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14. ABSTRACT The objective of this grant is to purchase a state-of-the-art high speed imaging system to analyze energetic material reactions. This new system enables capturing images at up to 1,000,000 frames per second and at resolutions up to 1280 x 800 pixels, leading to one of the world's fastest video data acquisition rate. This camera can also be easily coupled with existing ignition sources to characterize and quantify performance parameters associated with combustion of energetic materials.					
15. SUBJECT TERMS energetic materials, reaction propagation, combustion dynamics, imaging systems					
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Report Title

Final Report: Imaging Diagnostics for Analysis of Energetic Material Reactions

ABSTRACT

The objective of this grant is to purchase a state-of-the-art high speed imaging system to analyze energetic material reactions. This new system enables capturing images at up to 1,000,000 frames per second and at resolutions up to 1280 x 800 pixels, leading to one of the world's fastest video data acquisition rate. This camera can also be easily coupled with existing ignition sources to characterize and quantify performance parameters associated with combustion of energetic materials.

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>
03/11/2017	2 Dylan Smith, Michael Bello, Daniel Unruh, Michelle Pantoya. Synthesis and Reactive Characterization of Aluminum Iodate Hexahydrate Crystals $[Al(H_2O)_6](IO_3)_3(HIO_3)_2$, Combustion and Flame, (): . doi:
03/11/2017	1 Ethan Zepper, Michelle Pantoya, S. Bhattacharia, Jeremy Marston, Andreas Neuber, Ronald Heaps. Peering through the flames: imaging techniques for reacting aluminum particles, Applied Optics, (): . doi:
TOTAL:	2

Number of Papers published in peer-reviewed journals:

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

<u>Received</u>	<u>Paper</u>
TOTAL:	

Number of Papers published in non peer-reviewed journals:

(c) Presentations

Number of Presentations: 0.00

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

<u>Received</u>	<u>Paper</u>
03/11/2017	3 Ryan Bratton, Michelle Pantoya. A closer look at determining flame speeds from imaging diagnostics, 10th U. S. National Combustion Meeting. 24-APR-17, College Park, MD. : ,
TOTAL:	1

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

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

<u>Received</u>	<u>Paper</u>
TOTAL:	

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

(d) Manuscripts

<u>Received</u>	<u>Paper</u>
TOTAL:	

Number of Manuscripts:

Books

Received Book

TOTAL:

Received Book Chapter

TOTAL:

Patents Submitted

Patents Awarded

Awards

None

Graduate Students

<u>NAME</u>	<u>PERCENT_SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Names of Post Doctorates

<u>NAME</u>	<u>PERCENT_SUPPORTED</u>
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

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

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>
Total Number:

Names of other research staff

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

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

See Attachment

Technology Transfer

Technology transfer has included several meetings with scientists at the Army Research Laboratory in Aberdeen Proving Ground. Individuals include: Drs. Jennifer Gottfried, Steven Dean, Jesse Sabatini, Kevin McNesby.

Project Report - Grant # W911NF1610181
(Final Report: January, 2016 – January, 2017)

Imaging Diagnostics of Analysis of Energetic Material Reactions

Dr. Michelle Pantoya
Mechanical Engineering Department
Texas Tech University, Lubbock, TX, 79409

Objective

The objective of this grant is to purchase a state-of-the-art high speed imaging system to analyze energetic material reactions. This new system enables capturing images at up to 1,000,000 frames per second and at resolutions up to 1280 x 800 pixels, leading to one of the world's fastest video data acquisition rate. This camera can also be easily coupled with existing ignition sources to characterize and quantify performance parameters associated with combustion of energetic materials.

Approach

- (1) Purchase a Phantom v2512 that has the flexibility to capture 677,000 fps at standard resolution but also has the option for a 280 nm exposure for 1,000,000 fps at reduced resolution (128 x 32 pixel). Overall the resolution can be adjusted up to 1280 x 800 pixels. The data records directly to a 1TB CineMag IV in seconds and has a 288 GB high speed memory that can be divided into up to 63 segments, for flexibility in data collection. Also, the camera is compatible with all of our existing lenses.
- (2) Integrate the new camera with existing imaging diagnostics that will enable a multi-dimensional understanding of the effect of surface reactions between the alumina passivation shell surrounding aluminum particles and any halogenated oxide inducing reaction with alumina on the overall composite's thermal combustion performance.
- (3) Design experiments that will enable an understanding the fundamental chemistry associated with solid energetic material combustion.

Relevance to Army

- Our previous camera was purchased in 2004 and is the *Phantom v7.1*. At full resolution the *Phantom v7.1* camera can capture images at only 4,800 fps (and full resolution is only 800 x 600). The newer version of this camera will enable a significantly broader range of spatial AND temporal resolution and thorough characterization of reaction fields.
- The data will isolate reaction behaviors that are specific to surface chemistry and enable a macroscopic understanding of how small changes in surface chemistry produce large changes in exothermic behavior.
- The instrumentation can be used on various Army supported thermite, reactive material, and energetic composites including metal fuels combustion research also to establish fundamental reactivity mechanisms for a variety of energetic materials.
- The data will provide new information on reaction propagation and reaction mechanisms from energetic material reactions.
- Results obtained from this study will have a dramatic impact on the SAFE handling and use of energetic materials in addition to providing a significant understanding of the thermal response, ignition sensitivity as well as energy transfer.

Accomplishments for Reporting Period

1. Purchased the Phantom v2512 (Vision Research) high speed imaging system to resolve energy propagation and combustion dynamics. **Figure 1** is a photograph of this new imaging system.
2. To establish functionality, we operated the Phantom v2512 in two modes: (1) alone and (2) in conjunction with an existing copper laser. In the first mode of operation, the camera recorded monochromatic images of combustions samples. **Figure 2(a)** illustrates still frame images from high speed imaging of an aluminum powder combustion event ignited by an electrically induced plasma. Neutral density filters were applied to this camera lens to aid visualization of the high brightness reactions.
3. We also operated the Phantom v2512 in conjunction with the existing copper laser that illuminates the sample with high brightness, narrow bandwidth light. The camera was programmed to accept the laser light and reject the broadband light emitted by the subject under study. This feature allows the camera to 'see' through flames and record details of processes that would otherwise be totally obscured by the background light. The illuminating laser light is directed at the subject. Synchronization electronics are used to provide the necessary timing signals to synchronize the camera with the shutter and laser pulses and a laser line filter is placed in front of the camera lens. This set-up reduces the self-luminescence to a minimum level. The combination of a copper laser with mega resolution digital camera provides short pulse durations for applications where the reaction is occurring at a very high speed and the subject is imaged at high magnification. An existing long distance *Nikon* microscopic lens (i.e., K2 lens) attached to the camera will allow detailed, close-up imaging of sample surface reaction behaviors. This feature would be mainly applicable to resolving the solid surface propagation of energy for a reacting mixture. **Figure 2(b)** illustrates still frame images of the same reaction in **Fig. 2(a)** but with the coupled copper vapor laser – high speed camera system. In this image, rotating agglomerates of aluminum can be seen along with a gas/vapor trail moving in the rotational, spiral pattern. This level of detail was not available with our previous camera demonstrating the important higher resolution capabilities of this system.
4. Analysis of the experiments shown in **Fig. 2** reveal quantitative differences. For example, agglomerate size measured without the filtration system was about 160 microns in diameter and with the copper vapor laser, agglomerate size was measured to be 50 microns. **Figure 3** illustrates the distinction in quantitative results from the two modes of camera operation.
5. Imaging diagnostics have been extended to energy propagation of powder reactants using a flame tube. **Figure 4 (top image)** illustrates a still frame image using the high speed camera and **Figure 4 (bottom image)** presents the copper vapor laser imaging complement. Various filtration techniques were applied and for the two reactions examined (i.e., Al + MoO₃ using micron scale powders and also using nanoscale powders) corresponding to two very different average flame speeds (i.e., order of cm/s versus m/s). **Figure 5(a) and (b)** show the measured average flame speed did not change significantly for the slower and faster reactions. These initial results imply that the standardized measurement of flame speed can be measured without significant uncertainty associated with diagnostic approach.
6. Additional imaging studies helped to elucidate the heightened reactivity of an aluminum iodate hexahydrate species we synthesized from aluminum in an iodic acid solution. The speeds of these reactions approached 3000 m/s and could not have been measured with our previous imaging diagnostics. However the Phantom v2512 offers the capture rate to enable multiple frames and a complete analysis of the reaction front.

Collaborations and Technology Transfer

- Drs. Barry Homan, Kevin McNesby, Brad Forch (ARL)
- Drs. John Schmidt, Stephen Howard, Richard Beyer (ARL)
- Dr. Jesse Sabatini, Jennifer Gottfried (ARL)
- Dr. Steven Dean (ARL)
- Drs. Ron Heaps and Dan Prentice (INL)
- Drs. Shawn Stacy & Christopher Applett (SNL)
- Drs. Emily Hunt & Oliver Mulamba (West Texas A&M)
- Dr. Valery Levitas (ISU)
- Drs. Charles Crane & Cory Farley (LANL)
- Dr. Kyle Sullivan and Dr. Alex Gash (LLNL)
- Dr. Christopher Junk (DuPont)
- Drs. Carol Korzeneski and Adelia Aquino (Chemistry Department, Texas Tech University)

Resulting Journal Publications during Reporting Period

1. Zepper, E.T., Pantoya*, M.L., Bhattacharya, S., Marston, J.O., Neuber, A. A., Heaps, R.J., Peering through the flames: imaging techniques for reacting aluminum particles, *Applied Optics*, In Press 2017.
2. Bratton, R., Pantoya, M.L., A Closer Look at Determining Flame Speeds with Imaging Diagnostics, 10th U. S. National Combustion Meeting, Organized by the Eastern States Section of the Combustion Institute, April 23-26, 2017, College Park, Maryland
3. Smith, D. K., Bello, M. N., Unruh, D. K., Pantoya, M. L., Synthesis and Reactive Characterization of Aluminum Iodate Hexahydrate Crystals $[Al(H_2O)_6](IO_3)_3(HIO_3)_2$, *Combustion and Flame*, In Press 2017.

Graduate Students Involved During Reporting Period

- Ethan Zepper (MS)
- Ryan Bratton (MS)
- Dylan Smith (PhD)

Awards, Honors and Appointments

M. Pantoya's Army research was featured in Discovery Channel Daily Planet "Green Ammunition" and aired internationally in 2014. This was a 15 minute segment. M. Pantoya was also featured as *Dr. Michelle The Engineer* on PBS Kids introducing children to engineering. These are a series of 5 short (30-second) segments aired between *PBS Kids* programming throughout the Texas Panhandle region. M. Pantoya received recognition by the YWCA in 2015 with the Women of Excellence award in Science. M. Pantoya received the Outstanding Researcher Award from TTU in 2016.



Figure 1. Vision Research Phantom v2512. Purchased April, 2017 with grant funds.

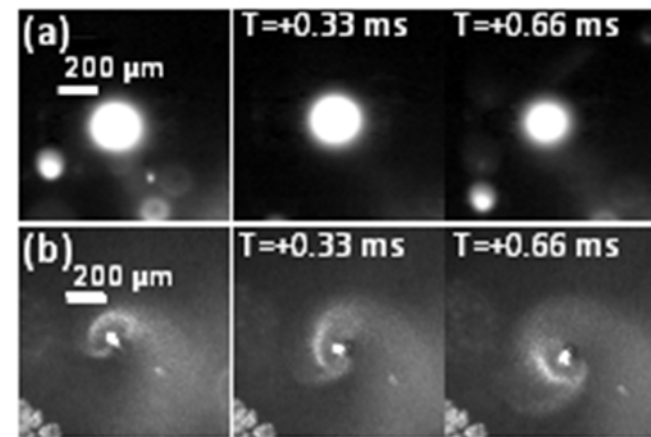


Figure 2. Still frame images of aluminum particles using two experimental methods. Images (a) show HSV-ND: particle oscillations and a 3-pixel difference in diameter. Images (b) show HSV-CVL illumination and a similar cluster of particles in rotation. Scaling and time stamps are shown.

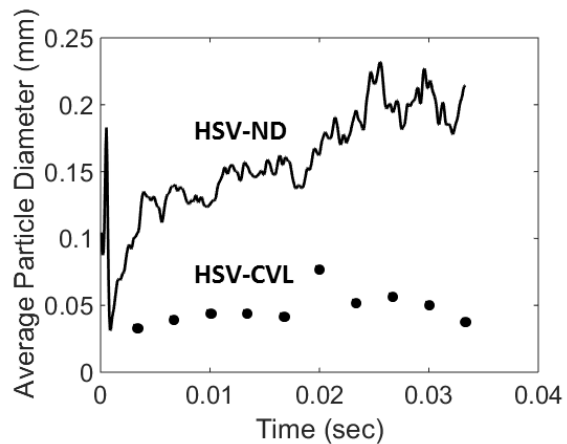
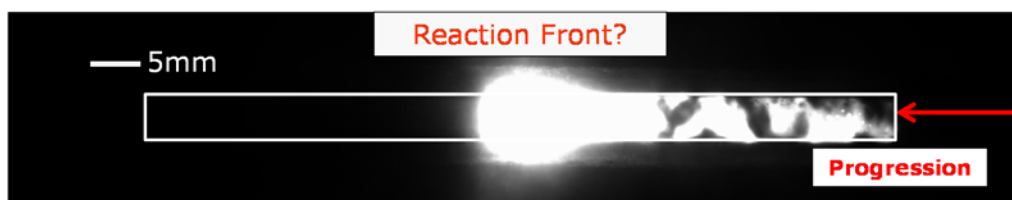
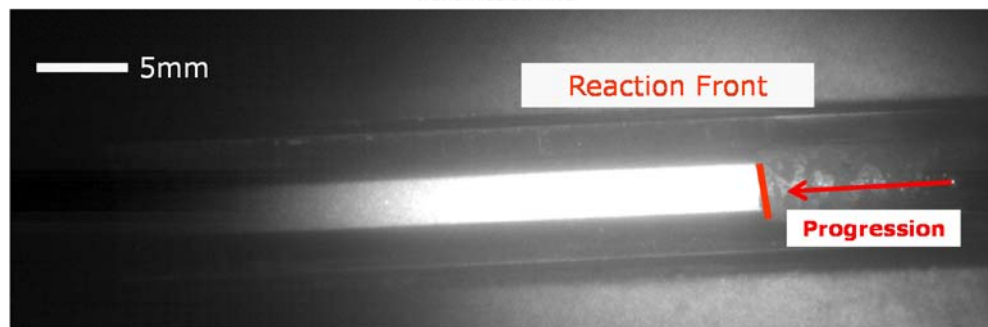


Figure 3. Average particle diameters as a function of time for HSV-CVL and HSV-ND illuminated experiments. Average diameters are 160 microns for HSV-ND (i.e., high speed video with neutral density filter) and 50 microns for HSV-CVL (i.e., high speed video with copper vapor laser)



Self Illuminated Al+MoO₃ with 13% transmission filter



Laser illuminated Al+MoO₃ with 50% transmission filter and CVL 511nm filter

Figure 4. Top: Still frame image of energy propagation with a 13% neutral density filter on the camera lens allowing 87 % light transmission. Bottom: Same powder configuration using a copper vapor laser and notch filter on the camera lens to see 'through' the flames.

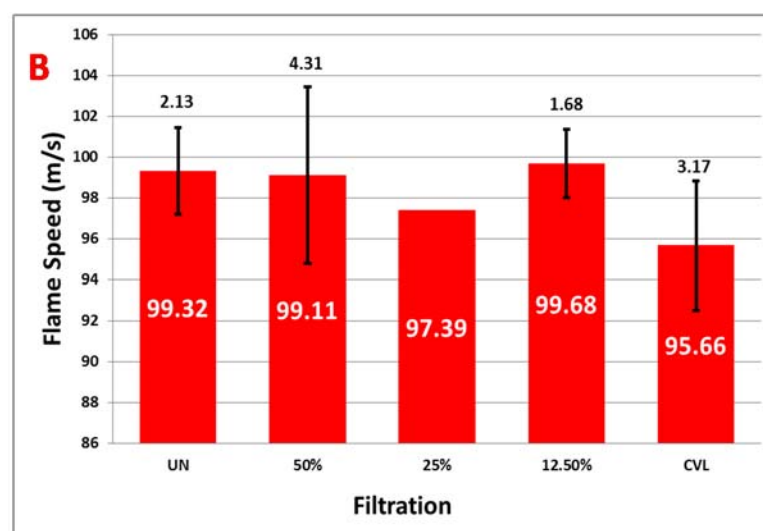
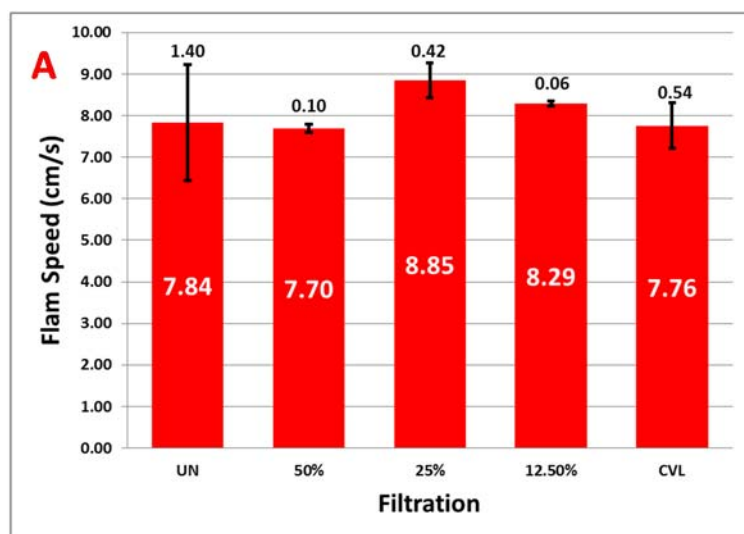


Figure 5. A. Micron powder Al+MoO₃ observed flame speeds B. Nano Al + MoO₃ observed flame speeds. Note filtration abbreviations: UN = unfiltered; 50, 25, 12.5 % corresponds to transmission; CVL = copper vapor laser (as illustrated in Fig. 4(bottom image)).