A SMALL-SCALE SCREENING TEST FOR HE PERFORMANCE: APPLICATION TO THE NEW EXPLOSIVE LLM-105*

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It is expensive and time-consuming to scale up the processing of a new explosive molecule or formulation to produce a sufficient quantity of material to characterize the detonation performance. The calculated performance does not always correspond with measured performance, so there is also a concern that the time and resources expended in firing performance tests on a new material may not be justified by the outcome. We have developed a smallscale test that permits a preliminary characterization of the performance of a new explosive using only a few grams of material. We have performed the test on several explosives in common use. These include LX-10 (95% HMX/5% Viton A binder), LX-16 (96% PETN/4% FPC 461 binder) and LX-17 (92.5% TATB/7.5% Kel-F 800 binder). The test results agree well with with calculations which use equations of state that have been measured in cylinder tests¹. We can also directly compare a new material with the test results on these well-known explosives. We have used the new test to characterize an explosive that we have recently synthesized, 2,6-diamino-3,5-dinitropyrazine-1-oxide (LLM-105), an insensitive energetic material with 25% greater power than TATB. The energy content and thermal stability of this material make it very interesting for several applications, including insensitive boosters and detonators. In this paper we will describe the evaluation of LLM-105 with only 30 grams of material.

SCREENING TEST

A schematic of the test fixture is shown in Fig. 1. The test pellet is initiated by an initiation train consisting of an exploding foil initiator (EFI), which initiates a 6.35-mm-diameter, 2-mm-thick LX-16 pellet pressed to a density of 1.7 g/cm³. The detonation of the LX-16 drives a 5.0-mm-diameter, 0.127-mm-thick aluminum flyer plate across a 1-mm gap to impact a 6.35-mm-diameter, 5-mm-thick test pellet. This initiation train provides a sufficient stimulus to promptly initiate ultrafine TATB at a density

of 1.8 g/cm³. For explosives less sensitive than ultrafine TATB we add a booster pellet of LX-10 or LX-14.

The test fixture is steel, consisting of reusable end plates that are bolted together to capture a stack of steel discs. Starting from the bottom of the stack, the first disc protects the bottom end plate and has a slot to contain the EFI and its flat cable. The next disc has a hole in the center, which contains a 6.35-mm-diameter, 2.03-mmthick pellet of LX-16 pressed to a density of 1.7 g/cm³. A 0.127-mm-thick aluminum foil is then placed over the LX-16 pellet and is held in place by the next disc, which is 1-mm-thick and has a 5.08-mm-diameter hole in the center. Next, another containment disc holds the 6.35mm-diameter test pellet. This disc is carefully sized so

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FIGURE 1. CROSS-SECTIONAL SCHEMATIC OF TEST FIXTURE.

that the test pellet protrudes $\sim 25 \mu m$ above the surface of the disc.

A piece of $13-\mu$ m-thick aluminum foil is cut to shape and flattened on a piece of glass with a steel roller. A tiny drop of mineral oil is placed on a LiF crystal and the Al foil is placed on the crystal and flattened by pressing it between a glass cover slip and the crystal. The cover slip is removed and the LiF crystal is placed in the next containment disc and pressed against the slightlyprotruding test pellet with the foil against the pellet. The LiF crystal is held tightly against the test pellet by an Oring, which is compressed by a final disc which protects the top end plate.

The motion of the 13- μ m-thick aluminum foil, which forms the interface between the aluminum foil and the LiF crystal is measured by a Fabry-Perot laser velocimeter², which can be used to measure velocity to within about 1%. In this instrument, a laser beam is focused onto the 13- μ m-thick aluminum. The Doppler shift of the reflected light is analyzed by a Fabry-Perot etalon and recorded with a streak camera. The pressure wave transmitted into the LiF may be determined from the foil velocity and the Hugoniot of the LiF. The laser velocimeter was also used to measure the velocity-time history of the 0.051-mm-thick Kapton flyer from the EFI and the 0.127-mm aluminum flyer accelerated by the detonating LX-16 pellet. The time response of the velocimeter is estimated to be about 10 ns.

Figure 2 shows the timing schematic for the experiment. Times of events are referenced to t_b , the current start in the EFI. A common fiducial signal allows us to relate electrical records to the film records from the laser velocimeter. Zero time is taken to be the start of current in the EFI. The laser velocimeter records the velocity-time history of the flyer from the EFI and the velocity with which it strikes the test pellet. The flight of the aluminum flyer accelerated by the LX-16 pellet is recorded in a subsequent experiment. The time at which the flyer strikes the test pellet is determined by the jump in interface velocity when the flyer is allowed to strike an aluminized glass surface at the position of the impact surface of the pellet. Detonation breakout time from the test pellet is the time at which the 13- μ m-thick aluminum foil starts to move.

Detonation velocity is of interest in evaluating a new material. In our test, we can determine the transit time of the detonation through the test pellet from the difference between the impact time of the aluminum flyer on the test pellet and the detonation breakout time at the center of the pellet. We assume that the time from bridgefoil burst to the impact of the aluminum flyer on the test pellet is constant but, of course, there is some jitter due to variations in the EFI function time and to variations in the density of the LX-16 pellet. We estimate this jitter to be $\sim \pm 0.02 \ \mu s$. For an explosive with a detonation velocity of 8 km/s, this uncertainty leads to a relative error in velocity of about $\pm 3\%$. With a 1- μ s-long velocimeter record, we can read the time to within $\sim \pm 0.01 \ \mu s$, so we estimate a total error of about $\pm 5\%$ in determining the detonation velocity from the transit time through the test pellet.



FIGURE 2. TIMING SCHEMATIC FOR THE EXPERIMENTS.

DISCUSSION OF EXPERIMENTAL RESULTS FOR STANDARD EXPLOSIVE SAMPLES

Experimental results for the explosive LX-10 are shown in Fig. 3. The experimental interface velocity data, shown as gray lines, are from six different shots and the scatter in the curves shows the reproducibility of the measurement. The solid black line is a calculation using the CALE hydrocode³ and a JWL equation of state¹, determined from cylinder tests. The agreement is good except near the peak at the shock arrival. The fact that the measured peak velocity is larger than the calculated peak velocity may be due to the Von Neumann spike, which has not been completely attenuated in its passage through the 0.13-mm Al foil. There is also more scatter in the experimental data near the shock jump, due to difficulties which will be discussed below.

Figure 4 shows an experimental streak record of fringes from the laser velocimeter record as the detonation wave collides with the interface. The interface velocity is determined from the spacing between the fringes, and at the shock jump, the error is greater than the 1% error we estimate for the bulk of the record. This is because the fringe spacing is decreasing rapidly after the shock jump which, coupled with the finite width of the fringes, makes the peak value very difficult to read. This difficulty can be overcome by streaking faster, so the slope of the fringes is reduced.

Figure 5 shows the six experimental records from Fig. 3 separated in time. The first two records were recorded at a 2 μ s sweep rate and the last four at 1 μ s. There is a consistent trend for the faster sweeps to give higher peaks. At the time of the experiments we were

limited to a 1 μ s sweep because we did not have a comb generator that would generate time marks faster than every 100 ns. This meant that at sweeps faster than 1 μ s, we could not generate an accurate time fiducial to give us cross timing with the digitizer records of the electrical signals. In future work we will use a faster comb generator and, for some experiments, plan to use a much thinner aluminum reflector in conjunction with a sub-ns-time-resolution VISAR velocimeter.

LX-16 has a very small reaction zone and one would expect it to behave even more ideally than LX-10. Figure 6 compares experimental data from three shots with a CALE calculation. Here, the calculation actually overpredicts the peak, but gives good agreement at later times. One of the experimental curves is clearly defective near the peak and this is probably due to an assembly problem that resulted in a small gap or a bubble in the assembly.

Finally, in Fig. 7, we show a comparison of calculation with experimental data for LX-17, a very nonideal explosive that is known to have a large reaction zone. The LX-17 could not be initiated directly by the aluminum flyer, so an additional booster of LX-10 was used. The simulation of this experiment included the LX-10 booster. The experimental curves lie above the calculated curve for more than 0.1 μ s, consistent with the existence of a large reaction zone. The generallypoor agreement between experiment and calculation at later times may indicate that the detonation in the LX-17 has not yet reached steady state, indeed, the diameter of the pellet may be below the critical diameter for LX-17.

The agreement between the data and the CALE simulations for nearly-ideal explosives shows that our test gives a good representation of the performance of an explosive in the high-pressure region near the C-J state but not too close to the detonation front. Near the detonation front, the simulation usually gives a lower interface velocity than is measured, probably a result of the Von Neumann spike, which is not accounted for in the simulations. There is also more experimental uncertainty in the measurement of the shock jump, but this can be alleviated by a faster sweep rate on the camera which records the velocimeter data.



FIGURE 3. VELOCITY-TIME RECORDS FOR 6 DIFFERENT SHOTS WITH LX-10 EXPLOSIVE, COMPARED WITH A SIMULATION USING THE CALE HYDROCODE³.

Agreement between experiment and simulation for the non-ideal explosive, LX-17, was much poorer. LX-17 has a very large reaction zone, which would lead to a wide Von Neumann spike and an under-prediction by the simulation near the detonation front. Also, if the LX-17 detonation has not reached steady state, one would not expect agreement with the simulation at later times.

EXPERIMENTAL TESTING OF LLM-1054

We have synthesized 2,6-diamino-3,5-dinitropyrazine-1-oxide (LLM-105), as an insensitive target molecule. The predicted power⁵ is 125% that of the extremely insensitive explosive TATB. The energy content, power and thermal stability of LLM-105 make it very promising for several applications, including insensitive boosters and detonators. As an initial screening we produced \approx 30 grams of LLM-105 for characterization by the new performance test and small-scale thermal explosion experiments. LLM-105 has a density of 1.913 g/cc. In differential scanning calorimetry the primary exotherm occurs at 342°C at a sweep rate of 10°C/min (vs. \approx 320°C for HNS and \approx 355°C for TATB). Drop impact height is \approx 120 cm versus 30–32 cm for RDX and HMX.

LLM-105 parts with adequate mechanical integrity could not be made by pressing neat material and, therefore, was formulated with 5 wt% Viton A. The crystal morphology of LLM-105 was needle-like in these experiments. This morphology contributed to difficulties in obtaining high pressing densities, only 92.4% of the theoretical maximum density was obtained.

SMALL-SCALE PERFORMANCE TEST

Early in the evaluation of LLM-105, we used the new detonation test to determine whether the LLM-105 molecule was performing according to expectations.

Figure 8 shows the interface velocity record compared with a CALE hydrocode simulation for LLM-105 (neat) pressed to a density of 1.72 g/cm³. The equation of state used for the simulation was determined using the CHEETAH chemical equilibrium code⁵, and the agreement is quite reasonable, indicating that expectations had been met.

ONE-DIMENSIONAL, TIME-TO-EXPLOSION TESTING OF LLM-105

As part of the small-scale testing protocol the thermal response of LLM-105 was evaluated in the onedimensional, time-to-explosion test (ODTX)⁶. In this experiment 12.7 mm spheres are rapidly inserted between two aluminum anvils, preheated to a fixed temperature. The time-to-explosion response is determined quantitatively and the violence of reaction qualitatively.

Figure 9 shows the ODTX results obtained with the LLM-105 formulation and other common materials. TATB is extremely insensitive to thermal explosions. It can be seen that LLM-105 has a time-to-explosion which is between HMX and TATB and better that HNS-IV. Unlike HMX all reactions with LLM-105 were very mild, i.e., TATB like. Due to limited material a precise determination of the critical temperature of LLM-105 could not be made. No explosive response was observed with LLM-105 at the critical temperature of HMX. LLM-05 may have has a critical temperature as much as 15°C higher than HMX.



FIGURE 4. STREAK RECORD OF VELOCIMETER FRINGES. INTERFACE VELOCITY IS DETERMINED FROM THE SPACING BETWEEN THE FRINGES. IT IS MORE DIFFICULT TO DETERMINE THE FRINGE SPACING AT THE SHOCK JUMP THAN FOR THE REST OF THE RECORD.



FIGURE 5. SIX EXPERIMENTAL LX-10 RECORDS, SEPARATED IN TIME. THE FIRST TWO RECORDS WERE RECORDED WITH A 2 μ s SWEEP, THE LAST FOUR WITH A 1 μ s SWEEP.



FIGURE 6. THREE EXPERIMENTAL VELOCITY-TIME RECORDS FOR LX-16 EXPLOSIVE COMPARED WITH A SIMULATION USING THE CALE HYDROCODE³.



FIGURE 7. THREE EXPERIMENTAL VELOCITY-TIME RECORDS FOR LX-17 EXPLOSIVE COMPARED WITH A SIMULATION USING THE CALE HYDROCODE³.

SUMMARY

We have developed a small-scale test that permits preliminary characterization of the performance of a new explosive using only a few grams of material. We have performed the test on several explosives in common use. These include LX-10 (95% HMX/5% Viton A binder), LX-16 (96% PETN/4% FPC 461 binder) and LX-17 (92.5% TATB/7.5% Kel-F 800 binder). This allows us to compare the experimental results with calculations which use equations of state that have been measured



FIGURE 8. EXPERIMENTAL VELOCITY-TIME RECORD FOR LLM-105 EXPLOSIVE COMPARED WITH A SIMULATION USING THE CALE HYDROCODE³.



FIGURE 9. ODTX RESULTS OBTAINED FOR THE LLM-105 FORMULATION.

in cylinder tests and to directly compare LLM-105 with the other explosives using the same test.

We have synthesized the interesting, new insensitive energetic material, LLM-105. With only 30 g of LLM-105 we were able to characterize the material adequately to evaluate it for future applications. We have used the new, small-scale test to confirm that LLM-105 has 25% more energy than TATB. In the ODTX configuration LLM-105 was found to be very thermal stable and produces mild, low-order explosions. These experiments indicate LLM-105 may have uses in initiation systems and as a main charge material. Further scale-up appears to be warranted.

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DISCUSSION

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What are the solvents which dissolve the new explosive LLM-105?

REPLY BY JACK L. CUTTING, RONALD S. LEE AND RANDALL L. SIMPSON

LLM-105 is generally not soluable in most solvents, however, DMSO will dissolve LLM-105. Extreme care should be taken as DMSO spray is ignitable.

DISCUSSION

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What was the measured detonation velocity of LLM-105 from these tests?

REPLY BY JACK L. CUTTING, RONALD S. LEE AND RANDALL L. SIMPSON

The predicted detonation velocity at crystal density is 8.56 mm/ μ s. In shock loading studies on RX-55-AA (95% LLM-105 with 5% Viton-A) we measured 7.98 mm/ μ s at 93% TMD. We also measured a detonation velocity at 7.8 mm/ μ s in another experiment, but the density of the explosive was not precisely known.