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Effects of Microstructural Properties on Chemical Energy Release and Initiation Processes of Energetic Materials

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14. ABSTRACT
DoD energetic systems vary in complexity and microstructural heterogeneity from the detonator to the main fill. Research has shown that these heterogeneities influence the initiation behavior of our systems, thereby dictating performance and reliability; however, only general trends have been established. A case in point is the established trend, whereby reduction of RDX particle size decreases the shock sensitivity of formulations being developed to meet Insensitive Munitions (IM) requirements. While these trends are used for formulation guidance, a lack of fundamental understanding of the initiation behavior has hindered our ability to satisfy IM requirements. More broadly, the DoD suffers from a poor foundational understanding of the underlying mechanisms that lead to energy localization and the formation of "hot spots" (see below). This issue manifests as a degraded ability to effectively quantify the safety and reliability of ordnance devices. Furthermore, it handicaps our ability to develop precise specifications for explosive formulations, despite our best efforts utilizing parametric experimental studies as done during the development of a new penetrator formulation.

We seek to establish a basic understanding of <u>which</u> microstructural features correlate with the sensitivity and performance of energetic materials, and if/how these structures influence the chemical energy release reaction kinetics. This foundational knowledge will enable the quantification of performance and reliability, identify microstructural features influencing initiation behavior, establish microstructural standards by which material sets are tuned for specific purposes, generate novel data to improve the predictive capabilities of reactive flow models, and guide the future improvement of energetic materials formulations.

15. SUBJECT TERMS
HMX, Microstructure, Shock Initiation, Electric Gun Flyer, PDV, RALF, Doppler Velocimetry

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Title: Effects of microstructural properties on chemical energy release and initiation processes of energetic materials

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Research Objectives: Generate novel statistical descriptions of various microstructural features of energetic materials (EM) used to identify correlating trends to chemical species evolution and critical stages during initiation. The defined trends will guide the generation of controlled microstructures to validate the correlations. This will be done using materials with specific large-to-small particle size ratios. The Lee-Tarver Ignition and Growth Reactive Burn (IGRB) model partitions the initiation process into ignition, growth and completion phases. Those phases are not discrete steps but methods are employed to segregate them in a manner that will increase our understanding of the effects of microstructure on the initiation process as a function of growth. Additionally, monitoring the chemical identities and molecular velocities of gaseous products from small EM samples reacting in vacuum will challenge the state-of-the-art mesoscale models and the simplified single parameter (λ) – extent of reaction treatment of chemistry in continuum level models. Data from the described experiments will be used to validate mesoscale models as well as enable the parameterization of continuum level reactive flow models. Ultimately, the knowledge gained here will enable a more precise approach to formulation development.

Technical Summary:

Overall

We are determining which microstructural features correlate with the sensitivity and performance of energetic materials, and how these structures influence the chemical reaction kinetics. This foundational knowledge will: Enable the quantification of performance and reliability, identify microstructural features influencing initiation behavior, establish microstructural standards by which material sets can be tuned for specific purposes, enable improved predictive capabilities of reactive flow and mesoscale models, and guide the future development of energetic material formulations.

To date, we have produced new experimental data that may reveal correlations between microstructure and initiation thresholds in reacting explosives. These results coupled with modeling may identify the dominant mechanisms occurring during shock to detonation processes. The experimental data will validate/constrain existing models, and encourage the development of new models. Our microstructural data have already motivated modifications to existing mesoscale models that predict initiation phenomena and chemistry evolution. We have selected three modeling frameworks in an attempt to identify the relevant physics linked to energy localization in the EM from the shock. We believe we can ascertain the microstructural features which localize energy by integrating our experimental data with selected models.

Modeling and Simulation Collaborations

We have continued collaborations with two modeling and simulation groups: University of Iowa (H.S. Udaykumar – SCIMITAR3D) and Georgia Tech (Min Zhou – CODEX). We have also integrated ALE3D as an in-house capability, with efforts led by Dr. Hardin. These models utilize different frameworks: SCIMITAR3D is Eulerian, CODEX is Lagrangian, and ALE3D is capable of switching between Eulerian and Lagrangian. Each framework has strengths and limitations and the goal is to couple experimental and modeling results to identify the aspects of microstructure that significantly contribute to energy localization, thus highlighting the physical mechanisms at play.

Our deepened collaborations have revealed areas of needed focus, for example with SCIMITAR3D limitations currently exist that force the use of very short duration shock pulses. These pulses are 1–2 ns. This limitation makes it difficult to map to the existing initiation threshold data, where pulse durations applied are ~8-100 ns. That said, efforts are being pursued to extend the pulse duration to match the experimental conditions.

We have constructed experiments to generate the world's most complete data set for validating state-of-the-art models. The three models chosen can be validated with our *unique* set of experiments. We have ascertained what experimental data these modelers need to validate their work. The validated models will then be put to the test by *predicting* the experimental results of new systems.

Experiment and Model Integration

Characterized powder and microstructural data was provided to the modelers from Class 3 and 5 HMX. Actual microstructures were used in SCIMITAR3D and the powder particle size distribution (PSD) were used to generate artificial PSDs for CODEX. CODEX then modeled fully dense microstructures generated from the artificial PSDs. SIMITAR3D simulations have implemented and compared the Henson-Smilowitz, Tarver, and Schweigert chemical description for HMX while tracking species evolution. CODEX is running non-reactive simulations.

Our experimental results indicate Class 5 having a lower threshold velocity than Class 3 for the thin flyers (<40 μm, see *Initiation Studies* section). Prof. Udaykumar's initial results indicated the Class 3 as being more reactive than Class 5. It was determined that this result was influenced by the lack of computational cell resolution for the Class 5 material. This prompted determining proper computational cell resolution, which took the remainder of the year. This resulted in increased reactivity in the Class 5 material; however it is not on par with the experimental results. It is suspected the short pulse duration is playing a role, but this will be the focus of future work.

In contrast, Min Zhou's work shows trends that suggest the Class 5 would be more reactive than the Class 3 material. CODEX was able to run multiple simulations using simulated microstructures. This has enabled them to generate data in the James-type framework. As a point of comparison, Figure 1 shows the experimental data overlaid with the modeling results for particle sizes near the experimental particle size. The three particle sizes chosen were strategic to establish trends with limited computational resources. What is shown here is the trend of large to small particle size correlating to a decrease in energy fluence requirements, i.e. lower velocity requirements for thin flyers.[1]

The preliminary nature of the experimental and modeling integration work causes us to be cautious with the interpretation of the results. However, this implementation did enlighten us in three areas: 1) We have different optimizations for fields-of-view/resolution for all three models. 2) We needed to experimentally determine how the PSD changes with pressing for CODEX. 3) One can't help but wonder how the results might change running a three-dimensional simulation versus a two-dimensional simulation. This is especially the case for the large aspect ratio features in the Class 3 material as they are sites in which ignition occurred first as predicted by SCIMITAR3D.

As for the optimized fields-of-view, we have implemented new equipment with better capabilities. We can now generate five times the field of view with similar resolution. This presents new challenges in that very large data sets need to be processed and analyzed. We are working on image processing algorithms to deal with these very large data sets.

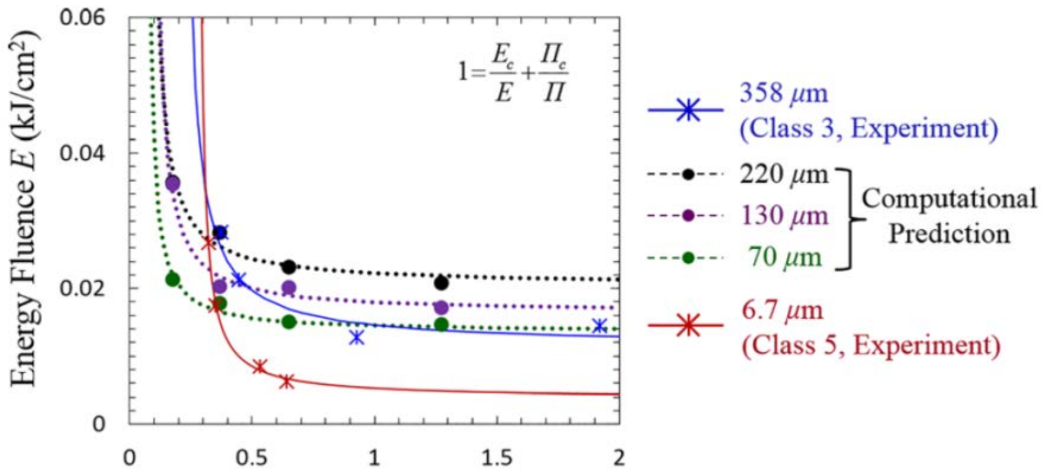

Figure 1: Computationally predicted 50% ignition thresholds from all grain sizes analyzed (d_{avg} = 70, 130, and 220 μm) and experimentally measured thresholds for Class 3 and Class 5 HMX

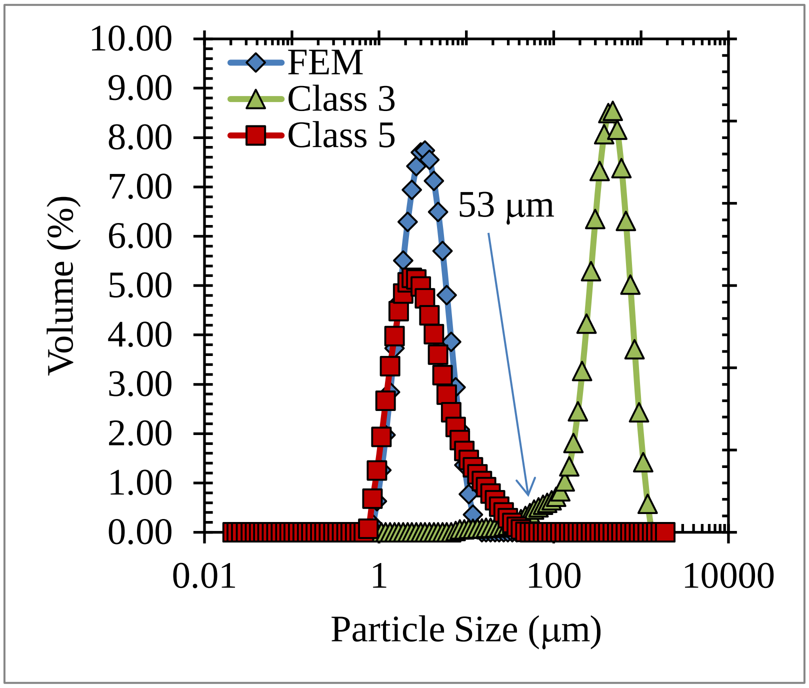

Figure 2: Volume percentage versus particle size for the various classes of HMX. Particle size indicated by the arrow (53 μm) defines the sieving size to generate the 4 custom materials defined in the text below.

In regards to determining how the PSD changes with pressing, this is a difficult task and the details will be expanded upon in the next section. Briefly, we are using a different description for the powder PSD, i.e. measured volume percentage as a function of size, Figure 2.

Describing the PSD in this way enabled definition of custom PSDs. We are taking a single parent lot of Class 3 material and generating 4 different materials:

Sample Type 1: FEM material with a mean PSD ~ 10 μm

Sample Type 2: Class 3 material without a small particle tail (~53 μm and below missing)

Sample Type 3: "Class 5 like" material is the FEM from 1) with the added small particle tail from 2)

Sample Type 4: the original Class 3 material

There are three main benefits from this approach. First, we reduce lot-to-lot variability for the different PSD. Secondly, we can assess the impacts of a narrowed particle size distribution "Class 3" to the full particle size distribution Class 3. Thirdly, we can access the impacts of expanding a PSD by introducing larger particles into a smaller PSD in the "Class 5 like" material. This custom material should generate an understanding of the effects of the various microstructures on initiation. This could enable tailored microstructures through specified particle size distributions, which is of tremendous practical importance. To illustrate, blending of two or more PSDs is often done to enhance manufacturing aspects of formulations. Gaining the knowledge sought in this work will allow us to more precisely tune performance and manufacturability of future formulations. We have funded Aerojet for our customized PSDs for HMX. The expectation is that we will have our material by this fiscal year.

To address the 2-D/3-D simulation effects, we have acquired new data sets that will enable us to elicit the magnitude of these affects. We have generated micro-CT scans of the Class 1 and

Class 3 HMX and have resolved the large extended voids suspected of causing reaction in SCIMITAR3D. This data set will be utilized to understand the effects of hotspot formation in a 2-D versus 3-D simulation. The three-dimensional simulations should also be compared to our chemical diagnostic results. For instance, the results from SCIMITAR3D indicate only Class 3 generates HCN product gas.

Microstructural Characterization

Ultimately there is a link between powder morphology and our pressed samples. To identify this link, we are using a better description of PSD for our starting powders in combination with quantitative microstructural characterization at several densities for our original Class 3 and Class 5 materials. We have started to characterize the microstructure with respect to the flyer impact surface, which has revealed conical type cracking in a critical place that may impact the initiation behavior. One such crack is shown for Class 5 material where the crack near the centerline of the pellet is roughly 270 µm deep, Fig 3. This was surprising since the pellet appeared to be mechanically intact. Additionally, we have assessed the density gradient as a function of distance from the flyer impact interface. Each red box in Figure 3 indicates a scanning electron microscope image acquired (100 µm wide). The calculated density is near the nominal 94% of theoretical maximum density (TMD) in most cases except the dramatic drop in density in the area of the conical crack, Figure 4. The distance "0" is defined as the flyer impact interface. The implications here are that the reactive front needs to jump this gap that is on the order of 10 µm and it is unknown how this impacts the early stages of initiation. This cracking is noticed in several of our pellets using the fine grain HMX as revealed in our micro-CT data.

Ultimately, this has caused us to change our target density, pressing protocol and perform a pressing study to obtain high-quality pellets. This pressing regimen will be done on pellets that are

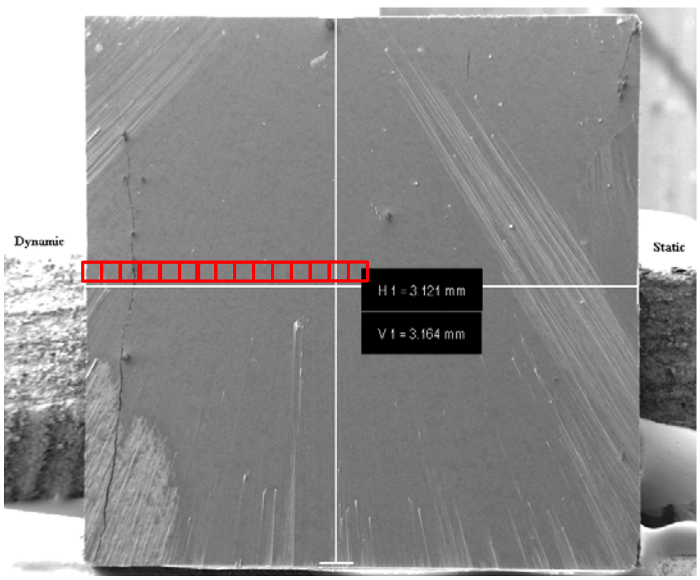

Figure 3: Class 5 HMX pellet at 94% TMD cut in half in which the conical crack is apparent near the dynamic or flyer impact surface. Red boxes indicate scanning electron microscope images acquired in 100 µm sections to access the density variation moving into the pellet.

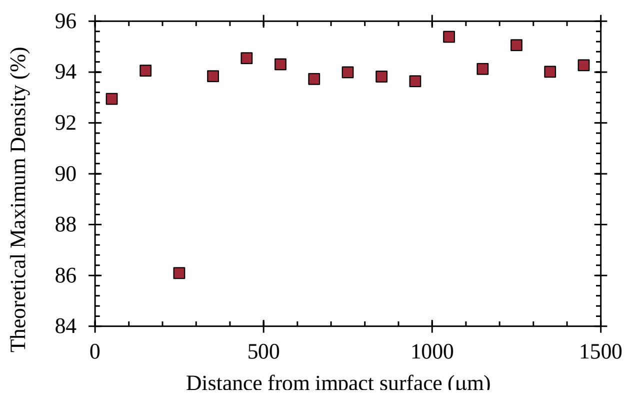

Figure 4: Density calculation for each red box in figure 3. Distance "0" corresponds to the dynamic side, i.e. flyer impact side. Density is close to the nominal 94% TMD throughout the pellet except where the conical crack is encountered and the density drops.

smaller than the initiation threshold pellets, however it is expect this will scale to initiation threshold size samples. We will confirm this with our characterization methods. The pressing study will enable us to identify correlations between powder morphology and the resulting microstructure, a critical aspect enabling the generation of custom microstructures.

Initiation Studies

In year one, we conducted initial initiation threshold studies of various particles sizes of HMX. Those studies allowed us to establish general trends that would guide future definition of particle morphologies to investigate in the future. The materials evaluated included FEM, two Class 3 materials, a Class 5 and a PBXN-5 material. That work included quantification of initiation threshold behavior of those materials as a function of density, and initiation spot size using a James-type framework. The focus this year was to further investigate the initiation characteristics of another pure HMX material, Class 1, a second type of PBXN-5 as well as to acquire additional firing systems needed for future experiments.

Figure 5 reports the results of the Class 1, 3, and 5 initiation threshold experiments. Only two Class 1 data points were experimentally determined and the third was derived by averaging the results of the Class 5 and Class 3 studies conducted at the same flyer thickness. The third point was added to enable the estimation of a James-like trend-line. The two Class 1 points measured where in the high power flux regime where past studies have shown that variations in microstructure lead to the larges changes in threshold behavior. The calculated energy fluence and power flux are noted on the plot for the two measured conditions for the Class 1. The Class 1 sensitivity in the strong shock and thin pulse regime was found to lie between the Class 3 and Class 5 materials. The mean particle size for the Class 1 is 201 μm, where it is 358 and 6.7 μm for the Class 3 and 5 respectively. The results shown in Figure 5 further support that threshold behavior is sensitive to microstructure in the thin pulse

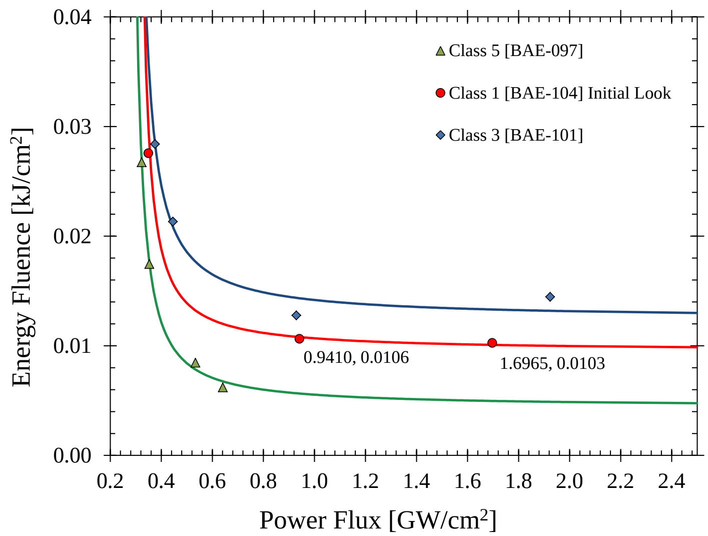

Figure 5: Initiation threshold behavior for various HMX classes

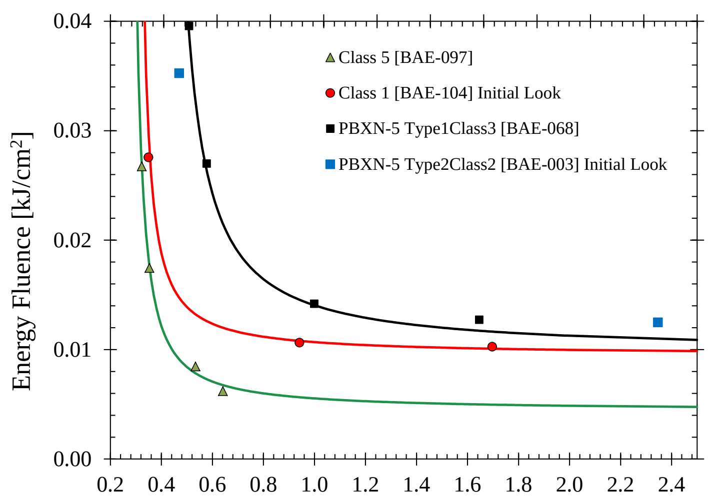

Figure 6: Initiation threshold behavior for HMX based materials

regime. Using the unreacted Hugoniot's for the flyer plates used in the initiation studies and the HMX, the calculated shock pulses in the HMX for the thinnest cases ranged from 30 - 40 μm. Those shock events correlate to critical ignition events occurring during the first 8-11 nanoseconds if ignition occurs prior to unloading when the axial relief wave arrives at the impact surface. We expect that the critical hotspots will be closer to the impact surface and will be somewhat less than the 30 - 40 μm, noted above.

To better understand the effects of inert components within a formulation, we further investigated PBXN-5 initiation behavior. PBXN-5 is a mixture of 95% HMX and 5% Viton by mass. Type I PBXN-5 is composed of Class 5 HMX only. Type II PBXN-5 is composed of 75% Class 1 and 25% Class 5 HMX. Figure 6 reports James-like threshold data for Class 1 and 5 HMX and Type I & II PBXN-5. Only two points have been collected for the Type II PBXN-5 but qualitative comparison between the Type I and II data suggests the variation associated with the composition change is small relative to the variation seen between the two pure HMX materials that are used to formulate the PBXN-5 materials. Further investigation is forthcoming but this result may suggest that the initiation behavior is controlled by the Class 5 HMX in both PBXN-5 types in the loading regimes evaluated thus far.

Chemical Diagnostics

Integrating a fireset into the Benchtop Energetics apparatus has required an enormous amount of effort. This effort was worth it to establish the missing link between the evolving chemistry from differing microstructures during critical stages of initiation.

To summarize, our EM fixtures have been designed and tested. These are made to be plug-and-play with our existing XYZ translator in the Benchtop Energetics main vacuum chamber. These fixtures allow for fast vacuum expansion of the chemical products, Figure 7. Part #1 in Figure 7 is the base plate that mounts in the XYZ translator and the rectangular recess locates the flyer chip (not shown). All the other parts (2-6) mount to this plate. The expendable jumper cable (2) is used to make the electrical connection between the flyer chip and a reusable high vacuum feed-through cable – Figure 8 – by means of clamping plate (4, Figure 7). The design for high-voltage, high current feed-through provides a seal to achieve a vacuum of ~1 X 10^{-8} Torr. Part (3) is a shim to adjust the flyer travel distance. The flyer impacts an explosive pellet pressed into the washer disk (5). Part (6) attaches to base plate (1), thus fixing in place the flyer chip, expendable jumper cable, shim, explosive pellet/washer.

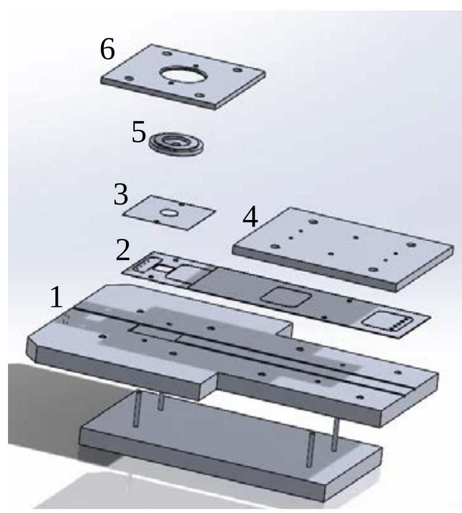

Figure 7: Expanded view of electric flyer plate assembly

We fired the first 200 μm Kapton flyer at 9.8 kV. This was a milestone in determining proper functioning of the fireset and accessing the degree of RF interference on our Time of Flight Mass Spectrometery (TOFMS) detection capabilities. The result was successful! We successfully launched a flyer that impacted a block of PMMA 4 inches away, near center line, and the TOFMS system only experience interference in the first couple of microseconds. Even during that time frame the detector exhibits no indication of interference, Figure 9. This successful result has accelerated the incorporation of in-vacuum velocity measurements.

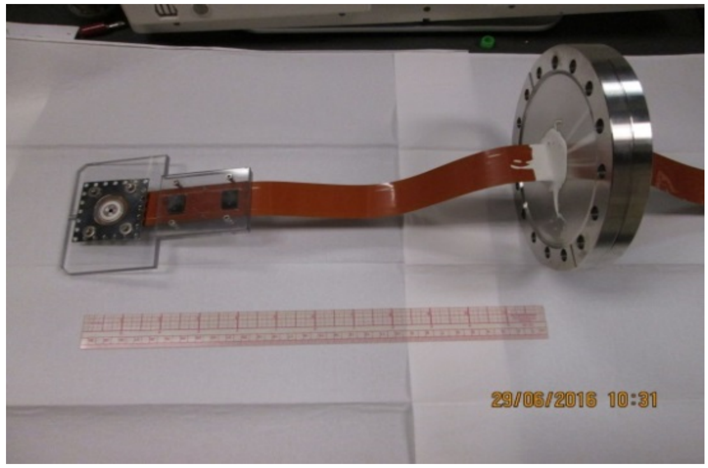

Figure 8: Custom high-voltage, high current feedthrough for throwing electrically driven flyers in the Benchtop Energetics apparatus

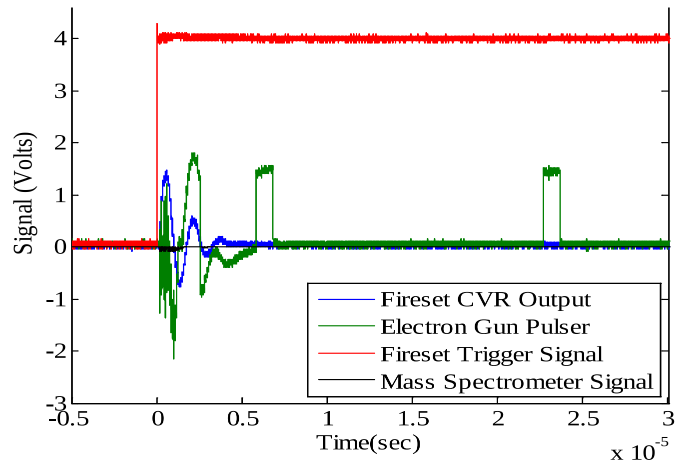

Figure 9: Raw oscilloscope traces of: Fireset response (blue), electron gun pulser (green), fireset trigger (red), and mass spectrometer signal (black). We observe RF coupling to the electron gun pulser cable, however this ends before the first electron pulse (mass scan). No indication on the Mass Spectrometer signal of RF coupling.

Thus far, we completed a series of single-point Rubidium Atomic Line Filtered (RALF) measurements of the velocities of laser driven flyers begun in FY14 – which were delayed during FY15 due to use-incurred damage to the 780 nm narrow-line illumination laser system. The first step was to repair the 780 nm laser, which was fortunately accomplished without requiring an onsite visit by the manufacturer.

Previous RALF experiments were optimized to collect the Doppler shifted light reflected from a mirror-polished surface.[2, 3] This approach suffers from extreme signal loss when the surface becomes a diffuse reflector upon shock breakout. Efforts to mitigate this problem by working with an initially diffuse reflecting surface returning ≈ 25 % of the original signal did not appreciably extend the useful measurement time beyond the present ≈ 10 ns observation window. Use of more diffuse (*i.e.* rougher) reflecting surfaces with higher illumination laser powers will be attempted in FY17.

In FY16 we improved the time response of all the PDV and RALF channels in the apparatus by upgrading the optoelectronic detection system to 12 GHz analog bandwidth, and by adding a second 780 nm laser to provide an upshifted-PDV (UPDV) capability. RALF seems perfectly compatible with surface acceleration profiles that reach velocities of ~ 1 km/s in a few ns.[2] The ultimate RALF time response is probably limited by the ~ 100 ps time between dephasing atomic-molecular collisions in the Rb/N_2 cells.[3] We will test this using true surface shock breakouts produced with the "Buelow" flyer-impacting-foil test surface sample geometry.[4]

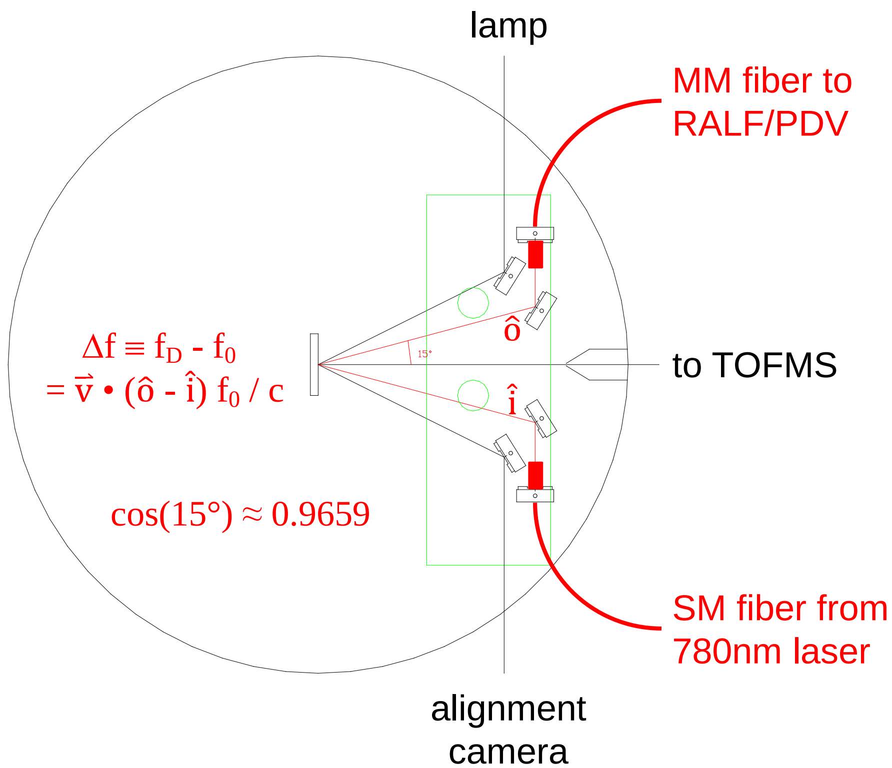

Figure 10: Schematic of proposed off-axis RALF/PDV implementation in Benchtop Energetics main chamber

We have developed an alternate approach to the "diffuse reflector" problem that abandons the canonical "single-mode-fiber + 3-port-circulator" PDV experimental architecture for one based on independent "send & receive" multimode (MM) fiber optical paths. An additional benefit is that the free-space beams from the incident (send) and observation (receive) optical fiber couplers will be arranged in an off-normal ($\theta_{inc} = \theta_{obs} = 15°$) specular reflection geometry, providing clear line-of-sight access along the EM sample surface normal for the TOFMS measurements. This approach yields the component of the velocity along the surface normal, scaled by a factor of 0.9659, as shown in Figure 10. This approach will also enable the future integration of the RALF/UPDV diagnostic into the Benchtop Energetics vacuum apparatus. We have procured the necessary multimode fiber components to perform this integration in FY17.

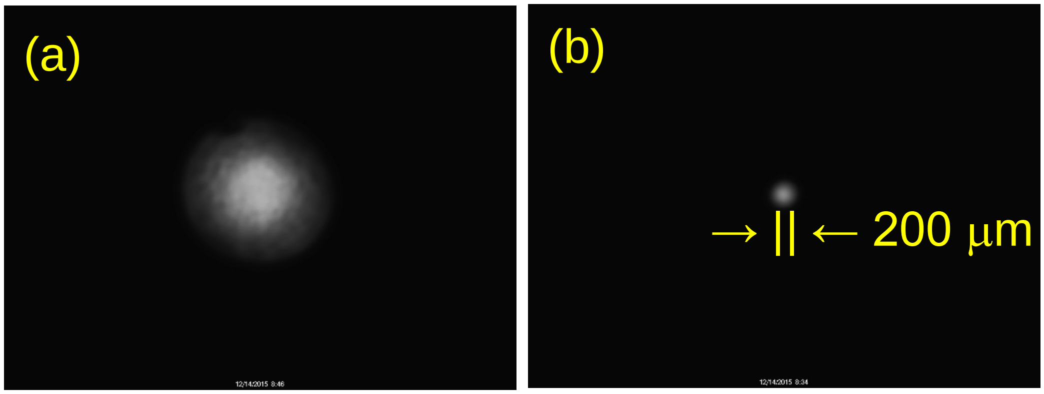

Figure 11: Focused spots for 780nm laser light launched from: (a) 50 μm core MM fiber, (b) SM fiber [MF16113]

Preliminary efforts at breadboarding an off-normal arrangement of send and receive optical fibers has made it clear that there are advantages to using a single-mode (SM) fiber to deliver the laser light to the test surface. Figure 11 shows the relative sizes of the best-focused spots obtained using MM and SM delivery fibers and a focusing fiber optic output coupler. However, the

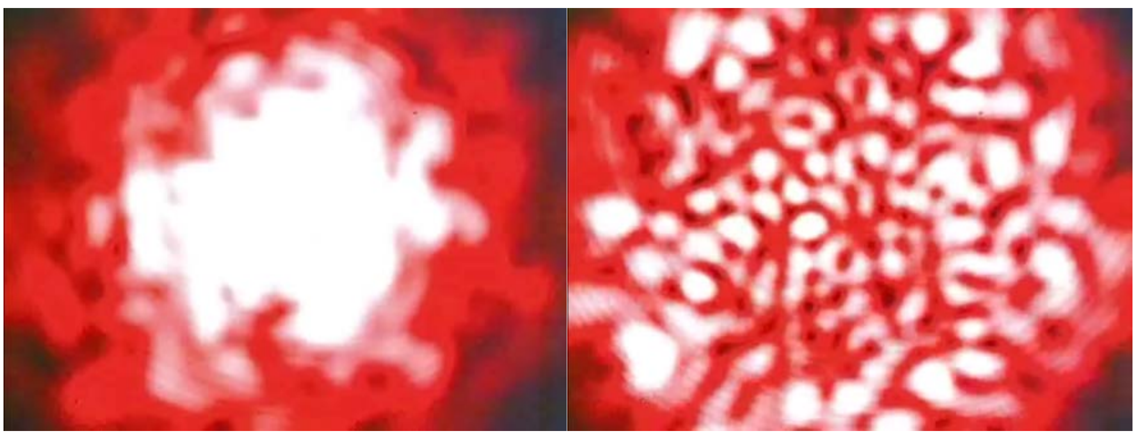

Figure 12: Output intensity distribution from 50 μm core MM fiber showing change in mode structure with input misalignment

greatly relaxed alignment requirements for coupling the reflected Doppler shifted light into a MM fiber should be a significant advantage in extending the duration of the RALF observation time window. Oddly enough, this may cause problems with the PDV measurement channel, since light injected into MM fibers at the extremes of the acceptance angle cone tends to couple into higher order modes, as illustrated in Figure 12. Since PDV relies on interference between the Doppler shifted light and some source of reference light, the orthogonality of the different modes in the MM fiber may limit the contrast of the interference signal unless the reference light is also distributed throughout the higher order modes.

FY17 Goals:

Microstructural characterization

As of result of characterization work in the past fiscal year we have identified the need to do a pressing study where we characterize the effects of pressing methods on macroscopic pellet structure and how it affects the resulting microstructure. This will be done to eliminate the previously observed cracking near the flyer impact surface. With the advent of the custom HMX, We will attempt to correlate HMX powder morphology and particle size distribution to resultant pressed microstructures. We will characterize the material coming from Aerojet looking for impurity changes due to milling of material. Additionally we will carefully characterize the powder from Aerojet and identify a method of coating the powders for the PBX work.

Initiation Studies

The initiation research focus will shift to the evaluation of the custom materials to be received in FY17 along with further investigations of PBX based materials. In FY16, we discovered the presence of microstructural imperfections in free standing pellets of the finer grained materials. For this reason, we will start our studies at a reduced density of 90% TMD and then evaluate 80% TMD followed by 94% TMD. The microstructures of the various materials at the target densities will be verified using micro-CT or focused ion beam cross sectioning techniques prior to executing the James-like experiments. Initiation experiments will be conducted that are quasi-1D as well as 2-D to assist in the determination of critical energy localization features within the microstructures that lead to successful initiation processes.

Chemical Diagnostics

We will attempt to gain improved signal levels in the RALF measurements through the use of more diffuse (*i.e*. rougher) reflecting surfaces with higher illumination laser powers. As a parallel effort to solve the "diffuse reflector" problem (described above) we will pursue the method involving multimode fiber optical paths. We have procured the necessary multimode fiber components to perform this integration in FY17. Additionally, we will focus on the integration of the RALF/UPDV diagnostic into the Benchtop Energetics vacuum system. Once completed, we will begin characterizing the fireset voltage versus flyer velocity. Additionally, we will start to characterize the base line mass spectrometry signals generated from launching of the polymer flyers.

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