R1-B.2: Small-Scale Characterization of Homemade Explosives

I. PARTICIPANTS INVOLVED FROM JULY 1, 2019 TO JUNE 30, 2020

Faculty/Staff			
Name	Title	Institution	Email
Steven F. Son	PI	Purdue University	sson@purdue.edu
Vasant S. Vuppuluri	Research Scientist	Purdue University	vvuppulu@purdue.edu
Graduate, Undergraduate and REU Students			
Name	Degree Pursued	Institution	Month/Year of Graduation
Dakota Scott	MS	Purdue University	7/2019

II. PROJECT DESCRIPTION

A. Project Overview

- **Small-scale detonability testing:** We developed the capability to perform small-scale detonation performance testing, using as little as 10 g of material. Normally, detonation performance tests require several kilograms of material, making it very resource-intensive to obtain data with the necessary breadth to parametrize models. We started with a microwave interferometer that could provide a near-continuous shock front position history. We have also performed testing with systems not amenable to microwave using high-speed imaging.
- Model development and calibration: Reliable models are required for the Department of Homeland Security (DHS) to be able to perform accurate threat assessments. We have demonstrated the ability to use data from small-scale detonation failure experiments to calibrate computational models. Through this work, we have determined the small-scale experimental configuration that would be most amenable to computational modeling.
- Collaboration with national research laboratories: We have collaborated with multiple national laboratories, including Lawrence Livermore National Laboratory and Los Alamos National Laboratory. Multiple students have completed internships at these locations.

B. State of the Art and Technical Approach

Typical homemade explosive (HME) formulations consist of a combination of ammonium nitrate (AN), an oxidizer, and an appropriate fuel. A mixture of 94% AN and 6% fuel oil (ANFO) is a common HME formulation. In some HME formulations, AN is mixed with nitromethane (ANNM) instead of fuel oil. AN, NM, and fuel oil have widely accepted civilian uses, making it relatively easy to acquire them for malicious use. ANFO and other HMEs behave as nonideal explosives that are characterized by a long reaction zone length (on order of mm). Their detonation behavior is highly dependent on a variety of parameters, including the particle size and morphology of the AN, confinement, etc. This makes the modeling of their behavior for threat assessment challenging. The dearth of data on these formulations exacerbates the challenge. Typical detonation performance tests to calibrate models are dependent on having a fully developed detonation. Being nonideal

explosives, HMEs have large critical diameters (25 mm or greater). This requires several kilograms of material, making it expensive and time-consuming to perform multiple experiments necessary to parametrize models.

To measure detonation velocity, microwave interferometry is a well-established diagnostic. In a detonating explosive, the reaction front acts as a conductor, which reflects microwaves [1,2]. The incident and reflected microwave signals are mixed, resulting in a beat frequency measured by a detector that can be seen on a digitizer. From this, the position and velocity history of the shock front can be inferred. In addition to measuring fully developed detonations, microwave interferometry can also be used to measure failing detonations. It has been shown that under appropriate conditions, shock front history in a failing detonation can be used to calibrate models [3]. Most explosives are partially transparent to microwaves. For explosives that absorb microwaves, high-speed imaging may be substituted. A 10 MHz camera can be used to obtain the shock front position. An appropriate numerical differentiation scheme may be used to obtain velocity history, from which failure rate may be calculated.

C. Major Contributions

Year 7

 Dakota Scott graduated with an MS in mechanical engineering and has accepted a position at Army Research Laboratory.

Year 6

 Performed detonation failure experiments on the AN/NM system using high-speed imaging. The AN/NM system is not amenable to being studied with microwave interferometry due to nitromethane-absorbing microwaves.

Year 5

- Developed an experiment to measure shock front position using high-speed imaging and calculate velocity history and failure rate. We have compared these data to microwave interferometry data and found them to be comparable.
- Published paper on shock sensitivity of AN and aluminum additives.

Year 4

- Developed an experiment to quantify and rank shock sensitivity using small-scale subcritical diameter experiments. We applied this approach to characterize the effect of aluminum additives of various particle sizes as well as other inert additives.
- Published results of ANFO modeling.

Year 3

- David Kittell graduated with a PhD and joined Sandia National Laboratories.
- Characterized effects of density and confinement on ANFO detonation failure behavior.
- Successfully simulated detonation failure using reaction flow modeling in CTH.

Year 2

- Peter Renslow graduated with an MS in mechanical engineering and joined Sandia National Laboratories.
- Obtained initial results on AN and aluminum (ammonal) and ANFO compositions.

• Published results of time-frequency analysis of microwave interferometry.

Year 1

Developed a small-scale detonation experiment using microwave interferometry.

D. Milestones

- Characterize effect of desensitizing agents: We tested various diluents reported in the literature, including diammonium phosphate (DAP) and calcium carbonate (CaCO₃). These additives were reported as having inhibited the detonation of ANFO. We also considered ascorbic acid, an antioxidant. Our results show that all these materials appear to behave as diluents and do not appear to desensitize ANFO.
- **Develop small-scale disc-acceleration experiment (DAX):** We obtained initial results with our photonic doppler velocimetry (PDV) system. However, we were not able to make further progress due to the COVID-19 pandemic impacting shipment of additional PDV probes and shutdown of university operations.
- **Heated experiments to quantify shock sensitivity:** We were unable to meet this milestone due to COVID-19 pandemic impeding implementation of this technique.

E. Final Results at Project Completion (Year 7)

In Year 7, we focused primarily on studying the effect of desensitizing agents on ANFO. Given the wide availability of AN and fuel oil, identification of an additive that mitigates the potential for malicious use of AN is useful. Many additives such as DAP and $CaCO_3$ have been claimed to inhibit detonation of ANFO. However, there is significant variability in the methods of evaluating the efficacy of these additives. In addition, it is not known whether these materials suppress reaction or behave primarily as diluents.

In our approach, we prepared various formulations of ANFO plus additive and compared their effect on detonation failure. We prepare compositions of ANFO plus 2.5% additive by mass and also ANFO plus 20% additive by mass. We compared DAP, CaCO₃, aluminum oxide (AlO), glass beads, and ascorbic acid. A picture of a test sample is shown in Figure 1. The sample is initiated with an RP-80 detonator. A booster charge of LX-14 is used to initiate the sample material.

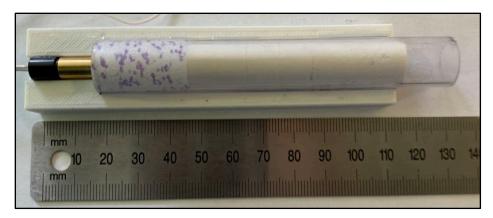


Figure 1: Image of test samples for characterizing desensitizing agents.

Using a Shimadzu HPV-X2 camera, we imaged the detonation of these materials at a 10 MHz frame rate and 50 ns exposure time. Using manual tracking, we obtained the position history of the shock front. A series of 256 images were acquired for each shot. Representative images are shown in Figure 2.

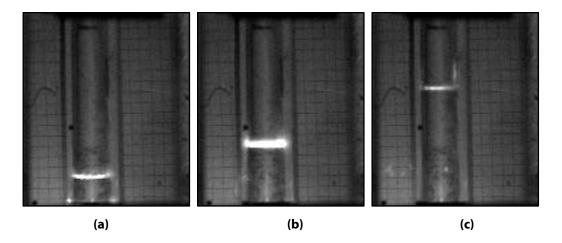


Figure 2: Representative images from detonation of test samples. In (a), the reaction front is propagating through the LX-14 booster. In (b), the reaction front has reached the booster/sample interface. In (c), the detonation is failing but at a relatively slow rate.

Position histories are shown for both the 2.5% and 20% additive cases (both by mass). The shock velocity is relatively steady initially for about $2.5 \mu s$, which is the time taken for the shock to travel through the booster charge. After about $2.5 \mu s$, the shock reaches the sample material, at which point failure begins.

It can be seen from Figure 3 that the position histories are nearly indistinguishable from one another. The shock front positions appear to diverge at later times. The type of additive does not appear to make a significant difference in the detonation failure behavior. In Figure 4, the position histories shortly after the booster/sample interface are relatively similar. Again, the nature of the material added appears to have little effect on the detonation failure behavior.

From this, it appears that all these materials function primarily as diluents and do not suppress reaction.

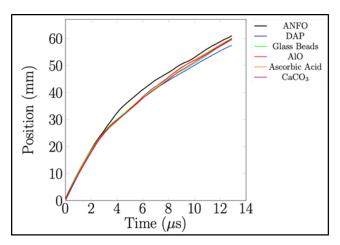


Figure 3: Results from 2.5% additive by mass. ANFO, diammonium phosphate (DAP), aluminum oxide (AlO), ascorbic acid, and calcium carbonate (CaCO₃) are shown.

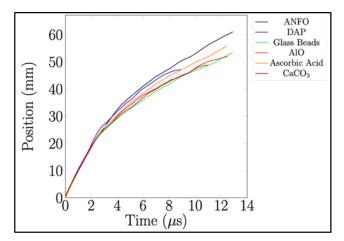


Figure 4: Results from 20% additive by mass. ANFO, diammonium phosphate (DAP), aluminum oxide (AIO), ascorbic acid, and calcium carbonate (CaCO₃) are shown.

Over the life of this project, we developed an experimental framework for performing small-scale detonation testing of nonideal explosive formulations. Our results demonstrated that one can obtain an understanding of shock sensitivity from small-scale experiments. In addition, our modeling results showed that overdriving highly confined HME samples will yield results that are amenable to modeling. As a result of this work, we have graduated three MS students and one PhD student, all of whom are currently working in the national security enterprise.

III. RELEVANCE AND TRANSITION

- A. Relevance of Research to the DHS Enterprise
- 1. Explosive behavior of AN-based HME formulations is highly dependent on a variety of factors, including size and morphology of the AN, presence of sensitizing agents, and type of fuel. Performing standard detonation performance tests with HMEs would normally require kilograms of material. This makes it challenging and costly to obtain enough data to parametrize models. We focused on methods to study AN-based explosives at small scale. With use of as little as 10 g of material, we were able to obtain data that could be used to calibrate computational models, representing a ten-times reduction in material requirement.
- 2. Our work has resulted in multiple journal articles and conference proceedings. As a result, we have made our results available to a variety of customers. In addition, all of the students who have been involved in this project are currently working in the national security enterprise.
- 3. We also studied the efficacy of desensitization agents. We focused on determining whether an additive could function as a true reaction suppressant. The key metric was effect on rate of detonation failure.
- B. Status of Transition at Project End

We recently submitted a proposal to the Defense Advanced Research Projects Agency on small-scale detonation performance evaluation that was heavily based on the expertise we developed as a result of this project.

C. Transition Pathway and Future Opportunities

From this project, we have graduated three MS students and one PhD student. Also, our work on this project has produced over three journal publications.

- D. Customer Connections
- David Kittell, Sandia National Laboratories—former ALERT student
- Scott Jackson, Los Alamos National Laboratory—mentored Nicholas Cummock in summer 2015
- Brian Bockmon, founder of Rocky Mountain Scientific Laboratory
- Mike Lindsay, Air Force Research Laboratory
- Rob Reeves, Lawrence Livermore National Laboratory—mentored Christian Sorensen, former ALERT student

IV. PROJECT ACCOMPLISHMENTS AND DOCUMENTATION

- A. Education and Workforce Development Activities
 - 1. This project provided partial support to Vasant Vuppuluri, research scientist.

V. REFERENCES

- [1] Cawsey, G. F., Farrands, J. L., & Thomas, S. (1958). Observations of detonation in solid explosives by microwave interferometry. Proceedings of the Royal Society of London. Series A. *Mathematical and Physical Sciences*, 248(1255), 499-521.
- [2] Kittell, D. E., Mares Jr, J. O., & Son, S. F. (2015). Using time-frequency analysis to determine time-resolved detonation velocity with microwave interferometry. *Review of Scientific Instruments*, 86(4), 044705.
- [3] Kittell, D. E., Cummock, N. R., & Son, S. F. (2016). Reactive flow modeling of small scale detonation failure experiments for a baseline non-ideal explosive. *Journal of Applied Physics*. 120(6), 064901.