5.0043586.

Reviews

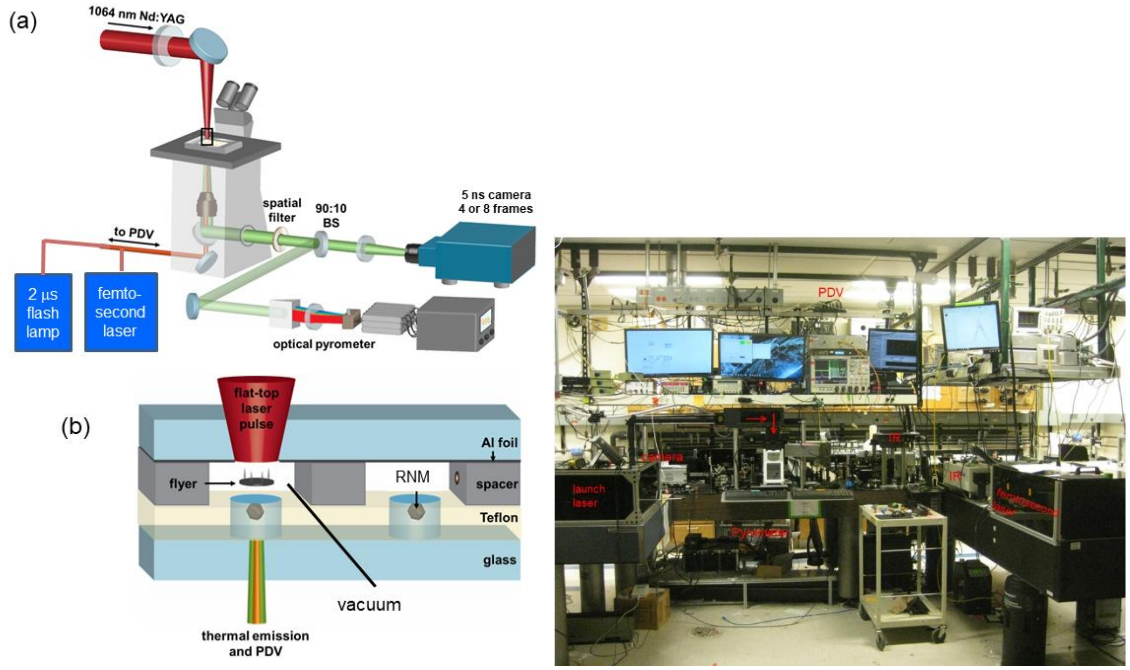
F. Li and D. D. Dlott, High throughput tabletop shock techniques and measurements, Journal of Applied Physics 131, 075901 (2022). https://doi.org/10.1063/5.0083651

(Invited Perspective) "Laser pulses into bullets: Tabletop shock experiments", Dana D. Dlott, Physical Chemistry Chemical Physics 24, 10653-10666 (2022) DOI: 10.1039/d2cp00418f

"In Operando Measurements of High Explosives", D. Sellan, X. Zhou, L. Salvati III, S. K. Valluri, and D. D. Dlott, J. Chem. Phys 157, 224202 (2022).

Extended narrative

The Dlott lab shock compression microscope system[1,2] is diagrammed in Fig. 1 and a photo is shown in Fig. 2. It consists of a tabletop pulsed laser that launches small hypervelocity disks, "flyer plates",[3] a photon Doppler velocimeter (PDV) to measure flyer and material velocities,[4-6] a visible-light 32-channel optical pyrometer[7] and a nanosecond camera. A variety of other light sources including a femtosecond visible and infrared laser, several nanosecond lasers and a microsecond flash lamp are also available that are used depending on the application. In order to conduct high-throughput tabletop measurements, we developed methods to mass-produce arrays of tiny PBX charges or liquid microcuvettes.[8-10]



Figures 1 and 2. (left) Schematic of shock microscope and sample array. (right) Photo of shock microscope.

Experimental advances

Early in the project we were contacted by Dr. Lara Leininger, who is the director of the DOE Energetic Materials Center located within Lawrence Livermore National Laboratory, who wanted to adopt our pyrometry techniques and fund some research to study TATB, which is a most unusual energetic material (EM) of particular interest to the DOE.[11] Optical pyrometry measures the spectral radiance of thermal emission and temperatures can be determined by fitting the spectral radiance to a graybody model devised by Planck.[12,13]

TATB presented a new challenge because it is yellow and absorbs blue light,[14] as shown in Figs. 3a,b. Consequently the spectral radiance of the thermal emission that pyrometers use to deduce temperatures was greatly distorted by the blue absorption of the TATB (Fig. 3c). We developed a couple of techniques to overcome this distortion and a corrected temperature history

of a PBX consisting of 80% TATB with a 3 km/s impact is shown in Fig. 3d where the hot spots are 3300K and the adiabatic flame temperature is 2250K.

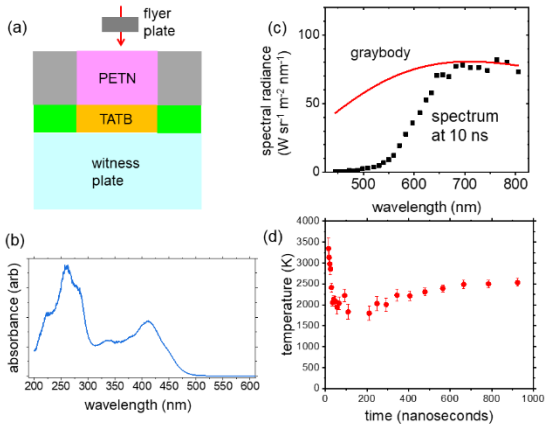


Figure 3. (a) Schematic of two-layer PBX. (b) Reflection-absorption spectrum of TATB PBX. (c) Thermal emission showing effects of TATB blue absorption. (d) Temperature profile with 3 km/s impact corrected for blue absorption.

A serious problem with pyrometric determinations of temperature, that can result in temperature errors of thousands of K, occurs when there are atomic line or molecular band emissions on top of the thermal emission, or if that emission is distorted by the presence of visible-absorbing material, as with TATB. Our solution to this problem was to identify troublesome regions in the spectral radiance and exclude them from our temperature analysis.

Wien's high-temperature approximation for the graybody law is,[13]

$$S(T,\lambda) = \varepsilon C_1/\lambda^5 \exp(C_2/\lambda T), \qquad (1)$$

Where ε is the graybody emissivity, $C_1 = 3.72 \times 10^{-16}$ W/m^2 and $C_2 = 1.439 \times 10^{-2}$ m·K.[15]

Equation (1) can be reformulated to yield,

$$Z = \ln\left(\frac{C_1}{\lambda^5}\left[S(\lambda,T)\right]^{-1}\right) = \frac{C_2}{\lambda T} - \ln(\lambda\varepsilon). \qquad (2)$$

Equation 2 shows that if we plot the function Z versus $1/\lambda$, a linear function will be obtained with a slope inversely proportional to T. For convenience we call this method the "Z-plot" method.[16] Examining Z-plots for deviations from linearity is a convenient way of identifying deviations from graybody thermal emission, and it allows us to select which regions of the emission spectrum should be used to determine graybody temperatures.

Figure 4 is a Z-plot for a PBX formulated from HMX which is colorless, with a 3 km/s impact. The Z-plot is linear from 1 ns to 150 μs so the detected emission is thermal at all times. But Fig. 5 is the corresponding plot for TATB and a severe deviation from linearity is observed at shorter times. Only with the higher impact velocities and at longer times is simple linear behavior observed. This is understandable because when yellow TATB is shocked to reaction the yellow parent material disappears and colorless products are produced.[14]

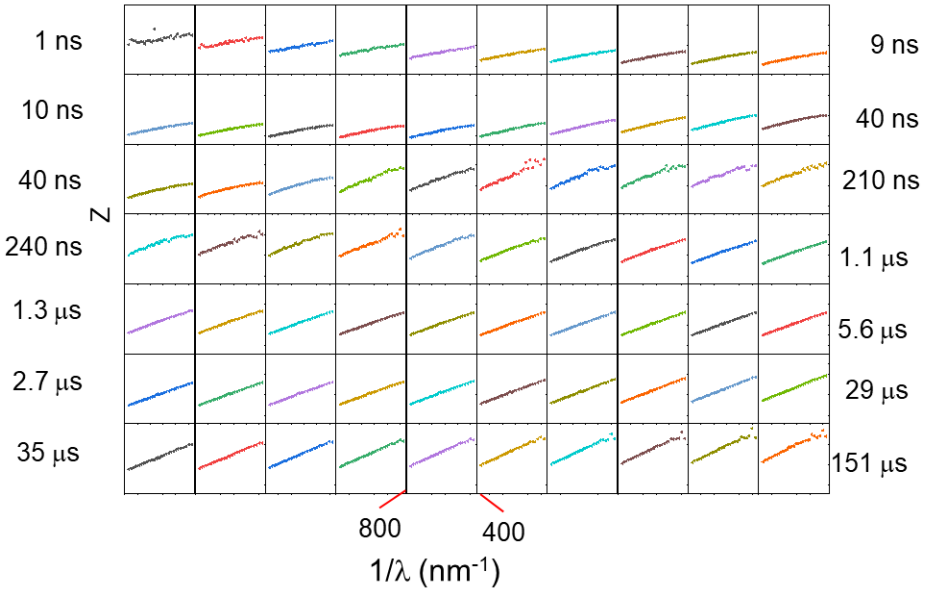


Figure 4. Z-plot for HMX PBX with 3 km/s impact. The emission is purely thermal at all times as indicated by linear spectral radiances.

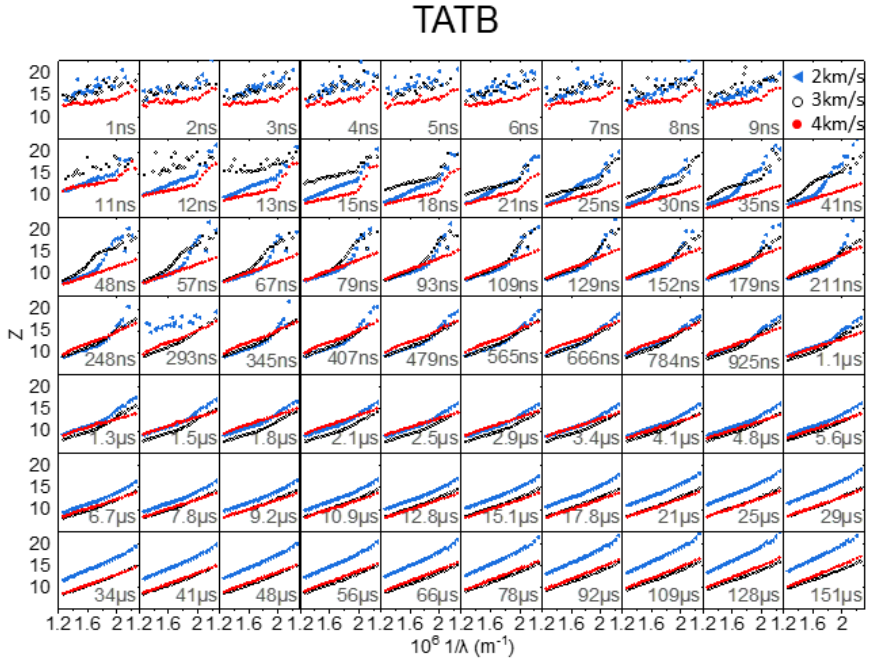


Figure 5. Z-plot for TATB PBX at three impact velocities. Effects of blue absorption result in deviations from linearity most prominent with lower velocity impacts and at longer times.

One way to get rid of the yellow material is to hit the TATB with a shock so powerful that the yellow material disappears as quickly as possible. TATB is quite insensitive, and difficult to ignite with shock, so we developed the method described in Fig. 6 where we developed a two-layer geometry[10,17] where the flyer plate drives a shock into a small Cl-20 charge. Cl-20 is a powerful explosive that is more sensitive to shock than TATB so it produces a more powerful,

longer duration shock in TATB than flyer plate alone. A temperature history of a TATB-PBX where the flyer plate velocity was varied from 1-4.2 km/s and the TATB was shocked by Cl-20 is also shown in Fig. 6.

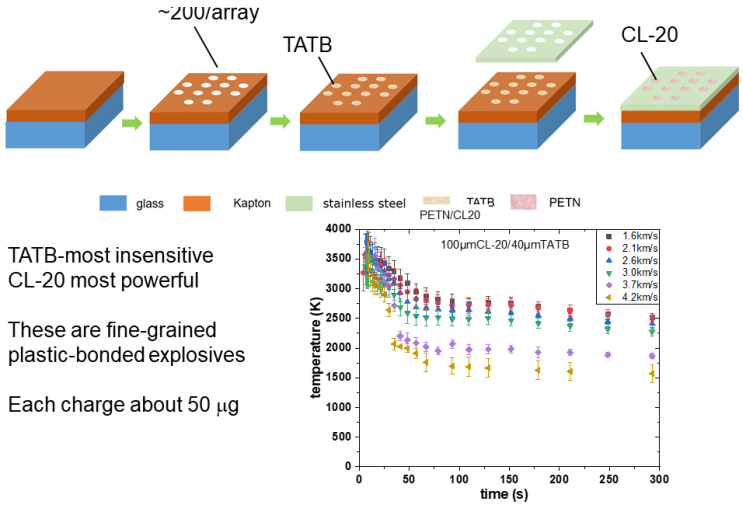


Figure 6. (top) Schematic of two-layer arrangement using Cl-20 booster to shock TATB. (bottom) Temperature history of TATB with various impact velocities.

In the past we used photoemissive dyes and nanoparticles that can register local temperatures and pressures in shocked microstructures.[2] However shocking these emitters generally resulted in a large decrease in quantum yield because the shock deactivates the emitting singlet state via intersystem crossing to triplet states. This observation, along with the problems associated with TATB absorption led us to develop a shock optical absorption technique. This technique was tested using the photoemissive dye Rhodamine 6G (R6G) in ethanol,[2] as shown in Fig. 7. The dye was excited by a long-duration green laser pulse during the shock. As shown in Fig. 7a, with a 3.3 km/s impact, the photoemission drops to practically zero but the absorption can easily be measured at all times. In addition a new transition appeared associated with proton transfer from ethanol to R6G which demonstrates that we can probe shock-induced chemistry in inert liquids.

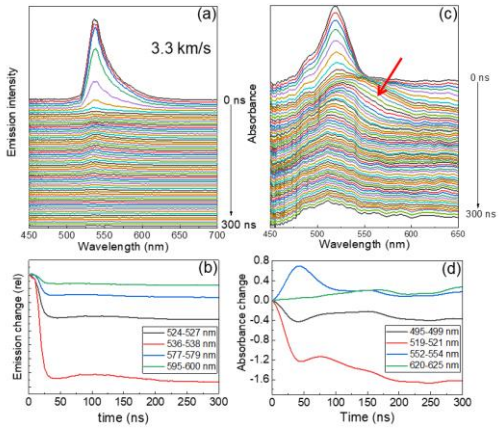


Figure 7. Emission and absorption spectra of R6G dye in ethanol with 3.3 km/s impact.

My colleague Prof. Scott Stewart in Mechanical Engineering, and I collaborated to model the laser launch of flyer plates and their impacts with flat plates of various materials. We mentored a joint PhD student Svetlana (Lana) Stekovic for this project. Lana did an internship at Livermore under the mentorship of Dr. Keo Springer, where she learned how to use the multimaterial multiphysics shock code ALE3D. Flyer plates are launched from metal foils epoxied to glass substrates, where an intense laser pulse deposits energy at the foil/glass interface (Fig. 8). In order to avoid the complexity of modeling the electromagnetic interactions, we simplified the problem by assuming the laser pulse (shown in Fig. 8a) was assumed to heat and vaporize the epoxy, since an epoxy equation of state (EOS) was built into ALE3D.[18-20] Figure 9 shows that using this method we were able to closely approximate the velocity of Al flyers as a function of laser energy with a single adjustable parameter that characterized the strength of the optical absorption in the epoxy.

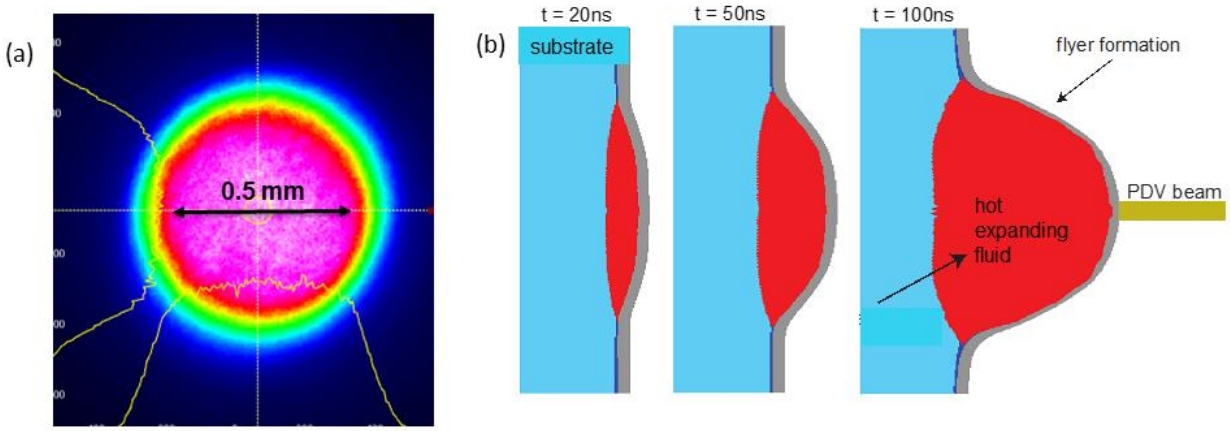


Figure 9. (a) Profile of launch laser beam. (b) Model for flyer launch probed by PDV.

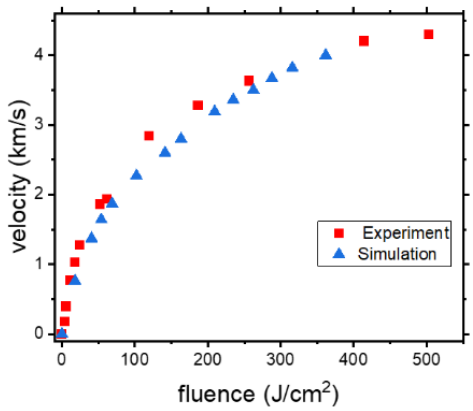


Figure 9. Predicted and experimental flyer velocities agree using a single adjustable parameter that characterizes the strength of nonlinear optical absorption of the interfacial epoxy.

1In Fig. 10, we used measured laser pulse characteristics to predict the acceleration profile of Al flyers with various laser pulses. Agreement is excellent except the simulation predicts a small reverberating shock in the flyer plate not evident in experiment. We do not see much effect of the reverberation because in the simulation the flyer is a perfect interferometer whereas in experiment is it an imperfect polycrystalline film where the reverberations damp out quickly.

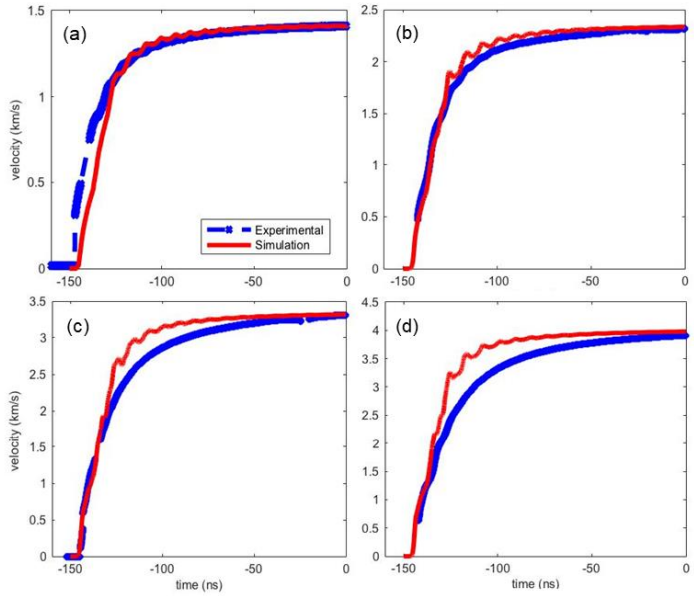


Figure 10. Predicted and measured flyer acceleration profiles.

We also modeled the impact of flyer plates with Pyrex glass at velocities of 1.36 km/s, 2.35 km/s, 3.36 km/s, and 4.00 km/s, as shown in Fig. 11.[20] Although general agreement is very good, there is a long tail in the velocity decay that was not predicted in the simulation. We believe this is a failure in the constitutive model we used for the Pyrex.

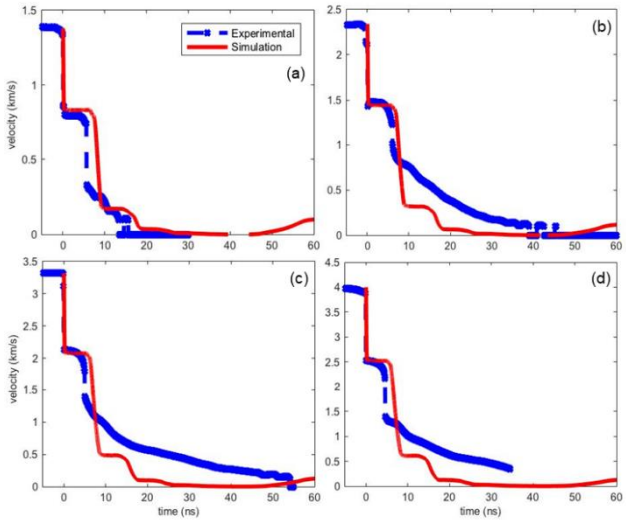


Figure 11. Predicted and measured velocity profiles for flyer late hitting Pyrex glass at velocities of 1, 2, 3 and 4 km/s.

Finally, we modeled our nitromethane (NM) experiments using a simple single-step kinetic model for NM reactive kinetics. Figure 12 shows velocity profiles observed at various distances downstream. Although agreement was good, there are oscillations in the simulation that are

smoothed out in experiment. The remaining disagreements are due to using a single-step kinetic model.

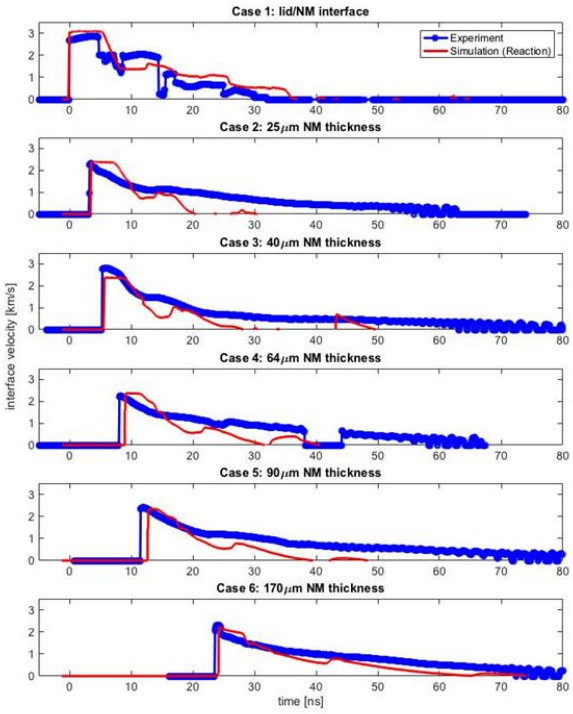


Figure 12. Predicted and measured nitromethane velocity profiles (breakout into glass window) at various distances downstream.

Nitromethane detonation

Nitromethane is a venerable model system for detonation in a homogeneous explosive.[21] We were able to produce detonations in NM with a very short run distance of about 60 μm or equivalently about 12 ns.[9] Figure 13 shows how a shock evolves in NM as it runs to detonation. Due to the small sample volume this is an unsteady shock that decays after about 50 ns, which still gives us plenty of time to study the NM.

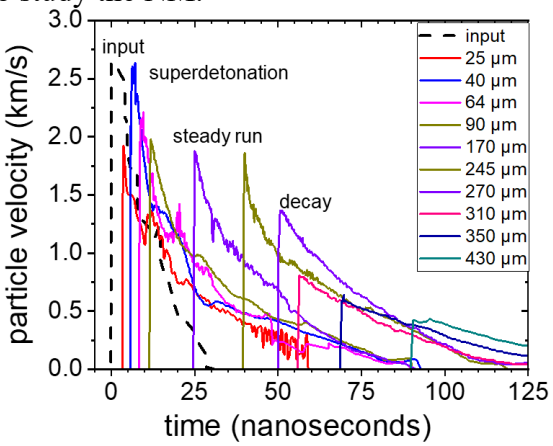


Figure 13. Nitromethane shock waveforms at different distances downstream showing run to detonation.

After we demonstrated detonation in NM, we acquired a nanosecond camera through the DURIP program that allowed us to see inside the NM where a variety of interesting structures were observed, as shown in Fig. 14.[22] These are images without external illumination and the white patches represent NM reacting at 3500K. Subdetonation refers to an input shock that does not quite run to detonation. NM/EDA refers to NM catalyzed by 1% ethylene diamine.[23] In subdetonation, a fine cellular pattern is observed and in the detonation a larger pattern is observed. These two patterns arise from a two-step explosion of the shocked NM. This two-step explosion launches a sequence of two shocks that catch up with the input shock downstream. The size of the cell pattern arises from the location where the shocks merge. Farther downstream, the more divergent shocklets from the NM explosion are filtered out.[23] This technique reveals both the number of important kinetic processes and their rates.

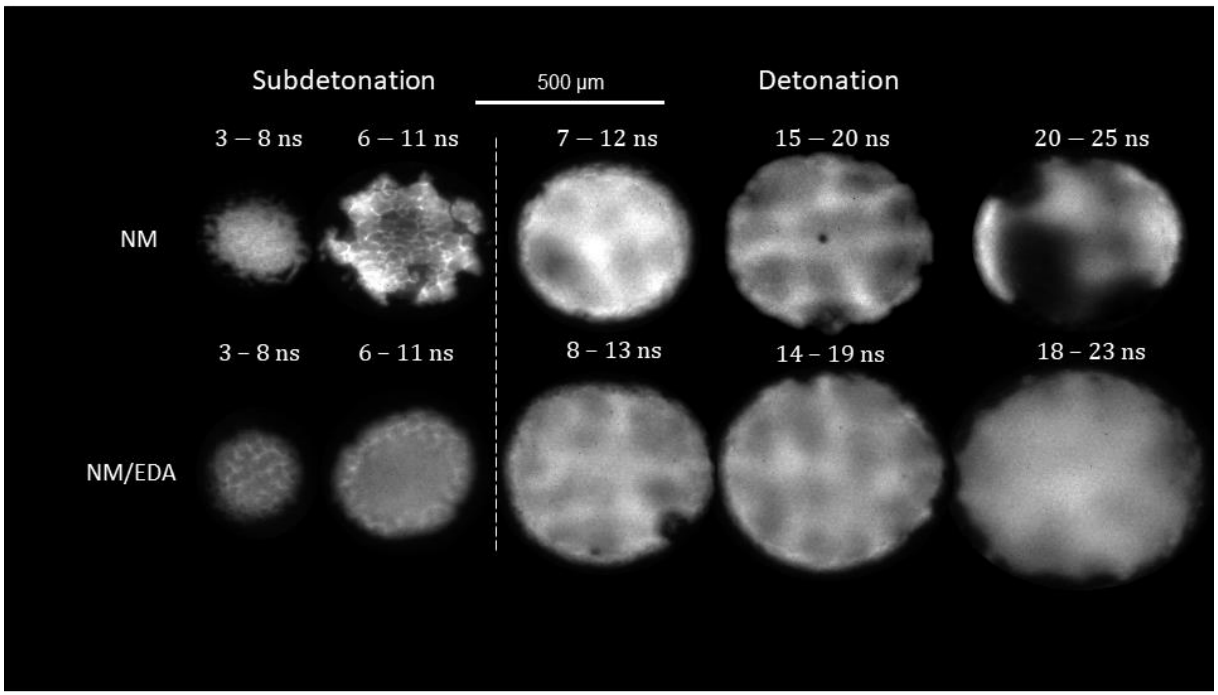


Figure 14. Images of thermal emission of nitromethane and nitromethane with EDA catalyst using shock that does produce detonation and detonation shock. The two distinct cellular patterns represent the merging of two shocks produced by two-stage nitromethane explosion with downstream input shock.

Since NM is a liquid, it is possible to employ additives in a way not really possible with solid explosives. Figure 15 shows images downstream where the detonation forms in pure NM. When EDA is added the detonations are similar but the NM/EDA detonation decays more slowly.[23] When acetone or acetic acid were added, no detonation was produced.

Amines, such as EDA which is a base (proton acceptor) are well-known to catalyze NM decomposition in shocks. Adding acetone can be viewed as adding an inert that simply reduces the energy available. Acetic acid prevents proton transfer to the EDA. In both these latter cases no detonation is produced.

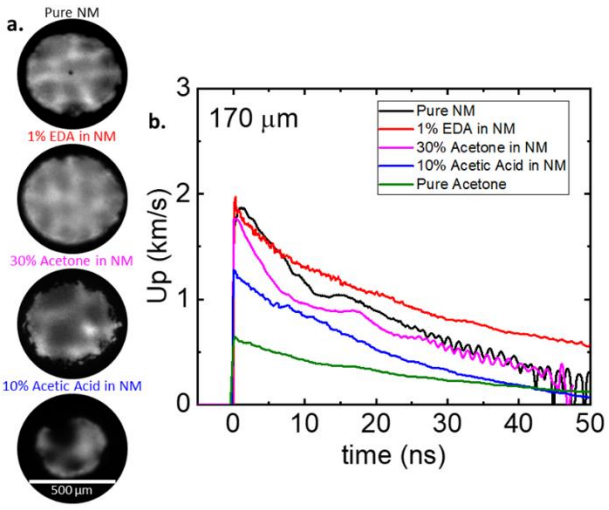


Figure 15. Nitromethane and nitromethane/EDA shock 170 m downstream where detonation is produced show large cellular pattern. Acetone and acetic acid quench the detonation.

We made a very interesting discovery about the mechanism of base catalysis of NM. In the past, only chemical mechanisms were considered, and reaction schemes where the base assisted an initial proton transfer step were discussed and were widely accepted. We found this was indeed the case but another important factor was in play. As shown in Fig. 16, adding just 1% EDA had a radical effect on the Hugoniot at lower velocities. At higher velocities where both NM and NM/EDA ran to detonation there was no such effect. The direction of the EDA effect was such that the NM/EDA was far more compressible than NM alone. We attributed this effect to the widespread ionization produced with base catalysis. With NM/EDA at lower input pressures the reactive flow was much more ionic than with pure NM. The intermolecular interactions in NM were non-bonded interactions but with NM/EDA these interactions were ionic and much stronger so with NM/EDA the volume collapsed as ions were produced more readily in the flow. The conclusion was that bases catalyze shocked NM in two ways,[23] by accelerating charge transfer and by drastically modifying the flow characteristics. This example in a model system highlights the need to view shock chemistry as both chemical events and reactive flow, not just chemistry.

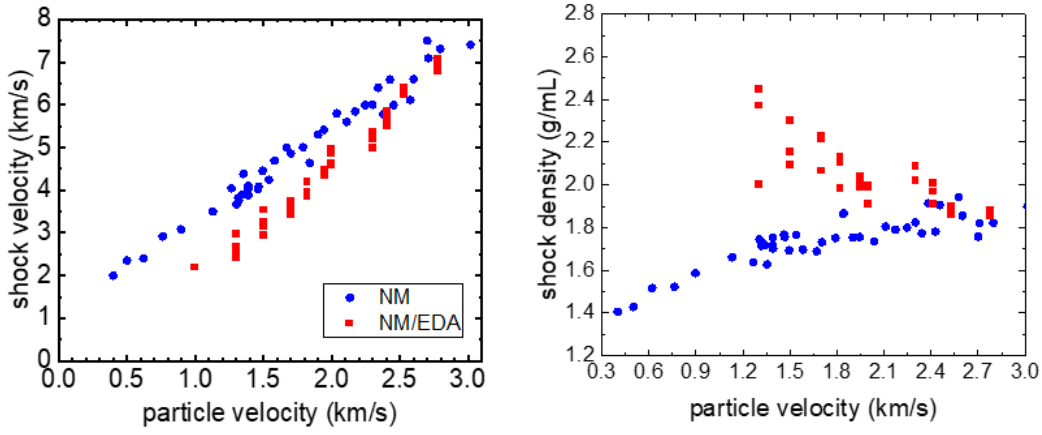


Figure 16. EDA catalyzes proton transfer that lowers the threshold for nitromethane detonation. It also has a large effect on the Hugoniot (left) and compressibility (right) at lower velocities.

Hot spots

The hot spots discussed here are formed by interactions between shocks and microstructures that can concentrate the shock wave energy.[24,25] Hot spots play a critical role in shock initiation and might be equally important for detonation propagation, at least in the most insensitive materials. Figure 17 shows some time-dependent temperature profiles for four energetic materials fabricated into PBX form with 80% solid and 20% PDMS binder with impact velocities ranging from 2.3 km/s to 3.8 km/s.[8] Since the intensity of thermal emission is proportional to a high power of temperature, when there is an inhomogeneous temperature distribution, the graybody temperature tends to reflect the hottest regions.

In Fig. 17, the shortest time temperatures are hot spot temperatures while the longest time temperatures represent deflagration temperatures of material not ignited by the shock. The very fast cooling observed during the first 100 ns with the highest flyer plate velocities is due to rapid volume expansion of the shocked PBX which cools the material by adiabatic expansion.

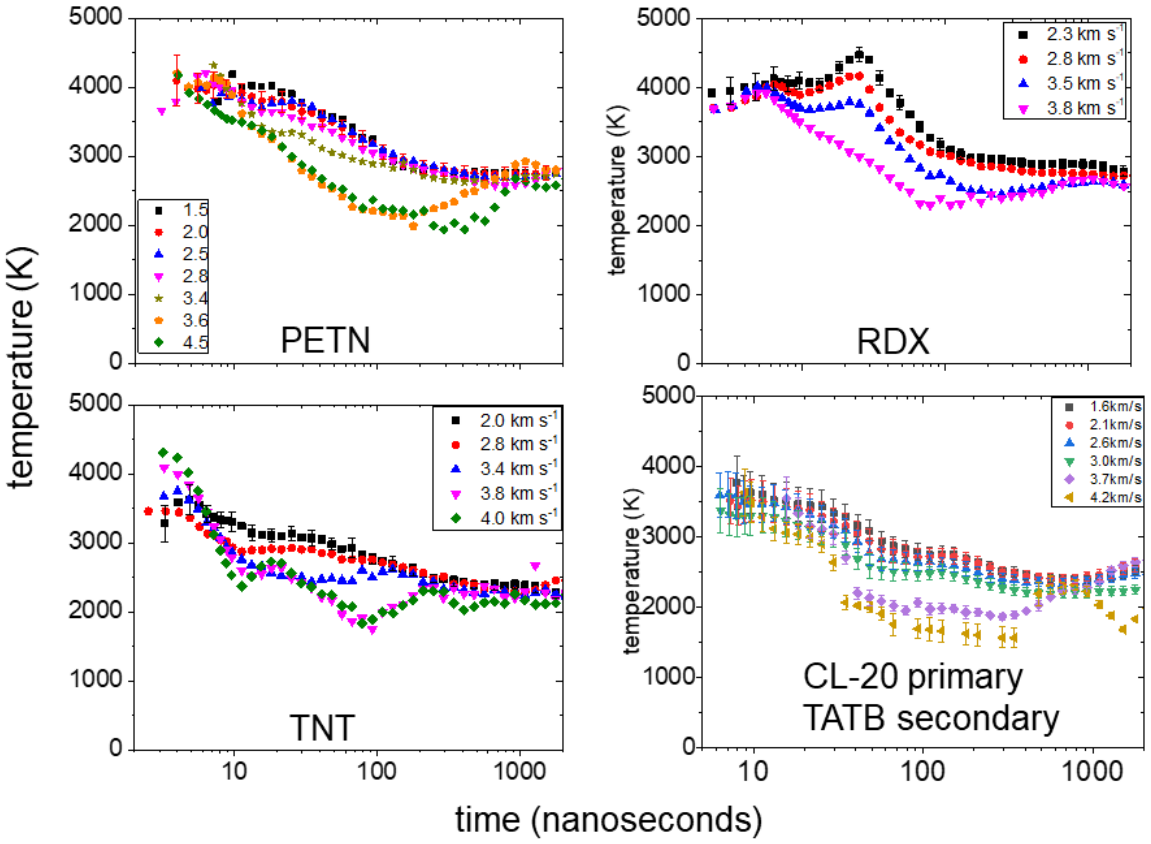


Figure 17. Temperature histories of PBX (20% PDMS binder) of four explosives with various impact velocities.

Following our pyrometer development, we acquired a high-speed camera to directly image hot spots. We studied shocked HMX single crystals and polycrystalline aggregates embedded in a transparent polymer, polyurethane produced by HTPB polymerization. Figure 18 shows a single crystal embedded in polyurethane, an array of single crystal samples, one crystal after flyer plate

impact where the polymer is cracked by the flyer plate and also by the crystal explosion, and a hole where the crystal used to be.[26]

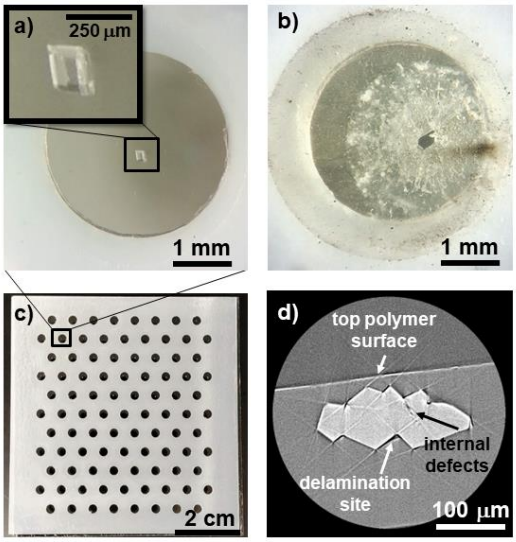


Figure 18. Sample consisting of HMX single crystal embedded in transparent polyurethane. (b) After shock crystal has vaporized and polymer is cracked. (c) Array of 50 crystals. (d) Nano-CT scan of a polycrystal in urethane. The crystal is located just under the impact surface.

Figure 19 shows hot spots evolving in time on single crystals with 20 GPa shocks. These hot spots always form at crystal-polymer interfaces, especially at crystal edges. Figure 20 shows similar results but on polycrystals evidencing a variety of crystal junctions, internal pores, cracks and so forth. Now the hot spots are very bright and they form at these defects rather than at crystal/polymer interfaces. In both single crystals and polycrystals, the hot spots we saw can grow into a deflagration that consumes the entire crystal in roughly 250 ns but the initial hot spot temperature is higher than in polycrystals.[26]

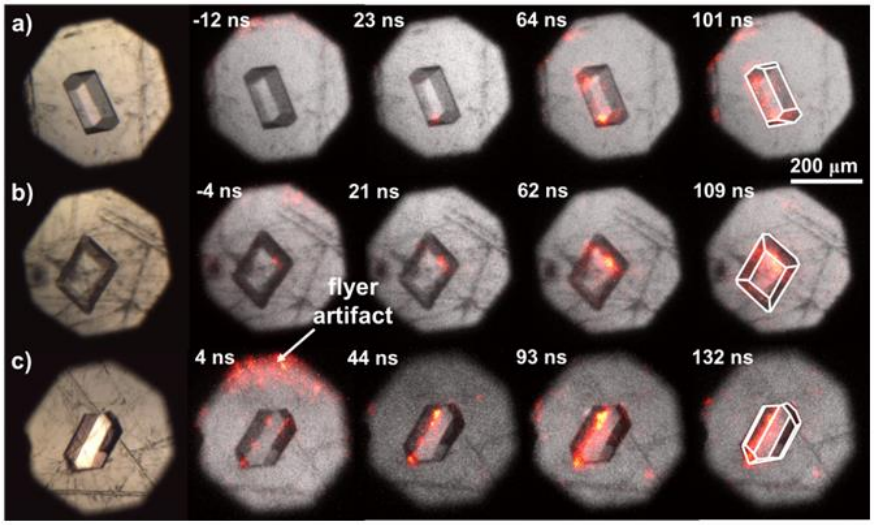


Figure 19. Hot spots in single crystal HMX embedded in polymer with 20 GPa input shock. Red spots are thermal emission. Static images of crystals are shown to indicate where hot spots are located. They appear at crystal/binder interfaces on crystal edges.

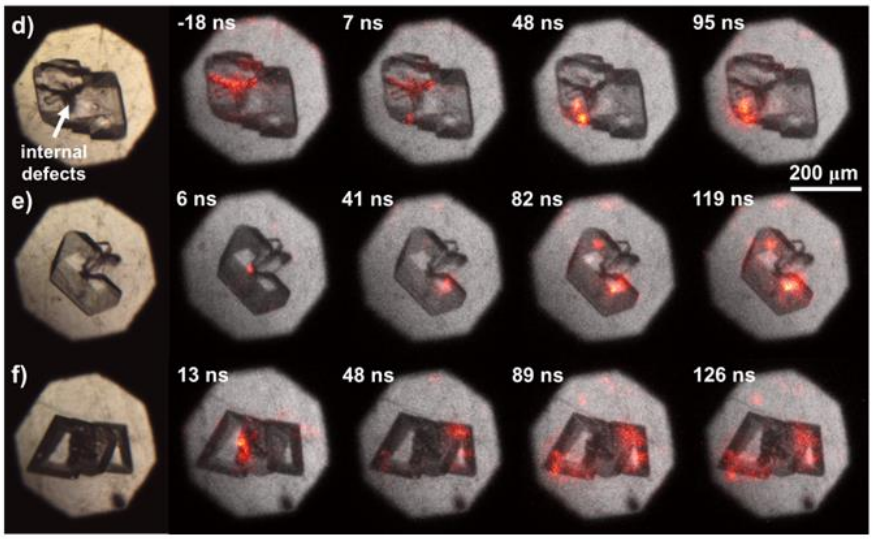


Figure 20. Hot spots in HMX polycrystals embedded in polyurethane with 20 GPa shock appear at crystal junctions and internal defects. Red spots are thermal emission. Static images of crystals are shown to locate hot spots on the shocked crystal.

Reactive materials

Reactive materials generally refer to various combinations of metal fuels and oxidizers.[27] Burning metal particles with oxidizers such as oxygen or fluorine can produce up to twice as much energy as the energetic materials described above, composed of CHON atoms, such as HMX, RDX, TATB, etc. Those materials react via deflagration, which is an energetic reaction in a medium where fuel and oxidizer are premixed. So with HMX $(CH_2\text{-}N\text{-}NO_2)_4$, for instance, the carbon atoms can react with oxygen atoms on the same molecule. Reactive materials undergo combustion, which is rate-limited by the need to physically mix initially separate fuel and oxidizer. So despite the greater potential for energy release, reactive materials tend to react too slowly to support powerful detonations, especially when mixing is diffusive. Many reactive materials produce only solid products, so there is the additional disadvantage of minimal gas production needed to drive detonation shocks through the material.

In collaboration with Prof. Jon-Paul Maria at Penn State, we studied shocks in reactive nanolaminates, where planar fuels and oxidizers (here Zr and CuO) meet at a high-quality interface,[28] as diagrammed on Fig. 21. This configuration allows us to see what is happening at the fuel/oxidizer interface with a shock. The images in Fig. 21 show something unexpected. The initial reactions occur in an annular region corresponding to the edges of the flyer plate. The radiance shows reactions occurring within 30 ns. This is explained by considering the types of material deformations produced by the flyer plate. At the center the sample experiences a primarily compressive load while at the edges there is a lot of shear that helps mix fuel and oxidizer. This experiment provides a clue how to cause reactive materials to react as fast as explosives such as HMX where the reaction zone is tens of nanoseconds.

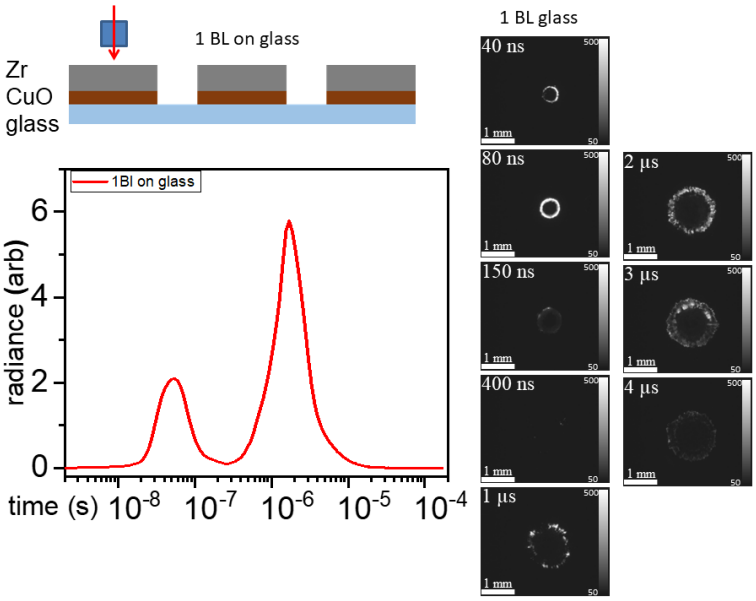


Figure 21. Flyer plate impact on Zr/CuO nanolaminate produces two emission bursts. The initial burst is from the flyer plate perimeter where shear mixing is most efficient, as shown in images on right. Second burst is from underneath the flyer where the load is primarily compressive.

In collaboration with Prof. Edward Dreizin of New Jersey Institute of Technology, we studied some reactive materials produced by arrested reactive ball milling (ARM).[29] The fuel was Al and the oxidizers were either Bi_2O_3 or BiF_3. The ARM process generates Al microparticles shot through and through with nanometric oxidizer, so fuel and oxidizer are mixed on the nanoscale, as shown in Fig. 22. This complex geometry seemed likely to produce a lot of shear mixing and the nanometric mixing should help promote rapid reaction.

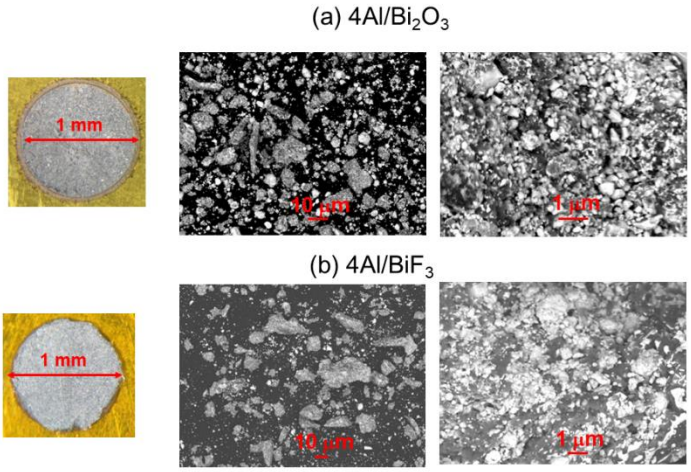


Figure 22. Reactive materials produced by arrested reactive ball milling (ARM) as loose powder in a 1 mm well prior to shock and electron micrographs at two resolutions.

Figure 23 shows pyrometry with a 3 km/s impact on a loose powder bed of reactive particles. The reaction temperature of 3200K is similar to HMX. The radiance plot shows that energy can be released in 20-30 ns. When we compare the oxide to the fluoride (Fig. 24), we

expect to see nonvolatile products such as Al_2O_3 with the oxide but volatile products such as AlF with the fluoride. The temperature profile in Fig. 24 shows a fast cooling process with the fluoride ordinarily associated with fast adiabatic volume expansion (c.f. Fig. 17). In fact the time-resolved snapshots of the loose powder bed shows significant volume expansion within 30 ns. Consequently we have demonstrated that reactive materials produced by ARM can produce high temperatures and volume expansion when shocked, at rates comparable to HMX, with the potential to exceed HMX performance.[29]

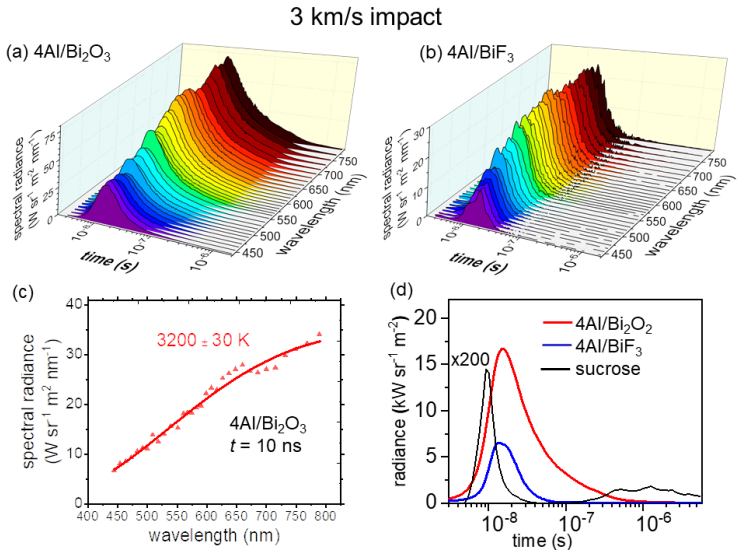


Figure 23. (a), (b) Spectral radiance from an oxide and fluoride based ARM material with 3 km/s impact. (c) Spectral radiance along with graybody fit showing 3200K temperature reached within 10 ns. (d) Time-dependent radiance. The sucrose control (x200) has weak fast emission due to triboluminescence. The ARM materials have longer-lasting emission indicating fast energy release within 20 ns.

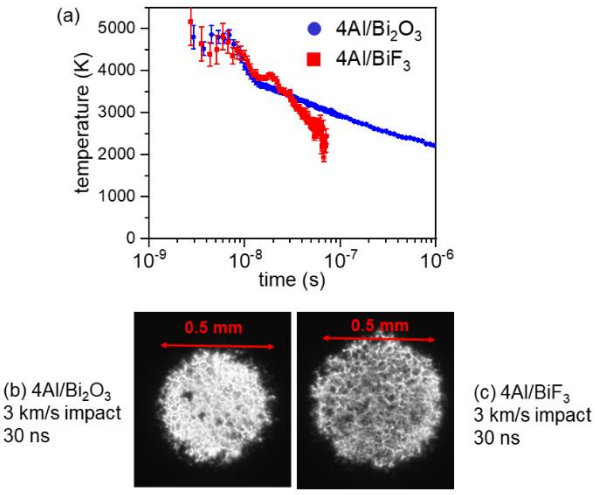


Figure 24. (top) Both oxide and fluoride ARM particles show high temperatures after 3 km/s impact. The fast cooling of the fluoride indicates fast adiabatic expansion. (bottom) The fluoride expansion can be seen in the 30 ns image.

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