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The Effect of Aluminum on the Explosive Performance of Different Chlorate-based Mixtures

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Abstract. A range of explosive performance experiments were conducted using both stoichiometric and off-stoichiometric potassium chlorate (KC) mixtures with the addition of aluminum: detonability rate stick tests for detonation velocity, kinetic plate tests for close-in impulse, and barometric calorimetry tests for time evolving impulse. The addition of aluminum to otherwise stoichiometric chlorate mixtures resulting in fuel rich mixtures increased performance in some tests but not in others. For rate stick tests, as the ratio of aluminum to fuel increases, the detonation velocity decreases. Impulse evaluated by close-in kinetic plate tests on a liquid fuel mixed with KC was greater for mixtures containing aluminum, and barometric calorimetry showed similar enhancement at late time with aluminum. As long as the mixture is oxygen balanced prior to adding the aluminum, there does not appear to be any degradation in performance with aluminum in the formulation up to a certain percent. Those mixtures have equivalent or greater performance than the same mixtures without aluminum.

Introduction

Aluminum is often included as an additive to a chlorate-based formulation (Urbanski, 1967) where another fuel is present, rather than as the lone fuel in combination with potassium chlorate (KC) or another oxidizer. Aluminized explosives are typically non-ideal, with long, sequential reaction zones that depend on particle size and other factors; such explosives are typically associated with lower detonation pressures (or 'brisance') but higher overall blast potential due to later time combustive effects. As with other highly electropositive metallic elements, aluminum oxidation reactions are strongly exothermic. In the case of KC and aluminum, a balanced reaction assuming full conversion of the aluminum-to-aluminum oxide (Al_2O_3) is as follows:

$$KClO_3 + 2\ Al = KCl + Al_2O_3$$

Aluminum present in an explosive formulation may or may not participate fully in the detonation of the explosive. Depending on the formulation and its physical characteristics, aluminum may react slowly compared to the timescale of a detonation and because of this the aluminum that is present may not be fully converted to Al_2O_3. Even if it is fully converted to aluminum oxide, the rate of reaction may be such that the aluminum contributes primarily to late-time afterburn and

little to the blast properties of the formulation. The aluminum may also form Al_2O_3 through reactions with water and carbon dioxide formed from the reaction of KC with another fuel present, rather than via direct reaction with the KC. Predicting the blast properties of an aluminized KC/fuel formulation is therefore less straightforward than predicting those of a binary KC/fuel formulation.

To assess possible blast enhancement using aluminum as an additive, three types of experiments were devised by Lawrence Livermore National Laboratory (LLNL) and Los Alamos National Laboratory (LANL). Traditional rate sticks were used to assess the influence of aluminum additive on detonation. To assess later time processes where aluminum may enhance blast through combustion, the kinetic plate test was used to assess overall impulse imparted to a rigid structure, and the barometric calorimeter was used to assess the temporal aspect of adding aluminum, including late-time quasi-static combustion effects.

For our recent exhaustive study on KC/fuel explosives we investigated seven different organic fuels both of liquid and granular form as well as many different additives including different aluminum content levels. A solid granular fuel was mixed with KC for the rate stick and calorimeter studies, while a liquid fuel was mixed for the kinetic plate study. A key property of the fuels is oxygen content, which varies from 0% in pure hydrocarbons to more than 51% by weight in some organic fuels.

In the tests discussed here, aluminum was added to KC/fuel mixtures. Some mixtures were balanced to form CO_2, H_2O and Al_2O_3, with sufficient KC as a direct oxidizer for all of the contained aluminum—these were identified as stoichiometric mixtures. Aluminum was also added to mixtures that were otherwise balanced to form CO_2 and H_2O, both of which can act as efficient oxidizers for Al—these were identified as fuel-rich. The fuel-rich formulations optimally utilize Al, because they maximize energy release as well as gas production. When KC directly oxidizes Al, all resulting products are solid, which decreases the amount of work that the explosive formulation can do. However, the reaction of CO_2 and H_2O with Al produces C, CO and H_2, and these species may act as fuels that reignite in air and couple that energy to the blast wave.

Remote Rate Stick Experiment Design

Rate stick tests were performed on fuel-rich, fuel-lean, and stoichiometric formulations to measure the detonation velocities of un-boosted formulations as a function of KC/fuel/Al mix ratio. The results obtained provide part of a broad overview of the detonation spaces associated with these mixtures.

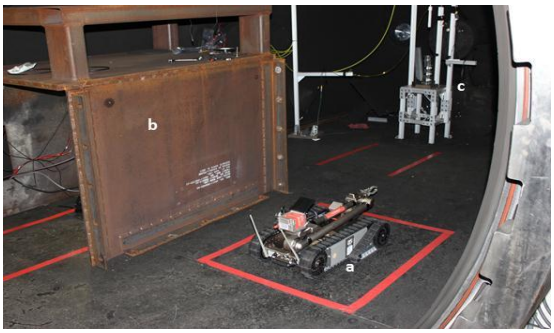

Fig. 1. Tank set up for remote operations – a) ‘LEXI’ iRobot, b) Steel garage that houses the LabRam, c) shot stand.

Preparation and firing of the aluminized KC mixtures were done remotely in a sealed explosives tank for safety and to maintain consistency between experiments (all formulations were prepared, mixed, and packed similarly). Figure 1 is a photograph of the inside of the tank prior to operations. The tank contains a steel garage, or blast shield, which houses and protects the LabRam acoustic mixer; the shot stand where the detonability test is done; and ‘LEXI’, a Packbot 510 iRobot, which is used to remotely deliver mixed material from the LabRam.

After remote mixing is complete, LEXI delivers the mixed cylinder to an outer Lucite cylinder on the shot stand. The tank is then opened so that LEXI can exit the tank whereupon the tank is closed. The detonator assembly is then remotely placed into the mixing cylinder. The detonator holder is also used to tamp down the material simultaneously and controlled remotely by an air cylinder. Once the detonator is placed, the volume of the cylinder is measured using a predetermined marked scale on the Teflon® tamper/detonator holder, which is used to calculate the density of the

mixture. The full sequence of preparation and firing operations are summarized by a photo-series in Figure 2.

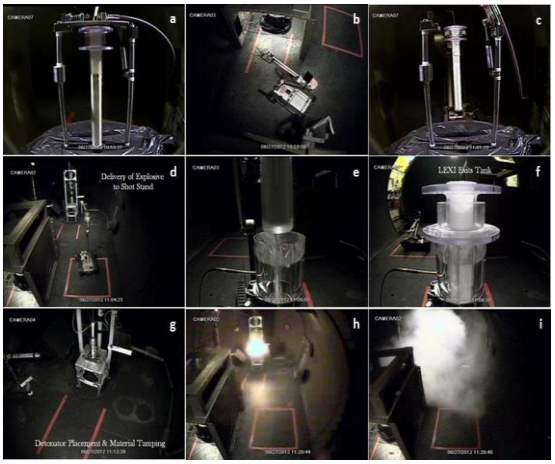

Fig. 2. Remote rate stick operations: a) Mixing in LabRam; b) LEXI drives to the LabRam; c) LEXI retrieves mixing cylinder; d) LEXI transitions to shot stand position; e) LEXI places mixing cylinder in outer cylinder on shot stand; f) LEXI exits the tank; g) Detonator/Tamper placed in cylinder; h & i) explosion.

Diagnostics included high-speed video (150,000 frames per second), one inch thick steel witness plates, and six shorting pins used to measure the detonation velocities of the material. The pins were positioned at 1.9, 5.1, 8.3, 11.4 and 14.6 (0.75, 2.0, 3.25, 4.5 and 5.75 inches) from the bottom of the 30.48 cm (12 inch) cylinder.

Kinetic Plate Experiment Design

Much work has been done in measuring the equations of state (EOS) of various types of explosives, but it remains unclear whether the EOS contains sufficient information to predict the performance of all types of explosive material, or whether the blast delivered by non-ideal explosives may couple more efficiently with structures of concern. In an attempt to understand the differences in blast effects between different explosive charges, the kinetic plate test was developed (Manner et al., 2013). Similar to a ballistic pendulum (Yuen et al., 2005), the test allows for an integrated measurement of detonation energy and early blast performance to momentum transfer. The experiment uses laser velocimetry to measure the momentum delivered to test structures.

The basic test setup is shown in Figure 3. The technique used a 12.7 cm (5 inch) square, 1.3 cm (0.5 inch) thick steel plate weighing ~1.6 kilograms, and a 0.95 cm (3/8 inch) thick steel collar to prevent blast waves from passing around the plate within the experimental time scale. The plate and collar were placed 15.2 cm (6 inches) from the center of the explosive charge. Test geometry was chosen to present a surface facing the explosive charge that was within the fireball generated by detonation of the charge, as this is appropriate for many relevant applications. The plate thickness and mass result in velocities fast enough to be measured accurately with the available instrumentation, but slow enough that the plate distortion would not change significantly on experimental time scales.

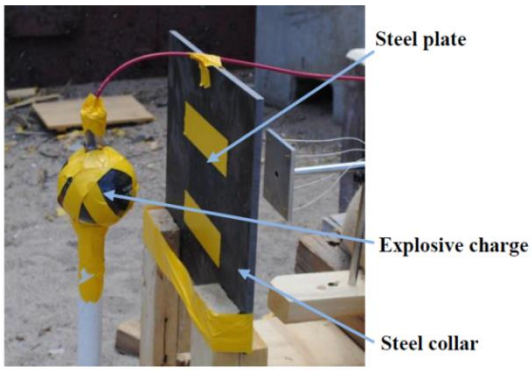

Fig. 3. Kinetic plate experimental setup.

The square plate was loosely taped inside the outer collar, and the tolerances were such that the gap between plates was as small as possible without inhibiting plate motion. The strip of tape stabilized the hardware prior to detonation, but was unlikely to impose significant resistance to inter-plate motion normal to the plate surface. There was likewise no significant resistance to collar rotation relative to its base. High-speed video demonstrated that the plate velocities were slightly faster than those of the collar, as intended.

The primary diagnostic employed during the test series was Photonic Doppler Velocimetry (PDV), a laser interferometric technique that utilized 1550 nm infrared laser light to measure the plate velocity during the first 300-400 μs of motion. Four PDV probes (three to uniquely define the movement of the plate, and one redundant probe) were mounted in an aluminum plate in a square array. The laser probes were mounted opposite the explosive charge at a distance of 10.2 to 15.2 cm (4 to 6 inches) from the plate, and aligned perpendicular to the plate surface using a mirror prior to detonation.

The KC/fuel explosive charges were formed using a hemispherical mold. The booster was placed in the center of the charge, and the RP-1 detonator was positioned parallel to the plate. The setup was lined in tape to hold it together and keep the spherical shape.

Aluminum was added to the mixtures while maintaining the stoichiometric ratio of KC to fuel, i.e., making them fuel-rich explosives as defined for the rate stick experiments. To determine the effect of aluminum addition in limited quantities, we compared both to mixtures without aluminum and to mixtures with an inert surrogate lithium fluoride (LiF). LiF was chosen because it has very similar density and shock impedance, and is not expected to participate in the detonation and post-detonation expansion. The charge mass was held constant for all tests.

Barometric Calorimeter Experiment Design

After detonation, many explosives will release additional energy as the detonation products mix and thermo-chemically react with the ambient environment. This additional afterburn energy release is due to combustion of detonation products with ambient oxygen. While the detonation energy release occurs on a time scale of microseconds, the afterburn energy release occurs on a time scale of milliseconds with a time varying energy release rate dependent upon the local temperature and pressure. Barometric calorimeter experiments have been executed in both nitrogen and air environments to investigate the characteristics of afterburn for KC/fuel explosives with and without aluminum additive. These tests, which provide pressure time histories, along with theoretical and analytical solutions, offer an engineering basis for differentiating the time-evolving role of explosives with and without aluminum.

Experiments were performed in a 506-liter barometric calorimeter rated for charges up to 147 gram TNT equivalent. The 506-liter calorimeter is a cylindrical vessel with inner diameter 86.4 cm (34 inches) and height of 86.4 cm. The circular lid has 17 available ports for pressure gauges and other diagnostics. Figure 4 shows the lid of the large calorimeter being lowered onto the base. Suspended spherical charges were employed for all KC/fuel/Al mixtures. The test configuration uses a spherical PBX N5 booster that has been integrated with an RP 3 detonator. The detonator/booster assembly is placed in the center of the spherical shell, which is subsequently filled with test explosive and sealed. The charge volume was held constant for these tests.

The principle of operation of the barometric calorimeter has been previously described in detail, together with experimental validation (Alves et al., 2011; Kuhl and Reichenbach, 2010). The primary advantage of barometric calorimetry over traditional thermal calorimetric approaches is the ability of a barometric calorimeter to measure the rate at which energy is released by explosives. In the 506-liter barometric calorimeter, pressure records of up to 100 milliseconds are routinely obtained with time resolution of a few microseconds.

The energy delivered to the structure by the first pressure wave is particularly important for predicting the mechanical response of metal plates. For example, Nurick and Martin (1989) examined the mechanical response of circular and rectangular plates to blast pressures, and found that the mechanical response and failure of the plates is very well-correlated with the first positive phase impulse, generated at the plate. The first positive phase impulse is the pressure integrated over time for the duration of the first positive pressure wave developed at the target.

In the barometric calorimeter, the total energy released by the explosive is simultaneously obtained with the time-dependent energy release record. This information is also relevant to structural damage, as late-time combustion can

release sufficient energy to influence deformation and failure in larger, more complex structures (Alves et al, 2011).

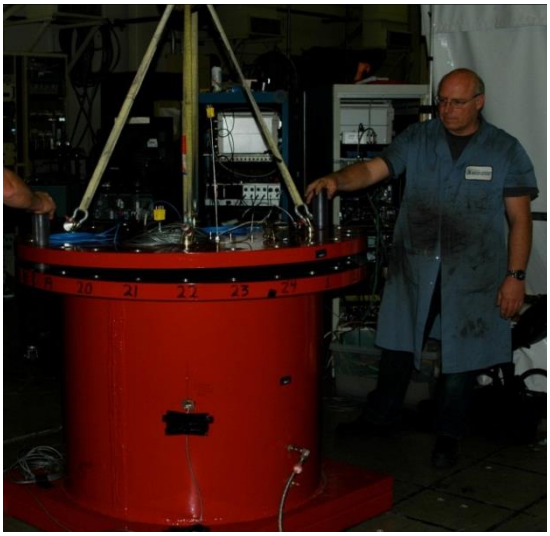

Fig. 4. Lowering the lid onto the 506 L barometric calorimeter.

Results and Discussion

Rate Stick Test Results

The aluminum level for the rate stick mixtures was held constant and the ratio of fuel to oxidizer was varied assuming complete combustion of the aluminum. Detonation velocities were calculated based on the slope of consistent shorting pin data shown in Figure 5. Stoichiometric and fuel-lean mixtures had similar slow velocities while the fuel-rich mixture was much faster. Note that if the assumption was made that the aluminum is inert then the ratio of KC to fuel in the fuel-rich mixture would be considered stoichiometric instead of fuel-rich. Each test also contained a steel witness plate beneath the cylinder of material. KC/fuel/aluminum mixtures showed no observed dent in the witness plate. All of the material reacted in each of the detonability tests.

It is apparent in Figure 5 that the mixtures start out at about the same velocity (the first pin shorts at about the same times) probably due to initially being overdriven by the detonator. The fuel-rich mixture continues to have a consistent propagation velocity indicating a detonation. The shorting pin data for the stoichiometric and fuel-lean mixtures is inconsistent. The pins closer to the detonator are not consistent with the pins further from the detonator. Assuming valid pin data, the velocity appears to transition to a slower rate and then remain relatively consistent toward the bottom of the cylinder, thus indicating deflagration for the fuel-lean and stoichiometric mixtures in 2.54 cm (1 inch) diameter cylinders. These data suggest that the specific fuel-rich mixture of KC/fuel/aluminum gives the best performance in terms of detonation velocity.

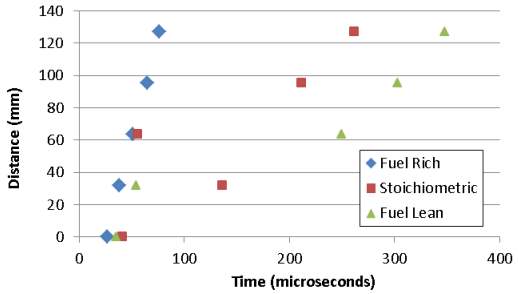

Fig. 5. KC/fuel/aluminum shorting pin data shows that initial pins short at similar times, but later velocities are slower for fuel-lean and stoichiometric mixtures.

High-speed video was recorded for each of the tests and still images from each mixture are shown in Figure 6. The figure's stills are at approximately the same time for comparison between tests. As with the pin data, the images for the stoichiometric mixture and fuel-lean mixture are very similar. With both detonability tests, the reaction front starts out uniform, but as it propagates down the cylinder non-uniform propagation or streaking of the reaction front is observed (this is not observed in the fuel-rich test). Streaking in the fuel-lean mix is not as evident as it is for the stoichiometric mixture due to the masking of the reaction front by the cylinder fixtures. For both stoichiometric and fuel-lean mixtures, the reaction front barely stays ahead of the debris cloud, an indication of slow reaction.

Conversely, the still images from the high-speed video of the fuel-rich mixture, at the bottom of Figure 6, indicate a uniform reaction front, propagating faster than the other mixtures. Here expansion of the cylinder is also apparent.

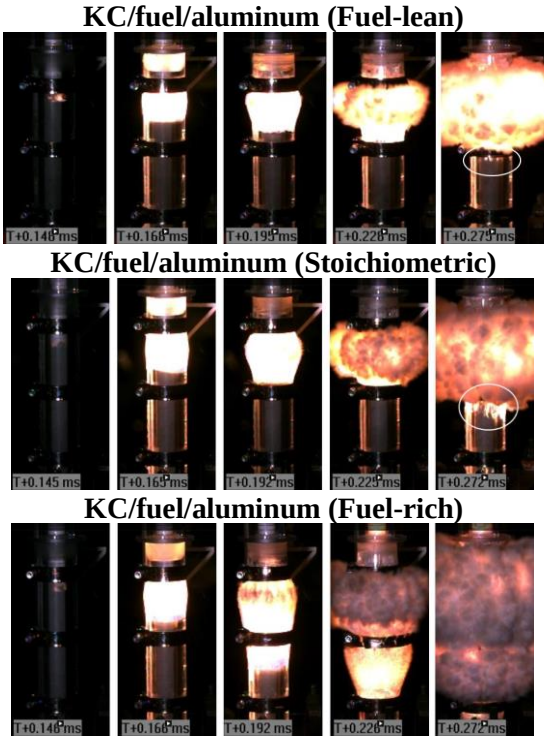

Fig. 6. High-speed video of KC/fuel/aluminum reaction propagating down the cylinders for, from top to bottom, fuel-lean, stoichiometric, and fuel rich mixtures. Note streaking highlighted in final images of the fuel-lean and stoichiometric mixtures.

The detonation velocities measured for KC/fuel/aluminum formulations in the rate sticks can be compared to the detonation velocities calculated using the thermochemical code Cheetah 6.0 (Bastea et al., 2010). The Cheetah velocities are calculated in two ways: one assumes that all of the aluminum reacts and is converted to Al_2O_3 during the detonation; the other assumes that the aluminum is inert and none reacts during the detonation. This is a useful way to analyze aluminum-containing conventional explosives since aluminum may react slowly, contributing to the overall blast but not to the explosive's metal driving ability.

Figure 7 shows this comparison between experiment and Cheetah for the 1 inch rate sticks where the x-axis shows the oxygen balance and the aluminum content is held constant. Oxygen balance is the ratio of the oxygen available in the formulation to the oxygen needed for complete combustion of the fuel. The experimental velocities are consistently lower than the calculated velocities.

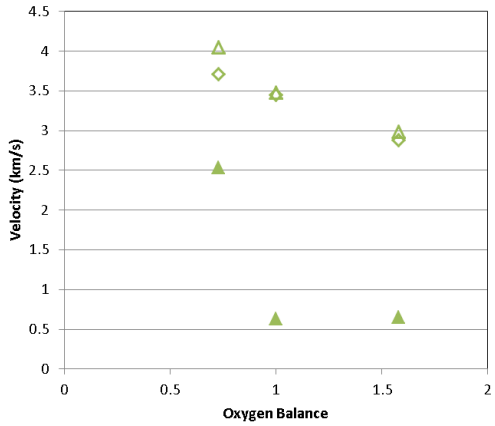

Fig. 7. KC/fuel/Al velocities compared to Cheetah predictions for formulations containing constant aluminum content. Rate stick data are closed points and Cheetah calculations are open points.

The trend in both the experimental data and Cheetah 6.0 calculations is that the detonation velocity decreases as one moves from the fuel-rich formulation to the stoichiometric formulation to the fuel-lean formulation. The stoichiometric and fuel-lean formulations may in fact be deflagrating instead of detonating. Once again the experimental values are lower than the Cheetah values, which is likely due to two factors. First, the experimental velocities are not obtained at infinite radius. Second, the lower measured velocities are probably due in part to the non-ideality of these mixtures, which means that a portion of the energy is released after the passage of the detonation front and therefore does not contribute to the detonation velocity. The non-ideal behavior of formulations

containing aluminum complicates the interpretation of these data.

The analysis is convoluted by finite diameter effects, and if most or all of the differences between the calculated and measured velocities can be accounted for on this basis, the Cheetah values calculated assuming reactive aluminum may be just as accurate, or more accurate than those calculated assuming inert aluminum. Part of the reason for this is the involvement of two different aspects of aluminum combustion—aluminum combustion (1) adds a great deal of energy to the product gases and (2) produces a solid product, alumina. The production of a solid product may be the reason the addition of aluminum does not increase the detonation velocity either experimentally or by calculation. Resolving this issue would require performing additional tests at several rate stick diameters.

Kinetic Plate and Barometric Calorimeter Results

High-speed video frames of a KC kinetic plate experiment is shown in Figure 8. The PDV probe data were analyzed using a Fourier transform spectrogram, and each resulting curve was fit to a purely empirical, modified exponential function that was used in analysis of every shot for consistency. The asymptote of each fit was taken as the maximum plate velocity for that probe. The probe velocities for each plate were combined, decomposing into their separate linear and rotational velocity components, in order to obtain the final linear velocity for each experiment. For each test, uncertainties in the plate speed are calculated from the standard deviation of the four probe velocities after accounting for position and plate spin. The plate speeds reported are determined from the steady-state velocities of the plate, which are generally reached at greater than 230 μs after detonation. To check the fitting procedure, an average of the probe velocities was taken after they reached steady-state values, and these average values generally gave plate velocities within 0.5 m/s of the linear velocity fits.

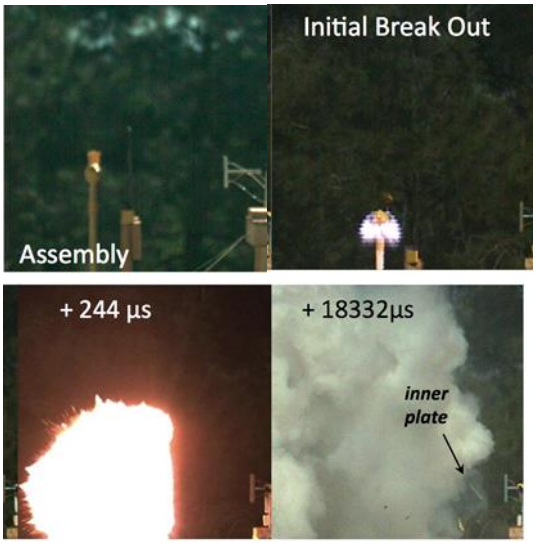

Fig. 8. Progression of a non-aluminized kinetic plate experiment.

Table 1 summarizes the average velocities measured from each experiment with different Al and LiF content. Performance increases with higher aluminum content. The probe velocities from the most Al rich (most fuel rich) and most LiF rich (least overall fuel content) is illustrated in Figure 9, while the impulse from the near-field pressure gages are illustrated in Figure 10. Both data clearly show higher plate velocities and peak pressures for the Al formulations relative to the LiF formulations that increase with fuel richness.

Table 1. Average kinetic plate peak velocities

Mixture	velocity (m/s)	Comment
KC/fuel/high-Al	21	Fuel richest
KC/fuel/low-Al	19	Fuel richer
KC/fuel	18	Stoichiometric
KC/fuel/low-LiF	17	Fuel leaner
KC/fuel/high-LiF	14	Fuel leanest

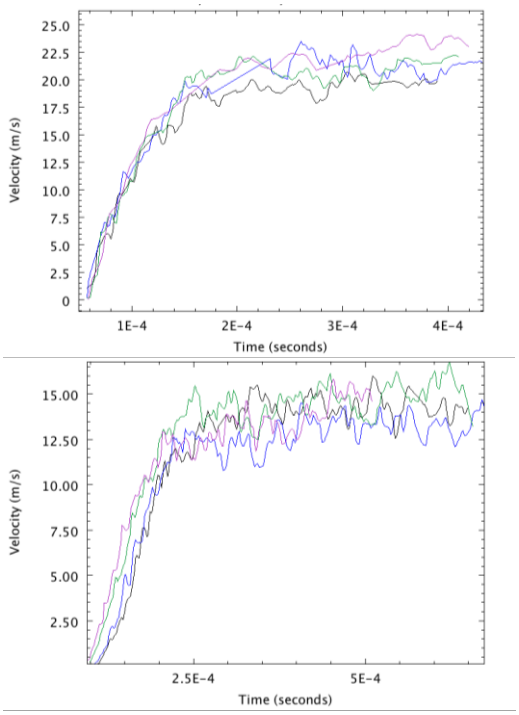

Fig. 9. Sample PDV probe velocities for (top) KC/fuel/aluminum (fuel richest) and (bottom) KC/fuel/LiF (fuel leanest).

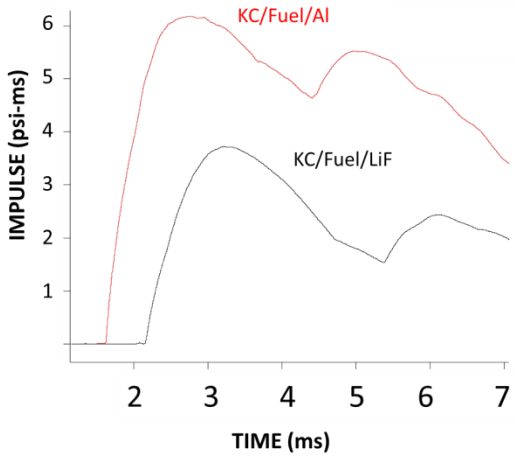

Fig. 10. Impulse associated with near-field pressures at the kinetic plate tests for KC/fuel/Al mixtures (fuel richest, red) and KC/fuel/LiF mixtures (fuel leanest, black).

The barometric calorimeter examined the same solid fuel investigated by the rate stick study described. Multiple pressure probes provided pressure history and, therefore, impulse both at early-time (up to 3 ms) and at late time.

When comparing barometric calorimeter data for the KC/fuels with and without aluminum, additives do not show as clear a difference as the kinetic plate at early time. Overall early time impulse is similar with and without aluminum, though shock arrival of the aluminized materials precedes the mixtures without aluminum (Figure 11). At late times, however, the quasi-static impulse as determined by the barometric calorimeter (not shown) is significantly higher for the aluminized mixture (25% higher for this specific mix).

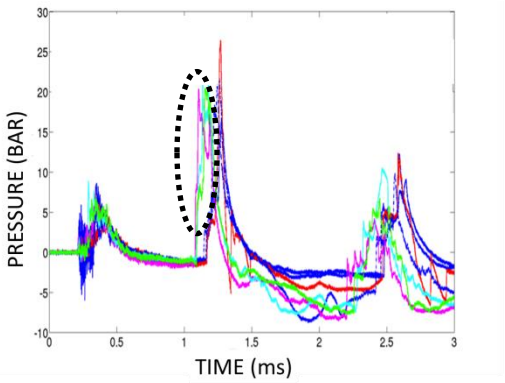

Fig. 11. Pressure time-history of first 3 ms for the 3 aluminized mixes (circled green, turquoise, magenta lines) precede the 4 non-aluminized shocks in the barometric calorimetry study; overall early-time impulse is not significantly different.

Table 2. Relative performance of KC/fuel mixtures with and without aluminum using different methodologies. All relative values are approximate. Note that the fuel used for the rate-stick and barometric experiments calorimeter was a granular fuel mixed with KC, while that used for the kinetic plate experiment was a liquid fuel.

Test/*mix*	*Aluminized KC mixture*			*Non-Aluminized mixture*
	Fuel-rich	*Stoichiometric.*	*Fuel-lean*	*Stoichiometric*
Rate stick	=Baseline	<Baseline	<Baseline	Baseline Velocity
Kin. Plate	>Baseline Impulse			Baseline Impulse
Bar. Cal. (early)	~Baseline Impulse	~Baseline Impulse		Baseline Impulse
Bar. Cal. (late)	>Baseline Impulse	>Baseline Impulse		Baseline Impulse

Conclusions

Blast effects measurements have been made using three experimental methodologies to address different explosive reaction zones. Table 2 summarizes the relative performance of mixtures with and without aluminum. The addition of aluminum to otherwise stoichiometric chlorate mixtures (resulting in fuel rich mixtures) increased performance in some tests but not in others. For the rate stick tests, limited study showed that fuel rich mixtures with aluminum additive have similar detonation velocities to stochiometric mixtures without aluminum; these fuel rich mixtures with aluminum show greater velocities than the aluminized stoichiometric and fuel lean mixtures. As the ratio of aluminum to fuel increases, the detonation velocity decreases. These trends are consistent with Cheetah calculations. The kinetic plate tests showed higher plate velocities for higher fuel and aluminum content when compared to a non-aluminized mixture containing an inert in place of the aluminum. Last, barometric calorimetry showed similar enhancement as observed in the kinetic plate tests, but only at late time. As long as the mixture is oxygen balanced prior to adding the aluminum, there do not appear to be any disadvantages in terms of performance with having aluminum. Those mixtures have equivalent or greater performance than the same mixtures without aluminum.

Acknowledgements

Special thanks to Ron Chambers, Raul Garza, Steven Pease, and Lisa Lauderbach for their work with the testing at the LLNL High Explosives Application Facility (HEAF). We would also like to acknowledge the assistance of Dr Jon Maienschein and Dr Jon Schoonover for their review and leadership, and Ms Byung-Hee Frantz and Mr Jason Tracy for their support of this study. This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344 and Los Alamos National Laboratory under DE-AC52-06NA25396. This paper is LLNL-PROC-655328 and LA-UR-14-24691. The experimental data collection and analysis were performed under sponsorship of the U.S. Department of Homeland Security, Science and Technology Directorate.

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Question
Charles Moore, NASA LSP
Did you do any testing with $KClO_4$ +Al? Do you believe that would detonate as well with Ucj~1.5mm/msec?

Reply by __Lee Glascoe_____
Write response here
No we did not test potassium perchlorate (KClO4) for this study, just potassium chlorates (KClO3). We suspect that the KClO3 tests having very low reaction velocities (less than 2km/sec) were not detonations, but violent deflagrations.

Question
Andrew Laing, QinetiQ
What composition details are you able to share on your energetic materials?

a. Grade of Al
b. Grade of KClO3
c. Exact ratios used

Reply by Lee Glascoe, Sabrina DePiero
Write response here

(a) We used a German black flake grade of aluminum from Skylighter with an average particle size < 3 microns.

(b) We used purified grade powder KClO3 from Columbus Chemical (Product ID 423000-57101).

(c) Unfortunately we are not at liberty to provide the exact mix ratios we used for these tests.