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14. ABSTRACT This project was a 3-year study of the fundamental science underlying impact initiation and ignition of nanotechnology reactive materials (RM). In a conventional shock initiation experiment, the duration of the emission bursts is controlled by the size of the charge. We operated in a limit where the charge was so thin that the emission duration was controlled by fundamental mechanisms. A novel laser-driven flyer plate apparatus was developed to launch metal foils at RM up to 4.5 km/s. A study to test the apparatus studied time-resolved emission					
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Report Title

Ultrafast impact dynamics of reactive materials (Dlott): Final Report

ABSTRACT

This project was a 3-year study of the fundamental science underlying impact initiation and ignition of nanotechnology reactive materials (RM). In a conventional shock initiation experiment, the duration of the emission bursts is controlled by the size of the charge. We operated in a limit where the charge was so thin that the emission duration was controlled by fundamental mechanisms. A novel laser-driven flyer plate apparatus was developed to launch metal foils at RM up to 4.5 km/s. A study to test the apparatus studied time-resolved emission from a dye embedded in an impacted sample. A detailed study was made using an RM consisting of nano-Al + Teflon which can generate more than three times the energy of TNT. With 50 micron flyers producing ~10 ns shocks, the RM was initiated above 1 km/s. During the shock, the emission looked just like Teflon alone. Afterward giant emission bursts lasting a few tens of ns were generated due to Al + Teflon chemistry. These observations indicated the mechanism involved initial shock decomposition of Teflon into carbon plus fluorine. After the shock unloaded, fluorine penetrated the oxide passivation layers of the Al nanoparticles, initiating violent reactions.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
04/16/2013	10.00 Hiroki Fujiwara, Kathryn E. Brown, Dana D. Dlott. High-energy flat-top beams for laser launching using a Gaussian mirror, Applied Optics, (06 2010): 0. doi: 10.1364/AO.49.003723
04/16/2013	14.00 Rusty W. Conner, Dana D. Dlott. Ultrafast Condensed-Phase Emission from Energetic Composites of Teflon and Nanoaluminum, The Journal of Physical Chemistry A, (07 2010): 0. doi: 10.1021/jp101539u
04/16/2013	13.00 Xianxu Zheng, Alexander D. Curtis, William L. Shaw, Dana D. Dlott. Shock Initiation of Nano-Al + Teflon: Time-Resolved Emission Studies, The Journal of Physical Chemistry C, (03 2013): 0. doi: 10.1021/jp312637g
04/16/2013	12.00 Yuanxi Fu, William L. Shaw, Dana D. Dlott, Kathryn E. Brown. Time-resolved emission of dye probes in a shock-compressed polymer, Journal of Applied Physics, (11 2012): 0. doi: 10.1063/1.4765687
04/16/2013	11.00 Kathryn E. Brown, William L. Shaw, Xianxu Zheng, Dana D. Dlott. Simplified laser-driven flyer plates for shock compression science, Review of Scientific Instruments, (10 2012): 0. doi: 10.1063/1.4754717
08/11/2012	6.00 Rusty Conner, Dana Dlott. Comparing Boron and Aluminum Nanoparticle Combustion in Teflon Using Ultrafast Emission Spectroscopy, Journal of Physical Chemistry C, (12 2011): 2751. doi:
08/11/2012	9.00 Christopher M. Berg, Kathryn E. Brown, Rusty W. Conner, Yuanxi Fu, Hiroki Fujiwara, Alexei Lagutchev, William L. Shaw, Xianxu Zheng, Dana D. Dlott. Experiments Probing Fundamental Mechanisms of Energetic Material Initiation and Ignition, MRS Proceedings, (02 2012): 0. doi: 10.1557/opl.2012.254
08/11/2012	7.00 Rusty W. Conner, Dana D. Dlott. Time-Resolved Spectroscopy of Initiation and Ignition of Flash-Heated Nanoparticle Energetic Materials, The Journal of Physical Chemistry C, (07 2012): 14737. doi: 10.1021/jp303077f
08/22/2011	2.00 Rusty W. Conner, Dana D. Dlott. Ultrafast emission spectroscopy of exploding nanoaluminum in Teflon: Observations of aluminum fluoride, Chemical Physics Letters, (8 2011): 0. doi: 10.1016/j.cplett.2011.07.036
08/22/2011	4.00 Hiroki Fujiwara, , Kathryn E. Brown, Dana D. Dlott. A THIN-FILM HUGONIOT MEASUREMENT USING A LASER-DRIVEN FLYER PLATE, AIP Conference Proceedings, (07 2012): 1. doi:
08/22/2011	3.00 Kathryn E. Brown, , Rusty Conner, , Yuanxi Fu, , Hiroki Fujiwara, Dana D. Dlott*. MICROSCOPIC STATES OF SHOCKED POLYMERS, AIP Conference Proceedings, (07 2012): 1. doi:
TOTAL:	11

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

Received Paper

09/22/2011 5.00 D. Dlott. New Developments in the Physical Chemistry of Shock Compression,
Annual Review of Physical Chemistry, (01 2011): . doi:

TOTAL: **1**

Number of Papers published in non peer-reviewed journals:

(c) Presentations

1. (invited) Army Research Office Review of Nano-engineered energetic materials, Aberdeen, MD, Mar. 2010, "Ultrafast dynamics of NEEMs".
2. NATO Munitions Safety Information Analysis Center (MSIAC) Workshop on Insensitive Energetic Materials, NATO Headquarters, Brussels, Belgium, May 2010, "Molecular mechanisms of insensitive explosives".
3. (invited) Army Research Office Symposium on Insensitive Energetic Materials, Aberdeen, MD, June 2010, "Fundamental Processes and Properties of Insensitive Energetic Materials".
4. (invited) "Ultrafast Dynamics of Impact Chemistry: Initiation to Detonation", Aug. 2010, Joint JSF-IAS Workshop on Interface Physics Singapore August 29-September 5, 2010
5. (invited) "Shock compression with atomic resolution", Workshop on Dynamic Phenomena, Jan 2011, Austin, TX.
6. (invited) "Molecular transformations and energy transfer at interfaces", USC-DOE conference on "Materials for Energy Applications - Experiment, Modeling and Simulations", Mar. 2011, Los Angeles, CA.
7. (invited) Studium Conference on in situ molecular spectroscopic technique and application, Orleans, France, June 2011, "In situ probing by time-resolved vibrational spectroscopy: shocked materials and energy storage media"
8. (invited) American Chemical Society National Meeting, Denver, CO Aug. 2011, "Interfaces under extreme conditions"
9. (invited) Materials Research Society National Meeting, Boston, MA Nov. 2011, "Experiments Probing Fundamental Mechanisms of Energetic Material Initiation and Ignition"
10. (invited) "Experiments probing initiation and ignition of energetic materials", International Center for Applied Computational Mechanics (ICACM) symposium New York, NY June 11-13 2012.
11. (invited) "Shock physics for dummies!", Gordon Conference on Energetic Materials, W. Dover, VT, June 2012.
12. (invited) "Laser-driven flyer plates for shock compression spectroscopy", International Symposium on Pulsed Power Applications, Kumamoto University, Kumamoto, Japan, Mar. 2013

Number of Presentations: 12.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

04/16/2013 15.00 Hiroki Fujiwara, Kathryn E. Brown, Dana D. Dlott. LASER-DRIVEN FLYER PLATES FOR REACTIVE MATERIALS RESEARCH, SHOCK COMPRESSION OF CONDENSED MATTER 2009: Proceedings of the American Physical Society Topical Group on Shock Compression of Condensed Matter. 2009/06/28 01:00:00, Nashville (Tennessee). : ,

TOTAL: **1**

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received Paper

TOTAL:

Number of Manuscripts:

Books

Received Paper

TOTAL:

Patents Submitted

Patents Awarded

Awards

Jupiter Laser Facility Program Advisory Committee, Lawrence Livermore National Laboratory, 2008, 2011
Executive Advisory Board, PULSE center, Stanford Linear Accelerator Center, Stanford University, 2011-12
ACS Physical Chemistry Division Award in Experimental Physical Chemistry, 2013.

Graduate Students

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	Discipline
Rusty W. Conner	0.50	
Kathryn E. Brown	0.10	
William A. Shaw	0.25	
FTE Equivalent:	0.85	
Total Number:	3	

Names of Post Doctorates

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
Hiroki Fujiwara	0.25
Xianxu Zheng	0.25
FTE Equivalent:	0.50
Total Number:	2

Names of Faculty Supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	National Academy Member
Dana D. Dlott	0.00	
FTE Equivalent:	0.00	
Total Number:	1	

Names of Under Graduate students supported

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

- The number of undergraduates funded by this agreement who graduated during this period: 0.00
- The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:..... 0.00
- The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:..... 0.00
- Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):..... 0.00
- Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:..... 0.00
- The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00
- The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields: 0.00

Names of Personnel receiving masters degrees

<u>NAME</u>
Total Number:

Names of personnel receiving PHDs

<u>NAME</u>
Rusty W. Conner
Kathryn E. Brown
Total Number:
2

Names of other research staff

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

Technology Transfer

This project was a study of the fundamental science underlying impact initiation and ignition of nanotechnology reactive materials (RM),³⁻⁷ using a laser-driven flyer plate apparatus⁹ that launches metal foils in the 0.1-5 km/s range. Time-resolved spectroscopy was used to monitor chemical and material transformations at a high level of detail. The motivation was to understand the interplay between mechanics, nanostructure and chemical reactivity that combine to produce impact response. The most studied RM was Al/Teflon,¹⁰⁻¹⁴ although we have obtained preliminary data on Al/Fe₂O₃ nanothermites¹⁵⁻¹⁹ and Ni/Al intermetallics.^{7,20-24}

The dynamics of conventional reactive materials containing micron to millimeter particles are usually viewed within a mechanical engineering framework where the rates of mass and heat transfer processes are rate-limiting, and the details of the underlying chemistries are simplified by treating them as phenomenological heat-evolution processes. In nanomaterials, heat transfer, mass transfer and chemistry can occur at similar rates, so more complicated theoretical frameworks are indicated. There are several reasons why an improved understanding of fundamental mechanisms would be useful. Weapons designers would like to design materials having tailored mechanics and controllable energy release. For instance an anti-ship or anti-tank missile should penetrate armor without much fragmentation, and only then release its energy. A reactive bomb casing should fragment without significant reactivity to a desired size distribution and then release energy upon impact with a target. Tasks such as these are better accomplished when their designs are based on first-principles' understanding of the relationships between mechanics and chemistry. Weapon design today involves heavy use of computer simulations. Engineering-based simulations are good at designing for known scenarios, but function poorly when extended to novel situations. Only fundamental science-based simulations are reliable in novel situations. One important feature of our experiments is that the time and length scales are

compatible with science-based simulation techniques,²⁵⁻³¹ and the level of detail extracted permits critical—as opposed to superficial—verification of simulation accuracy.

When an explosive charge is detonated, there is an emission burst and the duration of the burst is controlled by the size of the charge.^{32,33} In our experiments, the charge is made small enough that the emission burst is controlled by nanostructure and reactive chemistries, which allows us to investigate nanoscale material reactivity at a level of detail not previously achieved. One area of particular interest is to look at RM very close to the threshold for reaction, which we believe is more informative than looking far above threshold or far below threshold.

Because these are single-shot measurements, emission spectroscopy was the most sensitive optical probe method. However emission from condensed matter generally has a low information content when compared, say, to gas-phase emission. The emission bursts in our experiments were characterized by the spectral distribution and absolute intensity, the onset delay time after impact, and the burst structure and duration. These factors provided a great deal more information than has been previously obtained, but were not always directly evocative of detailed chemical and physical mechanisms. Those mechanisms were revealed by systematic studies of how the emission bursts vary with impact velocity, RM composition, nanostructure and impact conditions.

When the project began, we had what we view now as a primitive flyer plate capability^{34,35} and a lot of experience with Al/Teflon materials developed during a prior project that investigated the response of Al/Teflon (and related materials such as B/Teflon) to ultrafast laser flash heating.³⁶⁻³⁹ In fact during the project period we wrapped up the flash-heating studies which resulted in a number of nice publications and the Rusty Conner Ph.D. thesis.^{36-38,40}

Unfortunately, early on in the program we determined that our existing launch facility did not produce flyers with enough energy to initiate the RM of interest. We applied to the DURIP (instrumentation) program to purchase a larger launch laser, and we also developed a high-speed (8 GHz) photon Doppler velocimetry (PDV) capability.^{2,9,35} We spent more time in instrument and capability development than originally anticipated. Fortunately this new facility

worked out very well. It is now an agile technology platform for understanding impact initiation in a wide variety of RM using small sample quantities (milligrams) without the safety concerns associated with conventional testing. In addition precious prototype materials can be studied in detail. We conducted a series of shock compression science and spectroscopy experiments to test and verify our capabilities, and then we investigated the impact reactivity of interesting RM.

The method used to generate and probe shocks is illustrated in Fig. 1. The laser-launched thin metal foils are called

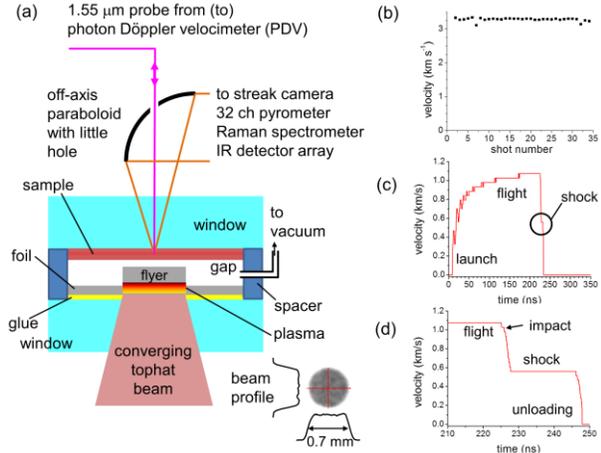


Fig. 1. Schematic of experiment for ns, μm resolution. A flattop laser beam (note beam profile) 0.7 mm in diameter, produced with a diffractive optic, launches a metal foil that flies across a gap to impact a sample. The flyer velocity is recorded by an optical interferometer (PDV). A parabolic mirror collects emission from the sample or brings in IR laser beams. (b) Reproducibility measurement where 33 flyers were shot from one assembly. (c) Velocity record where a 50 μm thick flyer was shot at 1.1 km/s onto a glass window. (d) Impact record on expanded scale. The velocity 0.55 km/s, lasting ~ 15 ns, is the speed U_p of the glass/foil interface.

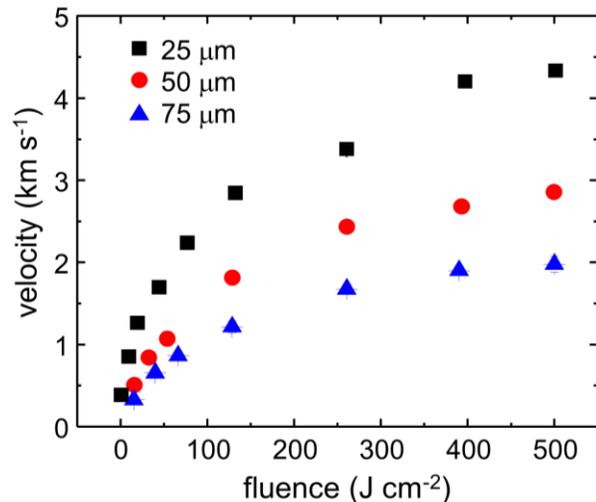


Fig. 2. Flyer velocity versus input laser fluence for different thickness flyers.

laser-driven flyer plates.⁴¹ Figure 2 shows the range of flyer velocities for different thickness flyers. The plates were 25-100 μm thick, creating 8-32 ns duration shocks. Notice Fig. 1b, which illustrates the high level of reproducibility in the flyer launch. The impacted samples were 1-100 μm thick.⁹ High-speed single-shot diagnostics were used to probe the plates and the samples. These included PDV for velocity measurements and a streak camera for time-resolved emission. In addition we made significant progress in developing a Raman capability and a high-speed pyrometry capability.

Our first high-speed spectroscopy experiments, used the set up diagrammed in Fig. 3, involved shocking a 5 μm thick layer of PMMA doped with R640 dye, on top of a thicker slab of PVA polymer.¹ In this configuration, the flyer plate hits the dye layer and the shock propagates through PMMA without reflection into the impedance-matched PVA dissipation layer. During

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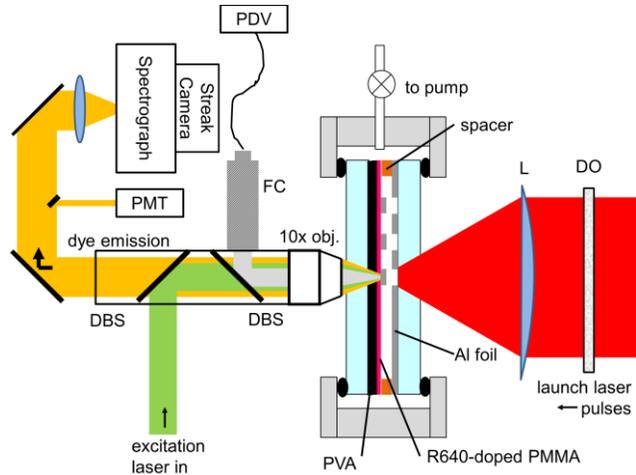


Fig. 3. Schematic of apparatus to study dye emission from a thin PMMA layer, during a shock. The sample was a glass window with 30 μm PVA cushion layer and 5 μm dye-doped PMMA layer. The polychromatic probe transmits and receives returned 1.55 μm light from the PDV. It transmits 527 nm light from the excitation laser and receives returned visible emission. Key: 10x obj. = 10X microscope objective; DO = diffractive optic; L = aspheric objective lens; DBS = dichroic beam splitter; PMT = photomultiplier tube; FC = fiberoptic collimator.

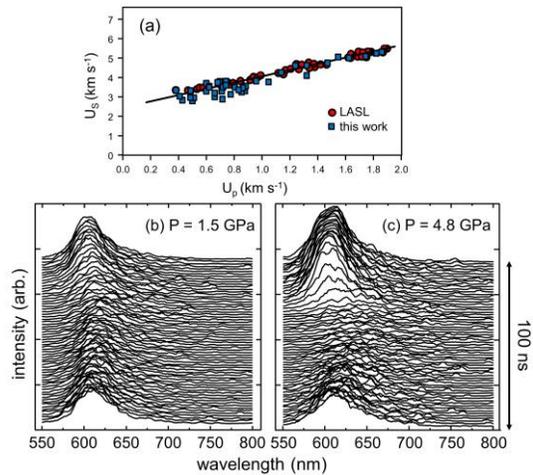


Fig. 4. (a) Hugoniot of 40 μm thick PMMA using laser flyer plates,^{1,2} compared to literature results⁸ from Los Alamos. (b) Dye emission spectra with 1.5 GPa shock. (c) Dye emission spectra with 4.8 GPa shock. Arrival of the shock front causes the emission to lose intensity and redshift, with minimal broadening.

the shock, the dye was quasi-continuously pumped by a green laser, making it a steady emitter. Dye emission transients were monitored with a streak camera and spectrograph. Simultaneously PDV monitored flyer velocity. Since we had measured the PMMA film Hugoniot (Fig.

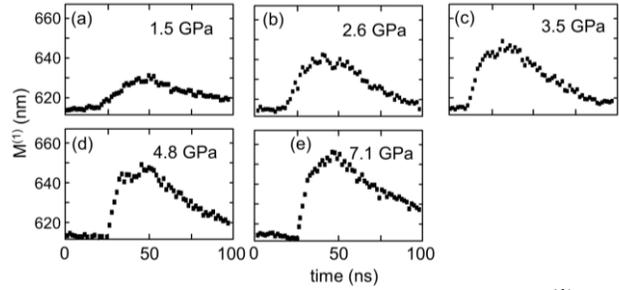


Fig. 5. Dye emission redshift (first moment $M^{(1)}$ of the emission spectrum) from thin PMMA layer at indicated shock pressures. The shock front steepens up above 3.5 GPa. The shock duration was ~ 15 ns.

4a),⁹ PDV gives the shock pressure. Figures 4b,c show some dye emission transient spectra (15 ns shock). When the shock arrived, the emission lost intensity and redshifted. The intensity loss occurred because the dye absorption was shifted away from the exciting laser line. The spectral broadening was minimal. The redshift is a useful monitor of time-dependent pressure transients. Figure 5 shows how the redshift varied with time at different shock pressures. We can see the steepening of the rising edge of the redshift, that tracks the shock front, with increasing shock pressure. These measurements validate our ability to measure and analyze spectra of shocked molecules, and are a first step in developing high-speed embedded optical shock probes.

We studied impact-initiation of Al/Teflon.⁴² Al/Teflon samples (Fig. 6b) 12 μm thick (~ 3 ns shock transit) were made from 50 nm oxide-passivated Al and 3 μm Teflon particles, with a small

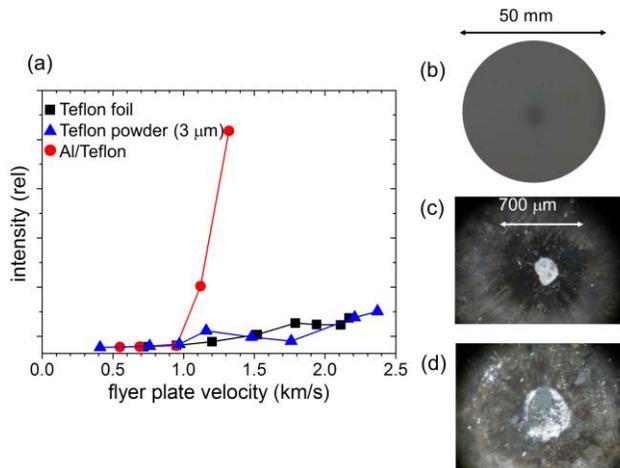


Fig. 6. (a) Total emission intensity from Al/Teflon and two forms of Teflon alone. Above 1 km/s, Al/Teflon initiation results in huge emission bursts. (b) Al/Teflon sample on a 2" window. (c) Teflon powder after impact with 700 μm diameter flyer, shows dark debris spread out from the center which was vaporized. (d) Al/Teflon after impact has a debris field extending outward from the center, and much of the central region has been vaporized.

amount of PMMA binder. Teflon-only control samples were also made. With 50 μm thick flyers, huge emission bursts from Al/Teflon not seen with Teflon alone start at 1 km/s (Fig. 6a). Shock-induced emission from Teflon alone was also observed. At 1 km/s, prominent Swan bands (C_2 emission) were seen, indicating Teflon decomposed into carbon and fluorine.⁴² Recovered Teflon and Al/Teflon samples are shown in Figs. 6c,d.

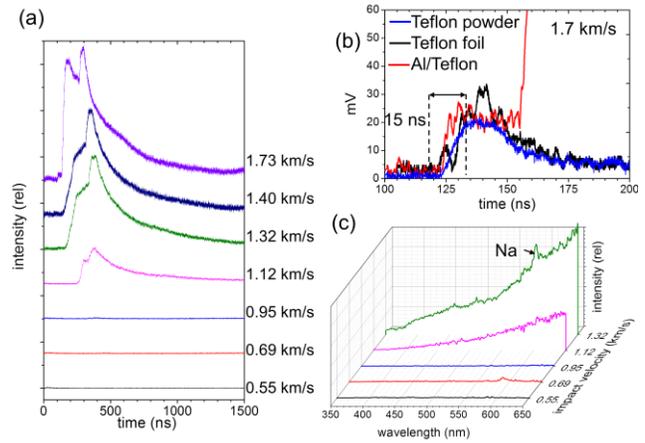


Fig. 7. Impact emission bursts from Al/Teflon 12 μm thick. (a) Temporal structure of emission bursts, offset vertically for clarity. (b) Emission transient from 1.7 km/s impact on Al/Teflon, Teflon foil and Teflon powder. Time zero was when the laser launch pulse arrived. During the 15 ns shock, the Al/Teflon emission was similar to Teflon alone. When the release wave arrives 15 ns after impact, the Al/Teflon emission jumps. (c) Emission spectra during the bursts. The peak at 589 nm is Na emission from the glass surface heated by the exploding Al/Teflon.

Figure 7a shows that emission from exploding Al/Teflon has a dual-burst structure. The emission is broadband (Fig. 7c), and what we see is consistent with the 3300K blackbody emission observed with large explosive charges.⁴³ According to Wien's law, a blackbody at 3300K would have an emission peak at 880 nm but our spectrometer does not work that far into the near-IR.

Figure 7b shows that during the 15 ns shock, the emission from Teflon alone and from Al/Teflon are quite similar in intensity, but a short time after the shock unloads, Al/Teflon emits the huge dual bursts. This timeline is seen in more detail

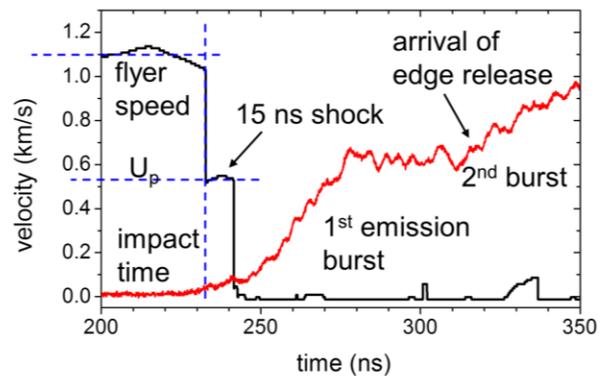


Fig. 8. Comparison of 1.1 km/s flyer plate velocity record from PDV with Al/Teflon emission transient. The onset of the intense emission bursts denoting explosive reactivity coincides with the time when the flyer velocity drops to zero.

in Fig. 8, which correlates the flyer velocity record with the 1st emission burst. The 2nd emission burst is correlated with the arrival of release waves about 100 ns later, from the edges of the 730 μm diameter flyer.⁴²

Summary

During the three year duration of this contract, we developed a unique suite of instrumentation that allows us to study impact initiation of reactive materials with high time resolution. Several experiments were done to characterize our instrumentation capabilities and a thorough study of an interesting reactive material, nano-Al + Teflon was conducted. The groundwork is in place to extend these studies to a wide range of other RM and efforts are underway to further improve our capabilities.

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