LA-UR- 10-01430

Approved for public release; distribution is unlimited.

Title: Material properties effects on the detonation spreading and propagation of Diaminoazoxyfurazan (DAAF)

Author(s): Elizabeth G Francois
John S Morris
Alan M Novak
James E Kennedy

Intended for: 14th International Detonation Symposium
April 11-16, 2010, Coeur D'Alene Idaho

Los Alamos NATIONAL LABORATORY EST. 1943

Material properties effects on the detonation spreading and propagation of Diaminoazoxyfurazan (DAAF)

Elizabeth G. Francois[*], John S. Morris[*], Alan M. Novak[*], James E. Kennedy[f]

[*]DE Division, Los Alamos National Laboratory,
Los Alamos, NM 87545, USA

[f]Hazards & Explosives Research & Education LLC
Santa Fe, New Mexico 87505, USA

Abstract. Recent dynamic testing of Diaminoazoxyfurazan (DAAF) has focused on understanding the material properties affecting the detonation propagation, spreading, behavior and symmetry. Small scale gap testing and wedge testing focus on the sensitivity to shock with the gap test including the effects of particle size and density. Floret testing investigates the detonation spreading as it is affected by particle size, density, and binder content. The polyrho testing illustrates the effects of density and binder content on the detonation velocity. Finally the detonation spreading effect can be most dramatically seen in the Mushroom and Onionskin tests where the variations due to density gradients, pressing methods and geometry can be seen on the wave breakout behavior.

Introduction

High nitrogen molecules have been a rich area of research and yielded many interesting explosives. One that has found significant attention is DAAF, which has proven perplexing with its dichotomous traits: insensitivity to impact and friction, but sensitivity to shock. Interestingly, this combination of properties is extremely favorable; DAAF can be easily initiated when required, but remains safe to normal insults. The insensitivity of DAAF as seen in small scale testing show it to be similar to triaminotrinitrobenzene (TATB), but DAAF has advantageous performance characteristics. Early in DAAF research, its critical diameter was measured to be less than 3mm, but the exact value was unknown. This was an unprecedented result for an insensitive high explosive (IHE). Wedge testing (Figure 1) and small scale gap testing (Table 1) showed DAAF to exhibit HMX-like sensitivity to shock and a similar run distance to detonation. Recent DAAF dynamic testing has focused on understanding the material properties affecting the detonation propagation, spreading, behavior and symmetry. Small scale gap testing and wedge testing focus on the sensitivity to shock with the gap test including the effects of particle size and density. The floret test focuses on the detonation spreading as it is affected by particle size, density, and binder content. The polyrho testing illustrates the effects of density and binder content on the detonation velocity. Finally the detonation spreading effect can be most dramatically seen in the Mushroom and Onionskin tests where the variations due to density gradients, pressing methods and geometry can be seen on the wave breakout behavior.

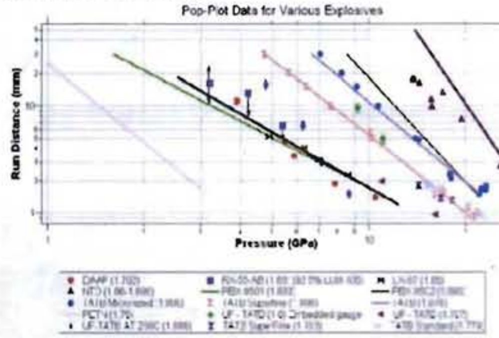

Figure 1: Pop-Plot comparing explosives.

LANL Small Scale Gap Test

The run distance to detonation of DAAF was evaluated in 2000 by Larry Hill et al [1] and is shown in Figure 1. This was interesting behavior for something as insensitive as DAAF. To corroborate this behavior, we performed the Los Alamos National Laboratory small-scale gap tests (LANL SSGT) in 2008 [2]. The goal was to quantify the shock sensitivity using the gap test, instrument the test so observations on detonation velocity and behavior could be collected, gain knowledge on the shock wave behavior in this test, and relate this to the pressure required to initiate DAAF. The LANL SSGT is shown in Figure 2 and measures the brass gap thickness required to allow an acceptor stack of explosive to "go" 50% of the time given a known input stimulus from a RP-1 detonator and a PBX 9407 (94% RDX, 6% oxy-461) donor pellet. It was desirable to be able to correlate the statistical data gathered to previous LANL SSGT data for other common explosives. For these experiments DAAF of three particle sizes (80 μm, 40 μm and <5 μm), was pressed to two densities: 97% TMD-high density and 91% TMD-low density and tested for shock sensitivity. The smallest particle size DAAF: <5 μm, could not be pressed above 91% TMD and was not included in the high density tests.

To investigate the shock velocity and pressure required to initiate DAAF, a custom fixture was designed. The detonator, donor explosive, and brass gap were the same as those used in the gap test; the area where the acceptor is normally placed was filled in with solid polymethyl methacrylate (PMMA). When backlit, the shockwave through the PMMA could be seen (Figure 3). The velocity was measured from the streak image, and used to construct P-u Hugoniots for brass and relate them to PMMA and DAAF (Figure 4).

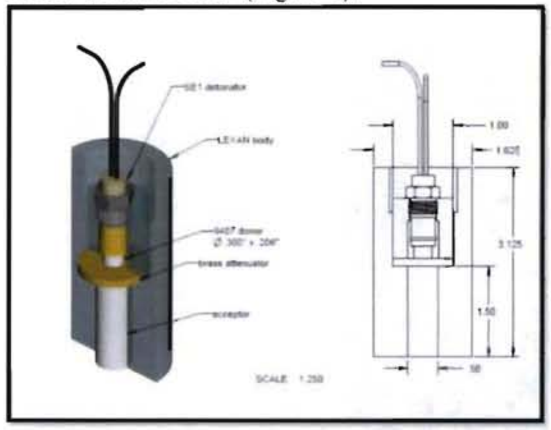

Figure 2: LANL SSGT fixture. For shockwave experiments, the acceptor pellet section of the fixture was absent and area was solid PMMA.

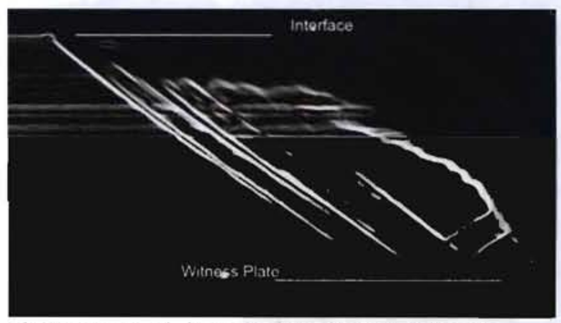

Figure 3: Streak image of shockwave through PMMA. For various gap thicknesses this image was taken. The velocity at the brass-PMMA interface can be evaluated and compared as a function of brass gap thickness.

Gap Test Setup

The donor explosive is a 0.300 inch x 0.207 inch PBX 9407 pellet initiated by a RP-1 detonator and the spacers are solid brass shims. The brass thickness for gap testing varies in 0.25mm increments. Results are reported in spacer thickness(mm) as analyzed by a shortened Bruceton method [3]. The fixture was placed on a

4-inch square by 1-inch tall block of mild steel. A dent in the witness block demonstrated a detonation. Three pellets of ½" x ½" acceptor explosive were used. The mass of pellets was held constant at 2.70 +/- 0.1 grams, and the force applied varied the density. Five series of experiments were run, varying density and particle size.

Table 1: LANL SSGT results for DAAF and compared to other common explosives. Initiation pressures from DAAF P-u Hugoniot (Figure 4).

Material	Density (g/cc)	50% point (mm)	Initiation Pressure (GPa)
DAAF (40μm)	1.69	1.91	**8.3**
	1.59	3.12	**4.5**
DAAF (80μm)	1.69	2.74	**4.8**
	1.59	3.35	**4.5**
DAAF (<5μm)	1.69	NA	
	1.59	3.02	**4.5**
HMX (hot pressed)	1.84	3.43	
	1.79	4.27	
PBX 9501 (hot pressed)	1.843	1.3-1.8	
	1.825	1.52	
PETN	1.757	5.21	
RDX (hot pressed)	1.735	5.18	

Results

At high density (97% TMD) DAAF is less sensitive to shock, and the difference between the 80-micron DAAF and the 40-micron DAAF was dramatic (see Table 1). At lower density (91% TMD) all materials were more sensitive than the high-density series, and slight differences based on particle size could be seen. The 50% point indicates a relative measure of the acceptor material's sensitivity to shock; the thinner the gap, the less sensitive the material. Table 1 shows DAAF and a number of well-characterized explosives. HMX and PBX 9501 are included largely because Pop-plot data for DAAF (Figure 1) has shown it to be similar to HMX in sensitivity to shock, and it can be seen that the brass gap thickness is comparable for low density DAAF and HMX. It is interesting to note that the brass gap thickness for PBX 9501 is around half the thickness for HMX, indicating that the 5% addition of binder has a dramatic effect on shock sensitivity. Future gap tests on DAAF will include a binder, and the desensitizing effects of binder were seen in the floret testing.

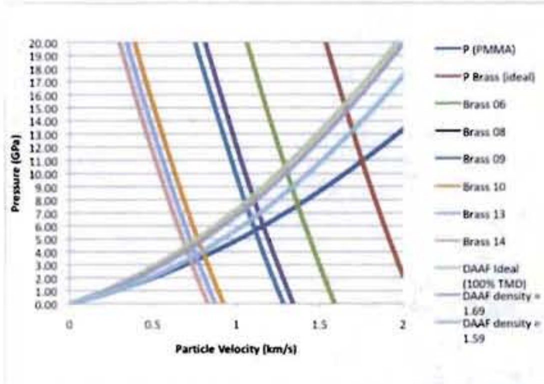

Figure 4: P-u Hugoniot for brass-PMMA-DAAF interactions.

Floret Test

The floret test is a valuable measure of an explosive's detonation spreading or "corner turning" as a function of particle size, density, binder content and other parameters. It is advantageous especially in early explosive development because it uses tiny amounts of material to evaluate detonation spreading. The test consists of an explosively driven flyer of variable diameter, which initiates the test explosive (Figure 5). The completeness of the reaction is measured by a dent profile in a copper block. The floret test has proven exceptionally useful when comparing IHE materials like TATB, PBX 9502, LX-17, UFTATB and all its incarnations [4]. The success seen with different particle size effects in TATB led us to believe this could complement particle size work done with the LANL SSGT on DAAF. In addition, DAAF had been formulated with a variety of binders and amounts, and we wanted to observe the effects of binder content on corner turning. In an effort to understand the effects of thermal and mechanical damage on PBX 9501, this test was redesigned to provide relevant data for more sensitive explosives than previously tested.

Experimental Set-up

Owing to the expectation that DAAF and PBX 9501 would effectively turn corners and had small critical diameters, this test series utilized a half-scale fixture with a 4 mm diameter x 2 mm PETN donor pellet and a 6.35 mm diameter x 2 mm acceptor pellet of DAAF and employed both stainless steel and aluminum flyers in the size range of 1-2 mm in diameter and 5 mil thick. The initiation train is started with a slapper detonator called a "blue light special" employing a 6mil bridge. The slapper initiates a 2 mm x 4 mm PETN pellet pressed to a density of 1.65 g/cc. The cutter is made of steel and has a barrel length of 1 mm.

The initial series of tests used an aluminum flyer 1mm in diameter to establish the lower bound of the experimental range. DAAF Parameters varied in the experiments included density: 97% TMD and 91% TMD; binder: neat vs. 0.5% FK-800 3% FK-800 and 5% Viton; and particle size: 40 µm, <5 µm, and a 3:1 coarse to fine mix. Unfortunately all examples detonated completely as evidenced by similar dent depth and shape and the observation that no DAAF remained in the fixture after the explosion. These results suggest that DAAF has a much smaller critical diameter than previously thought.

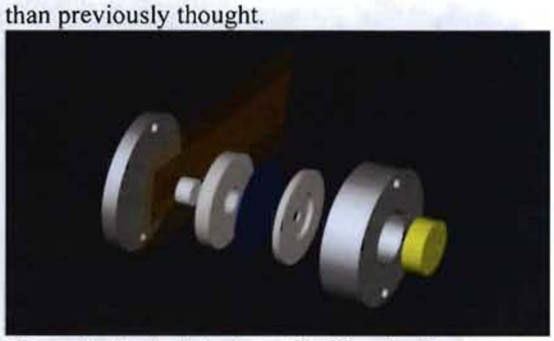

Figure 5: Exploded view of half-scale floret test.

Results

The critical diameter of PBX 9501 is well known [5] to be 1.52 mm. In order to develop a baseline for future damaged PBX 9501 floret tests, a series of tests were performed on pellets pressed to a density of 1.834 g/cc and employed both stainless steel and aluminum flyers at diameters of 1 mm, 1.5 mm and 2 mm. The results can be seen in Figure 6 and look promising owing to the fact that because the critical diameter is known, the "sweet spot" where density and material differences can be observed is at the same flyer size as the critical diameter. It is interesting to note that this holds true for stainless steel flyers only. Aluminum flyers exhibit a similar failing detonation at a smaller diameter: 1 mm in the case of PBX 9501 as opposed to 1.5 mm for stainless steel. Based on this, we chose a stainless steel flyer for DAAF tests in the hope that a failing detonation would be observed at a flyer size of 1 mm or greater.

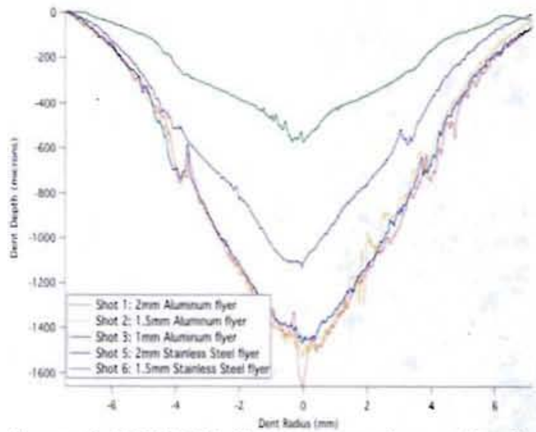

Figure 6: PBX 9501 floret test results varying flyer size and material.

Subsequent tests on DAAF and stainless steel fliers showed promise as the 1 mm flyer did indeed yield a failing detonation in all shots. The dent depths and shapes of all shots were too identical to yield any useful information except perhaps the fact that the detonation is failing too well and the critical diameter is greater than 1 mm and less than 1.5 mm; the sweet spot for measuring the material effects on corner turning has not yet been found and further testing configuration development is needed.

A study of binder amounts revealed that increasing the binder from none (neat) to 5 wt% required a larger stainless steel flyer: 1.5 mm as opposed to 1 mm, but still showed the same dent shape. Increasing the flyer size to 2 mm allowed a complete detonation. Once again the "sweet spot" for a meaningful measurement has not been found, but the trends are evident.

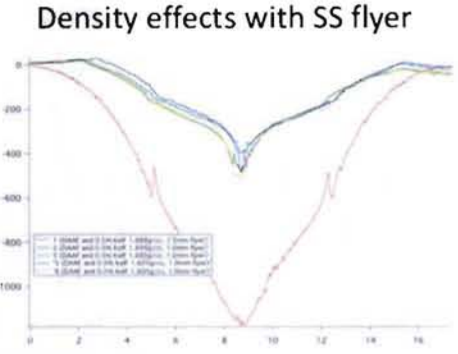

Figure 7: DAAF floret test results comparing densities at 1.69g/cc to 1.60 g/cc.

Floret Test Conclusions

Previous critical diameter experiments on DAAF had determined the critical diameter to be less that 3 mm due to the fact that these tests were performed using rate sticks. The smallest die available to press pellets was 3 mm and when rate stick results were analyzed using dent analysis and streak records, no evidence of a failing detonation was seen. The capability to make smaller rate sticks was not explored and the critical diameter remained unknown. We now propose that the critical diameter of DAAF is between 1mm and 1.5 mm: much smaller than would be expected for a material that behaves like an IHE in small scale sensitivity testing.

With the current configuration of the half-scale floret test, a flyer diameter yielding useful divergence has not been found. All shots with a diameter of 1.5 mm detonate completely and all tests with a 1 mm flyer show identical divergence even when the density is varied by a large amount. In most floret tests, a strong dependence of density on energy output and divergence behavior can be easily seen (6). Furthermore, each of the DAAF tests where the detonation was incomplete showed a small hole blown through the DAAF pellet, and a largely intact donut of remaining material. This suggest several directions for future tests: 1) use a flyer of diameter 1.25 mm to impart more energy to the acceptor and increase the output; 2) Change the dimensions of the acceptor pellet to have more height and allow the DAAF more material in which to develop a distinct and measurable output, and 3) slow the flyer down using a less dense PETN pellet, allowing the flyer to impart less energy and use a larger flyer.

PolyRho Testing Improvements and Results

The polyrho test is widely used to characterize density effects on detonation velocity of new and known explosives. It has proven useful because it uses a small amount of explosive to measure a critical performance trait. A wide variety of densities can be tested with a small amount of explosive through the polyrho test, and this information can be used for Cheetah modeling and to develop the density versus velocity relationship.

The original test used single pellets of each density with the claim that 12 to 15 different densities could be tested (7). They were arranged from low to high density and initiated with an SE-1 detonator. The 0.0254 mm foil switches were comprised of two thin parallel copper conductors backed by Kapton tape and were placed between each pellet. The detonation wave causes the conductors to short together, closing the switch. The results of these foil switches can bee seen in Figure 8, and were noisy and difficult to read.

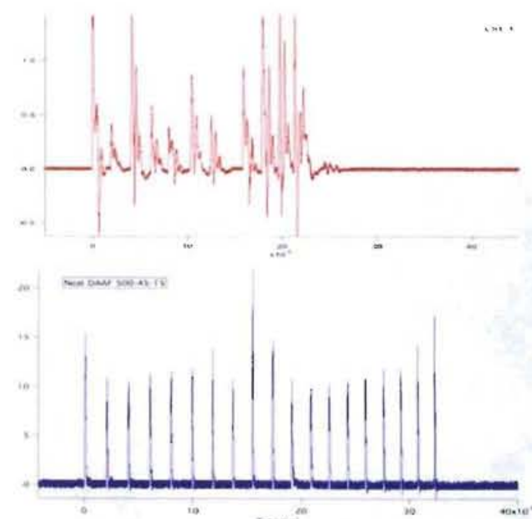

Figure 8: Scope data comparing foil switches (upper trace) to transformer wire (bottom trace)

Test Improvements

Owing to the difficulty analyzing the historic polyrho test's data and the difficulty in reading the switch data, the test was redesigned. One density pellet per measurement was too uncertain, so two similar pellets measured each representative density. Low-density velocities did not agree well with Cheetah and we thought that the PBX 9407 pellet was overdriving the first six pellets. Streak imaging was employed to prove or disprove this assumption. Additionally, the data from the streak image could be analyzed to corroborate the switch data. Later tests used 3-4 lowest density pellets to allow the detonation to approach steady state. A new fixture was designed to hold twenty pellets (see Figure 9). The foil switches were replaced with .002" transformer wire with a polyamide coating. The comparison of the time of arrival scope data from the foil switches and the transformer wire can be seen in Figure 8. The wire was sandwiched between each pellet and the pellets were stuck together with vacuum grease. Everything was held together with the detonator holder screw. The shot was initiated with an RP-2 detonator.

The formulations included in the new polyrho experiments were neat DAAF, DAAF and 0.5% FK-800, and DAAF and 3% FK-800. Two pellets identical to within 0.002 g/cc represented each density. The shots were painted with aluminum fluorosilicate paint to enhance the amount of light to the streak camera. This greatly improved the clarity of the streak image.

Figure 9: New polyrho fixture.

Polyrho Results

Figure 10 shows the velocity vs. density results for the three DAAF formulations tested. The effect of adding a larger amount of binder can clearly be seen with the velocity being compromised at higher density. The 0.5% binder formulation showed very little effect of the binder on the velocity, but it was the most difficult formulation to press. Interestingly, neat DAAF was significantly easier to press; the pellets held together and it was possible create sturdy pressed pieces. The 0.5% formulation did not hold together at the low %TMD pressings, and the corners broke off the pellets with little provocation. It is curious that a small amount of binder is worse than no binder at all at low densities. At higher densities the 0.5% FK-800 formulation pressed acceptably well.

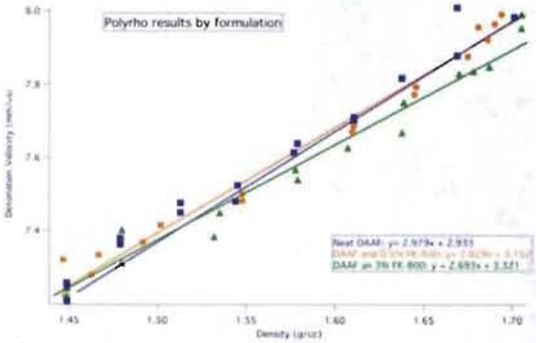

Figure 10: Polyrho results for each DAAF formulation. DAAF and 3% FK-800 shows a much greater reduction of velocity at high density than the other two formulations.

With these data at different densities, a valuable opportunity to validate Cheetah exists. These switch and streak data were compared to Cheetah 5.0 calculations from all libraries. There was poor agreement with all. Figure 10 show the polyrho data for neat DAAF and library exemplifying the closest fit: BKWC. The agreement between model and reality is poor, with the low-density velocities being worse than the high density ones with neat DAAF. Simply having this information to evaluate Cheetah for high nitrogen molecules like DAAF is useful, where the model may not adequately represent reality. Knowing the extent of the modeling/reality discrepancy is invaluable.

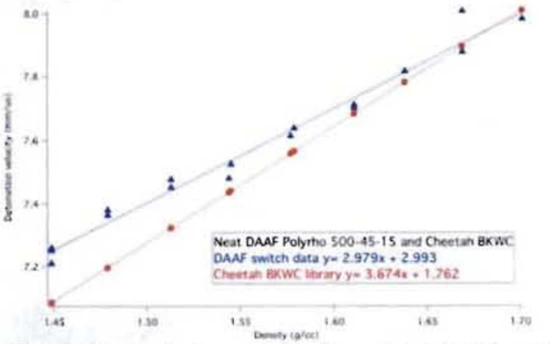

Figure 11: Polyrho results for neat DAAF and compared to Cheetah 5.0 BKWC library results.

Mushroom test

A hemispherical wave breakout test termed the Onionskin test is one of the methods of evaluating the performance of booster materials used in an initiation train assembly. In the Onionskin test, the wave breakout time-position history at the surface of a hemispherical IHE acceptor charge is recorded using a streak camera. The relative uniformity of breakout is qualitatively compared between booster materials as well as quantitative comparison of other performance metrics such as breakout angle, spreading efficiency, excess transit time and apparent center of initiation.

Because of interesting results seen in recent onionskin tests, we sought a quick test to validate theories about material issues, pellet density issues or assembly issues. Figure 11 shows a representative DAAF onionskin utilizing a hemispherical booster configuration and should be compared to Figure 12, which shows LX-07 breakout. The DAAF breakout is asymmetric and the theories surrounding this behavior include density variations in the binder/explosive distribution from the formulation method, pressing issues where density gradients are introduced, or assembly issues where gaps or improper adhesive distribution are causing the breakout to exhibit asymmetric behavior.

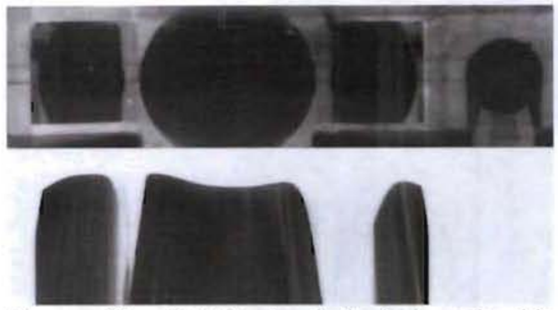

Figure 12: DAAF hemispherical onionskin showing asymmetric breakout.

Figure 13: LX-07 hemispherical onionskin showing symmetric breakout.

The Onionskin test is expensive and involved. While it provides a valuable qualitative and quantitative measure, it does not lend itself to investigation of subtle material changes. The thickness of the surrounding PBX 9502 (95% TATB, 5% FK-800) can wash out or obscure subtle booster variations. To simplify this test and investigate the effects of density, the Mushroom test was chosen because it also measures breakout angle at -55°C but does not require the addition of the PBX 9502 shell. We designed a new Mushroom test fixture (Figure 13) to allow the flexibility of firing 30mm pellets or 1-inch pellets in either hemispherical or cylindrical geometries. It utilized a stem of ¼" PBX 9407 pellets and is initiated with an RP-2 detonator similar to the historic Mushroom test (8). The stem streak record is used as a fiducial for detonation arrival at the test pellet of interest.

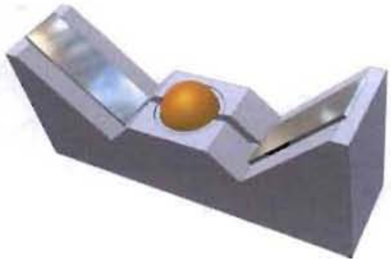

Figure 14: Modified Mushroom test fixture. Fixture is made out of cast aluminum for dimensional stability at low temperature. PBX 9407 stem behavior is observed through side mirrors.

The goal of these experiments was to introduce purposeful density gradients, observe the effect on the breakout, and relate this back to the Onionskin test. Binder/formulation effects were included by comparing the response of pressed hemispheres of DAAF and 3% FK-800, and neat DAAF. LX-07 was included as a benchmark. Seven tests were completed, and are outlined in Table 2. The first breakout angle is the response of interest. In the case of a hemispherical shot, it corresponds to the location where light first breaks out and is measured from the pole. Ideal breakout would be at the equator. In the case of a cylindrical pellet, breakout is measured on the pellet face. It propagates from the center to the edges. The density effects are measured by velocity changes in the streak record.

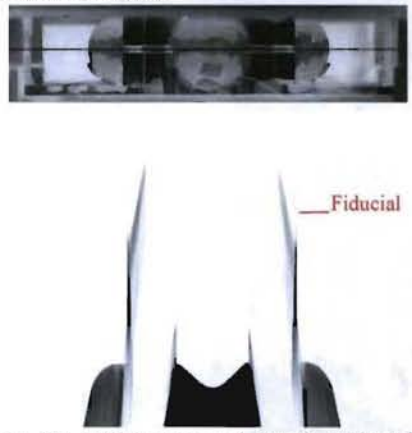

Figure 15: Still image and streak record from neat DAAF hemispherical shot. The fiducial is chosen from where the PBX 9407 stem contacts the hemispherical pellet.

Table 2: Modified Mushroom test shot series.

Pellet details	Temperature	Density details	Response
Shot 1: 1" x 1" cylinder. DAAF and 3% FK 800	Ambient	High-density (1.69 g/cc) ½" x ½" pellet inside low density (1.62g/cc) 1"x1" pellet. High-density pellet offset to one side.	Faster breakout on high-density side of pellet face. See Figure 15.
Shot 2: 1" x 1" cylinder. DAAF and 3% FK 800.	-55°C	High-density (1.69 g/cc) ½" x ½" pellet inside low density (1.62g/cc) 1"x1" pellet. High-density pellet offset to one side.	Identical response as experiment 1. No temperature effect. See Figure 15.
Shot 3: 30mm hemisphere of DAAF and 3% FK-800	-55°C	Density = 1.678 g/cc. No introduced density gradients.	Symmetrical breakout. FBA = 72.56 degrees
Shot 4: 1" x ½" cylinder. DAAF and 3% FK 800.	-55°C	½" x ½" high density (1.69g/cc) pellet inside 1" x ½" donut of low density (1.62 g/cc)	Breakout on center of face faster than on edges.
Shot 5: 1" x 1" cylinder. DAAF and 3% FK 800	-55°C	Material unleveled in die before pressing. More DAAF on one side of die and therefore higher density on that side.	Results inconclusive
Shot 6: 30mm hemisphere. LX-07	-55°C	Density = 1.843 g/cc. No introduced density gradients.	Symmetrical breakout. FBA = 81.5 degrees
Shot 7: 30mm hemisphere. Neat DAAF	-55°C	Density = 1.67g/cc. No introduced density gradients.	Symmetrical breakout. FBA = 71.35 degrees. See Figure 14.

Results

The three hemispherical tests all broke out symmetrically. The DAAF and 3% FK-800 pellet was from the same lot as the previous onionskin test exhibiting asymmetric breakout. No observable difference existed between neat DAAF and formulated DAAF suggesting that the binder is not introducing density gradients. The streak record for neat DAAF hemisphere is shown in Figure 4. The location of the end of the stem streak is used as a fiducial.

Shot 1 and 2 compared ambient response to cold temperature response. Both pellets contained a high-density region within a low-density pellet. The high density and low-density regions were 4% different in density; quite a large gradient for a pressed pellet, and not reasonably achievable under normal pressing conditions. While this did show a slower breakout on the low-density side, this is not likely to be a possible explanation for the observed behavior as seen in Figure 12. Temperature seems to have no effect on the breakout as shown in Figure 16. This is tremendously favorable. Overall, density gradients could explain the odd onionskin data, but the conditions for these gradients are so unlikely a more reasonable explanation may exist in assembly issues.

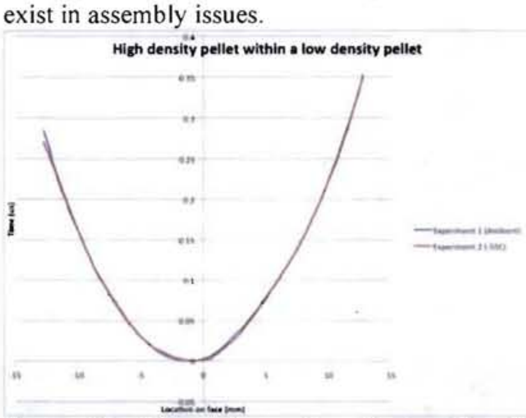

Figure 16: Comparison of ambient and cold shots of the high-density pellet inside a low density pellet. The breakout and behavior do not appear to be affected by temperature.

Conclusions

The goal of much of this work was to identify the material properties, which have an effect on DAAF's behavior. Along the way we have discovered a number of unexpected traits and greatly widened our knowledge of this fascinating material. For the most part, breakout behavior is not affected by temperature, and in order to change the symmetry of the breakout, large perturbations in density must be introduced. The critical diameter of DAAF is even smaller than previously thought: 1-1.5 mm. Floret testing will continue to seek answers to material property effects.

Acknowledgements

This work would have been impossible without the tremendous help and support of Robert Mier and Larry Vaughan at Chamber 8. Bart Olinger was instrumental with getting the Onionskin parts made and assembled. Ernie Hartline and Stephanie Hagelberg lent their pressing expertise in the precision-pressed pieces for both the floret testing and the polyrho testing. Eric Sanders and Joe Lloyd made the LANL SSGT work possible. Thank you all.

References

1. Chavez, D., Hill L., Hiskey M., and Kinkead S., "Preparation and Properties of Azo- and Azoxy- Furazans, *Journal of Energetic Materials*, Vol. 18, 219 (2000).

2. Francois, E., "Particle size effects on the shock sensitivity of Diaminoazoxyfurazan (DAAF) using an instrumented gap test approach," LANL report LA-UR 09-00967, 2009.

3. Dixon, W.J., Mood, A.M.,"A Method for Obtaining and Analyzing Sensitivity Data", *Journal of the American Statistical Association*, Vol 43, no. 241, 109-106 (1948).

4. Kennedy, J.E., Rumchik, G.E., Lee, K.Y., Asay, B. W., Thomas, K. A., Plakskin, I., "Floret Test Observations of Detonation Spreading in Insensitive Explosives", *Proceedings of the 13th*

Detonation Symposium, pp. 383, Norfolk VA, 2006.

5. Gibbs, T.R., Popolato, A., *LASL Explosive Property Data*, University of California Press, pp 114, 1980.

6. Tran, T. D., Pagoria, P. F., Hoffman, D. M., Cunningham, B., Simpson, R. L., Lee, R. S., Cutting, J. L., "Small- Scale Safety and Performance Characterization of New Plastic Bonded Explosives Containing LLM-105", *Proceedings of the 12th Detonation Symposium*, pp. 440, San Diego, CA, 2002.

7. Harry, H.H., Uher, K. J., Hagelberg, S. I., Hartline, E. L., "The Poly-Rho Test as a Screening Tool for Explosive Performance," Propellents, *Explosives and Pyrotechnics*, Vol 30, no. 6, pp 430-434, (2005).

8. Hill, L. G., Seitz, W. L., Forest, C. A., Harry, H. H., "High Explosive Corner Turning Performance and the LANL Mushroom Test", *Shock Compression of Condensed Matter—1997*, pp 751-754, Amherst, MA, (1997)