FINAL TECHNICAL REPORT

Federal Agency:	The Department of Defense Defense University Research Instrumentation Program (DURIP) PA-AFRL-AFOSR-2016-0001
Prepared for:	Office of Naval Research Dr. Chad Stoltz (N00014, 351)
Award #:	N00014-17-1-2741
Title:	Development of a Multi-Frame Detection System to Obtain Real-Time X-Ray Measurements in Shocked Energetic Materials
Principal Investigator:	Professor Yogendra Gupta Washington State University Institute for Shock Physics PO Box 642816 Pullman, WA 99164-2816 ymgupta@wsu.edu (509) 335-7217
Period of Performance:	September 1, 2017 – August 31, 2018
Distribution Statement:	DISTRIBUTION A. Approved for public release: distribution unlimited.

Development of a Multi-Frame Detection System to Obtain Real-Time X-Ray Measurements in Shocked Energetic Materials

Y. M. Gupta
Department of Physics and Institute for Shock Physics
Washington State University

Abstract

This DURIP award was used to acquire/assemble a Multi-frame Detection System to obtain real-time x-ray diffraction and phase contrast imaging measurements (micrometer resolution) in energetic materials (EMs) subjected to well-characterized shock wave loading. Such measurements will permit – for the first time – the ability to routinely examine changes in crystal structure (phase transformations), microstructure, and crystal-binder interfaces in real-time (while they are happening in nanoseconds to microseconds) in dynamically compressed EMs.

The importance/relevance of such measurements for fundamental research, and for the safe and optimal use of EMs, cannot be emphasized enough. X-ray diffraction and phase contrast imaging measurements will not only provide new mechanistic insights into EM response, but will also serve to evaluate multiscale modeling results.

The multi-frame detection system is deployed at the Dynamic Compression Sector (DCS) located at the Advanced Photon Source (Argonne), a first-of-its-kind user facility dedicated to dynamic compression science. Having this multi-frame detector capability (for x-ray diffraction and imaging) available for dynamic experiments on EMs provides a novel and unique experimental capability for the Office of Naval Research and other Department of Defense research related to EMs. X-ray diffraction and imaging measurements on dynamically compressed EMs have not been possible before. In addition, a new generation of research scientists will be educated on using such facilities for EM related research.

Background

As shown in Figure 1, high explosives (HE) – also referred to as energetic materials (EMs) and plastic-bonded explosives (PBXs) – are composite heterogeneous materials consisting of energetic molecular crystals embedded in polymeric binders and a variety of other additives. A long-standing and important need regarding the optimal design and use of EMs for Department of Defense (DoD) applications is the ability to accurately model and predict the spatial and temporal evolution of the physical and chemical changes in EMs when they are subjected to impulsive – high stress, short duration – loading.

Although the above-indicated need is widely recognized, addressing it constitutes a significant challenge because of the inherent scientific and technical complexities that result from the heterogeneous nature of the EMs, the strong coupling of the mechanical-thermal-chemical responses, and the wide range of length and time scales that need to be considered. A paper by Rice (ARL)[*] provides an excellent perspective on the computational state-of-the-art and the many scientific challenges associated with multiscale modeling of energetic materials. Rice's abstract stated, "The response of an energetic material to insult is perhaps one of the most difficult

[*] Rice, Betsy M. (2015). "A Perspective on Modeling the Multiscale Response of Energetic Materials." 19th American Physical Society Topical Conference on Shock Compression of Condensed Matter.

processes to model due to concurrent chemical and physical phenomena occurring over scales ranging from atomistic to continuum." Below, we briefly discuss the multiscale aspect of the relevant scientific issues.

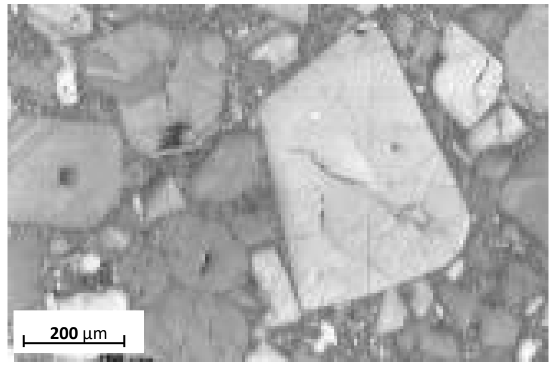

Figure 1. SEM image of PBX

When high stress and short duration loading is imparted to EMs, the resulting material response and energy release involves multiple length scales and a range of short time scales. Multiple length scales arise because of the composite and granular nature of the EMs, the microscopic scales associated with mechanical and thermal changes within the EM constituents and at the HE crystal/binder interfaces, and the resulting breaking and forming of molecular bonds. The onset and growth of chemical reactions (molecular phenomena) in EMs is both rapid and evolutionary, typically exhibiting an induction period following by rapid acceleration, spanning a range of short time scales. In summary, the sequence of physical and chemical processes culminating in rapid, sustained chemical energy release in EMs subjected to dynamic loading is inherently multiscale in nature.

To address the scientific issues indicated above, there exists a strong need to experimentally examine the real-time (ns to μs), multiscale response of EMs subjected to well characterized dynamic loading conditions. Because such measurements in single event experiments involving EMs are extremely challenging, a well thought out multiscale measurement effort needs to be started so that correspondence between measurements and computations continues to grow. The desired x-ray measurements, using the proposed Multi-frame Detection System, represents a key step that has been missing as explained below.

To date, the research effort at Washington State University – with sustained funding from the ONR Energetic Materials Program – has focused on a molecular level of understanding of the onset of chemical reactions in shock compressed, macroscopically homogenous materials: liquid nitromethane, PETN single crystals, and RDX single crystals. By pioneering, and subsequently using, time-resolved electronic and vibrational spectroscopy measurements (with ns resolution) under shock compression, a significant amount of mechanistic understanding has been achieved to date regarding the molecular changes associated with the onset of chemical decomposition in shock compression of liquid NM, PETN, and RDX.

As valuable as the time-resolved optical spectroscopy results to date have been, they have some inherent limitations for developing a comprehensive understanding of the EM response under shock compression. These limitations can be succinctly summarized as follows:

- Inability to examine structural transformations (or crystal structure changes) and microstructural changes (deformation-induced changes) in real-time
- Inability to examine crystal-binder interface changes in real-time

The above limitations are quite significant because the onset of shock-induced chemical reactions in EMs is intimately related to mechanical and thermal changes (within the energetic crystals and at the crystal-binder interfaces). Since optical spectroscopy measurements cannot probe shock-induced structural transformations (directly), microstructural changes, and crystal-binder interface details, it is difficult to relate mechanical deformation and chemical changes – mechanochemistry – under shock loading in a meaningful manner.

Although there have been many hypotheses (work by Armstrong and coworkers, and by Gilman) and a large number of multiscale simulations (e.g. Goddard et al., Vashishta et al., Rice et al., Sewell et al., Fried et al.) that have linked dynamic compression/deformation to the onset of chemical changes, the lack of meaningful experimental data makes it difficult to ascertain the validity and the accuracy of the calculation results. Even for dynamically compressed energetic crystals (without the crystal-binder interaction complexity), knowledge of the precise crystal structures and detailed understanding of the crystal microstructures (including different types of defects) are important factors in developing credible mechanisms that govern mechanochemistry under dynamic loading.

Real-time x-ray diffraction (XRD) and x-ray phase contrast imaging (PCI) measurements in shock compressed EMs are required to address the key experimental knowledge gaps listed earlier. With DOE/NNSA sponsorship, WSU has designed and developed the desired experimental facility – the Dynamic Compression Sector (DCS) at the Advanced Photon Source (APS), Argonne National Laboratory (Argonne, IL). The DCS, operated by WSU under a Cooperative Agreement with DOE/NNSA, is dedicated to understanding condensed matter response under dynamic compression.

The development of a multi-frame X-ray detection system (for diffraction and imaging measurements) – for use in dynamic compression experiments on EMs – will significantly advance the experimental state-of-the-art in understanding the response of energetic crystals (by themselves) and the response of the crystal-binder composites.

Accomplishments and Activities

Equipment Installation

The Multi-frame Detection System was assembled installed at the DCS to obtain real-time x-ray diffraction and imaging measurements in shock compressed materials. The Multi-frame Detection System (Figure 1) is comprised of the equipment items (listed below):

- PI-MAX4: 2048f-HBf Digital Intensified Cameras ($398,668): Three PI-MAX4 cameras were purchased from Princeton Instruments.
- Fiber Optic Tapers ($46,428) Three fiber optic tapers were purchased from Incom, Inc. to provide demagnification to 40 mm diameter, from three different image sizes with diameters of 150 mm (Part# 600-4777-A), 120 mm (Part# 600-5471-A), and 75 mm (Part# 600-4175-A).

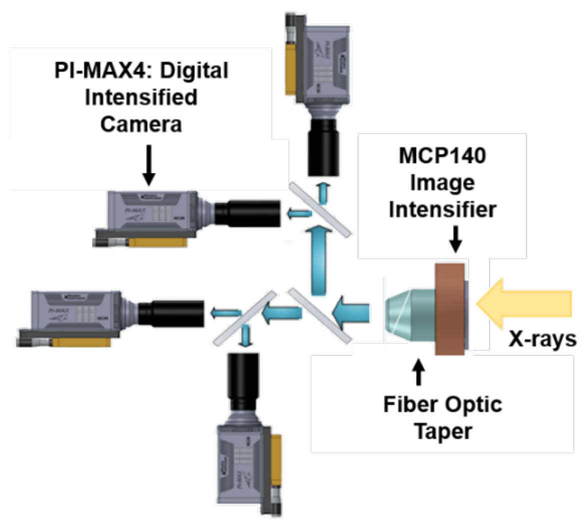

Figure 1. Multi-frame Detection System

- To complete the system, additional equipment items – the Image Intensifier and fourth PI-MAX4 Camera as depicted in Figure 1 – were funded by the DOE/NNSA Cooperative Agreement DE-NA0002442.

Activities

Two collaborations with NSWC IHEODTD are underway related to the dynamic compression of EMs.

1) Dr. Alexandra Reinert (Principal Investigator) and other NSWC team members will be conducting experiments at the DCS to examine the impact response of reactive materials (RMs). During their last visit to the DCS (November 2018), preliminary experiments were conducted to ensure that the experimental configuration (including relevant components) developed will satisfy the safety protocols and other experimental requirements for obtaining the desired PCI measurements. This was very useful in refining the experimental details needed for the desired measurements.

2) Dr. Zbigniew Dreger (Principal Investigator) has initiated a collaboration related to dynamic compression experiments on thin PBX samples. Although much of this work will utilize the capabilities at the Institute for Shock Physics in Pullman, WA to conduct time-resolved emission and optical imaging experiments, one of the collaboration goals is to conduct experiments at the DCS.

REPORT DOCUMENTATION PAGE	*Form Approved* *OMB No. 0704-0188*

1. REPORT DATE *(DD-MM-YYYY)*	2. REPORT TYPE	3. DATES COVERED *(From - To)*
27-11-2018	Final Technical Report	09/01/2017 - 08/31/2018

4. TITLE AND SUBTITLE
Development of a Multi-Frame Detection System to Obtain Real-Time X-Ray Measurements in Shocked Energetic Materials

5a. CONTRACT NUMBER

5b. GRANT NUMBER
N00014-17-1-2741

5c. PROGRAM ELEMENT NUMBER

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

6. AUTHOR(S)
Gupta, Yogendra M.

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Washington State University
Institute for Shock Physics
PO Box 642816
Pullman, WA 99164-2816

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Office of Naval Research
875 N. Randolph Street Suite 1425
Arlington VA 22203-1995
Technical Representative: Dr. Chad Stoltz (Code: 351)

10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S REPORT NUMBER(S)

12. DISTRIBUTION/AVAILABILITY STATEMENT
DISTRIBUTION A. Approved for public release: distribution unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT
This DURIP award was used to acquire/assemble a Multi-frame Detection System to obtain real-time x-ray diffraction and phase contrast imaging measurements in energetic materials (EMs) subjected to well-characterized shock wave loading. Such measurements will permit the ability to routinely examine changes in crystal structure, microstructure, and crystal-binder interfaces in real-time in dynamically compressed EMs. The importance of such measurements for fundamental research, and for the safe and optimal use of EMs, cannot be emphasized enough. X-ray diffraction (XRD) and phase contrast imaging measurements (PCI) will not only provide new mechanistic insights into EM response, but will also serve to evaluate multiscale modeling results. The multi-frame detection system is deployed at the Dynamic Compression Sector located at the Advanced Photon Source (Argonne). Having this multi-frame detector capability available for dynamic experiments on EMs provides a novel and unique experimental capability for the Office of Naval Research and other Department of Defense research related to EMs. XRD and PCI measurements on dynamically compressed EMs have not been possible before. In addition, a new generation of research scientists will be educated

15. SUBJECT TERMS
X-ray Diffraction, Phase Contrast Imaging, energetic materials, dynamic compression

16. SECURITY CLASSIFICATION OF: a. REPORT	b. ABSTRACT	c. THIS PAGE	17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
U	U	U	UU	5	Dr. Yogendra Gupta
					19b. TELEPHONE NUMBER *(Include area code)* 509-335-7217

Reset