Test Development for Characterizing Electrically Controlled Energetic Materials

Kimberly Chung, George Fischer, Gregory Valenta, and Melissa Liberatore-Moretti
U.S. Army CCDC Armaments Center
Picatinny Arsenal, NJ 07806

ABSTRACT

Controlling of the output of energetic materials provides the potential to develop munitions that are more effective and efficient. In the case of gun propellants, this translates into improved reliability, extended range without increase in energetic fill, and better control of trajectory for gun launched rocket motor systems. The U.S. Army has been pursuing the development of energetic materials that are responsive to electrical stimulus. The goal is to be able to activate, extinguish, and throttle the material in the presence or absence of electricity. This type of ignition is unique, so tests to support feasibility studies and characterize performance do not exist. A series of tests ranging from the proof-of-concept scale to determine ignitability, to larger tests used to collect quantitative data on performance have been developed. Results from the development phase and output of tests will be presented.

INTRODUCTION

Electrically controlled energetic materials (ECEMs) are materials that produce gas and flame in the presence of electricity. Traditional energetics are initiated mainly through shock or with the application of intense heat (via bridgewire). Once ignited, these materials continue to burn and the output can only be controlled by the grain geometry or through engineering designs which regulate the pressure of the chamber the propellant is in. The idea behind electrical ignition is that the output of the material can be tailored through the electrical parameters the propellant is subjected to. Benefits associated with the ability to control the burn rate include temperature compensation, in-flight course correction, simplification of the ignition train, etc. While ignition and throttling are the goals in this program, the ability to quench the reactions is also desired. However, the latter is a difficult goal due to the nature of working with materials that are able to sustain reaction at elevated temperatures and pressures.

The initial phase of the program sought to identify formulations that can initiate electrically. Gun propellants have incredibly high resistances as the main component, nitrocellulose, is not conductive. As an example, a study with M9 (40% nitroglycerin) revealed that at least 35,000V are required for reaction[1]. Since gun propellant formulations would be difficult to work with, the focus was shifted to gun launched rocket motor applications. Various oxidizers, binders, and additives were explored for the prospect of electrical ignition. As these formulations ignite in a unique manner, new tests were developed to determine which samples were responsive to electricity. Such small scale tests are appropriate for feasibility screenings but larger tests are needed to evaluate the reaction in more realistic configurations.

Figure 1: Small scale ignition of ECEM (left) and larger fixture for data collection (right)

Once the proof-of-concept tests show that a material can ignite consistently at the laboratory scale, additional tests are needed to continue to explore the material's behavior and burn characteristics at a larger scale. In addition, larger tests allow other factors to be evaluated (e.g. electrode configuration) and allow for data collection so that the performance can be compared quantifiably. This paper will outline the development of tests for the maturation of ECEMs.

EXPERIMENTAL DEVELOPMENT

Three tests were developed to screen formulations and measure the performance of ECEMs. The initial tests focus on evaluating feasibility while the last test is intended to gauge performance.

Screening Test

There are many combinations of oxidizers, binders, plasticizers, and oxidizers that can be considered for ECEM. Working with the smallest quantities of materials is necessary so that a large number of formulations can be evaluated. Feasibility is simply based on whether the material can ignite with electricity. As such, visual observations of the event are sufficient for determining if ignition occurred.

Initially, the samples were simply pressed between glass slides with stainless steel wires running through the samples. This setup was inexpensive and easy to prepare. Standard video was adequate in capturing the reaction as the sample initiated at the electrode and then quickly propogated through the sample. As this test was used more frequently, the contact area between the electrodes and sample became a concern. It was possible that the ignition requirements were too stringent because the contact area was too small resulting in insufficient electrical exposure. One solution to this problem was to switch to metal slides. Copper, aluminum, and stainless steel slides were used for testing. Although the reaction could not be viewed directly, go/no-go reactions could still be determined as flame and gas jetted out from between the edges of the slides.

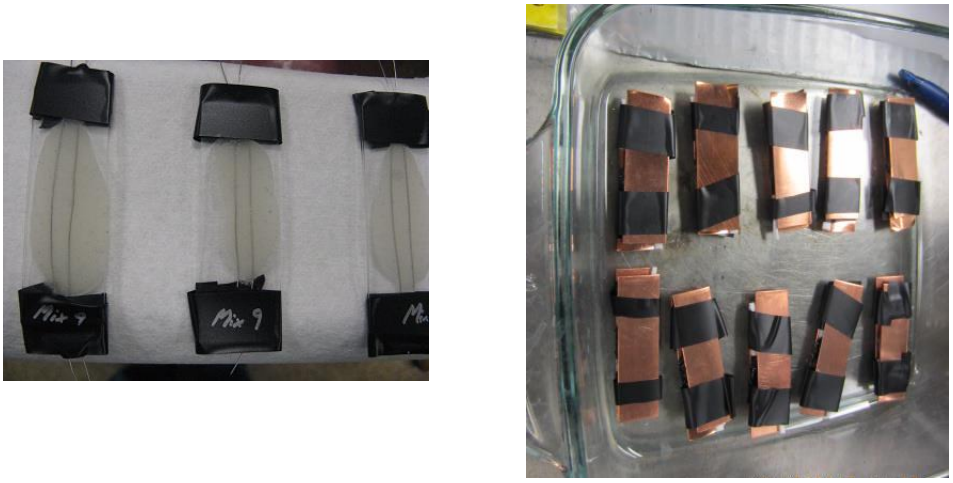

Figure 2: The proof-of-concept test involves pressing the material between slides. With the glass slides (left), stainless steel wires deliver power to the sample. When copper slides are used (right), each slide serves as an electrode.

Cylinder Test

While the screening test allows for high throughput in evaluating formulations, the configuration is far from realistic. Propellants are typically housed in a cylindrical geometry. The spacing between electrodes is on the order of a millimeter with the slides; this distance will be unrealistic for a fielded item. With the cylinder test, the idea is to increase the distance between the electrodes and allow for a larger quantity of material to be evaluated. In terms of contact area between sample and electrodes, this test falls between that of the glass slides and of the metal slides. Ignition will be more challenging than with the metal slides but the configuration is more representative of the actual system.

The goal of this test was to transition from flat to cylindrical geometry while retaining the ability to test at the laboratory scale. Cavities, that are 9.5mm in diameter, were drilled out from the center of 31mm polycarbonate rods. The height of the fixtures were varied at 3.2mm (disc), 25.4mm, and 31.8mm as shown in the figure below. The fixtures were able to accommodate 3 – 5g of material. Power delivery was achieved by applying copper tape to the orifices and connecting the tape via an external wire. The electrode contact area was varied by changing the amount of copper tape used. The other electrode was metal wire inserted through the center of the material. To load the fixtures, the mixture was prepared and then hand loaded into the cavity using a spatula. Once the material was loaded, the center wire was gently inserted through the sample. Visual inspection was used to minimize the possibility of voids in the fixture.

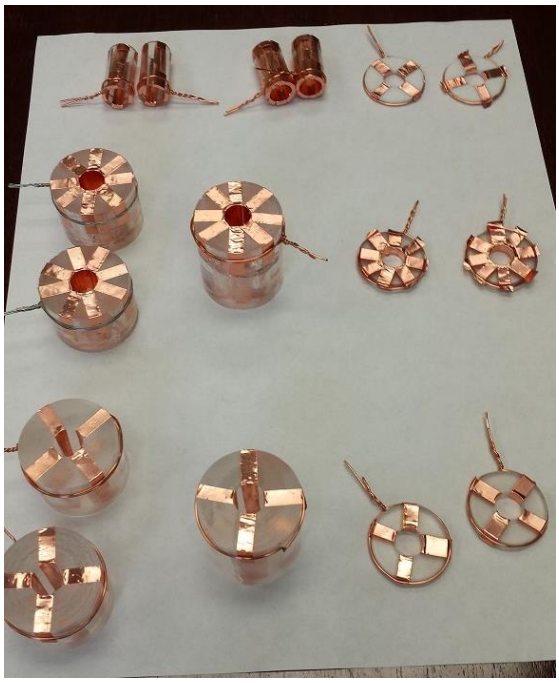

Figure 3: Cylindrical polycarbonate fixtures with copper tape. Various L/D ratios were explored.

Testing was conducted in a laboratory test chamber to ensure proper ventilation of the gases. The wires from the power supply were threaded through a side port of the chamber and then sealed to prevent reaction gases from escaping. The chamber door has glass window through which the reaction can be viewed. High speed video was used for several tests but the thick glass prevented capture of any meaningful data. Regular video and visual observations were sufficient for this setup. Power was applied for a maximum of one minute. If the sample did not react in that time, the power was turned off and a period of waiting ensued after which the sample could be removed.

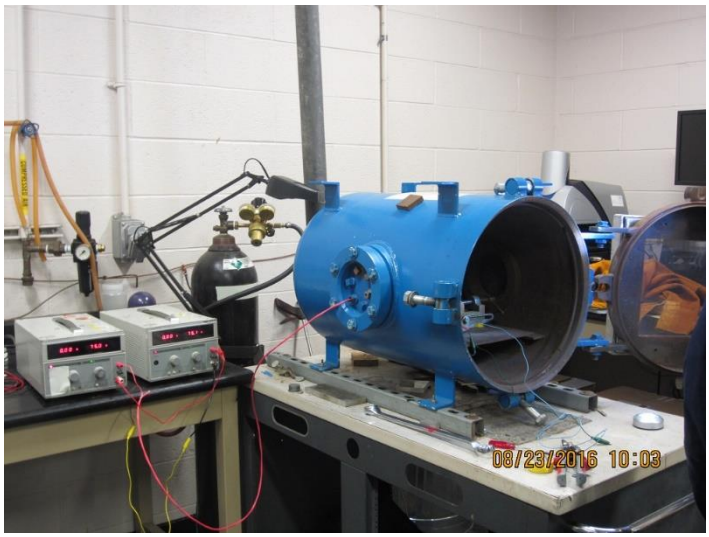

Figure 4: Polycarbonate cylinders were evaluated in a laboratory test chamber.

Subscale Thrust Fixture

Quantitative data is necessary to understand the ECEM reaction. Information desired from this test included pressure, force/thrust, and temperature. With the instrumented setup, the goals were to collect information on the ignition delay and the output as a function of the power profile the material is subjected to. If good thrust profiles are generated, these can be fed into models to predict performance in an actual system.

To keep costs down, a simple fixture was developed, in-house, using commercial parts. A metal test chamber would be machined to accommodate propellant and tapped with ports for the pressure sensor and electrode. A nozzle would be attached to the open end of the chamber while a force sensor would be affixed to the other end. A schematic of this design is shown in Figure 5 below:

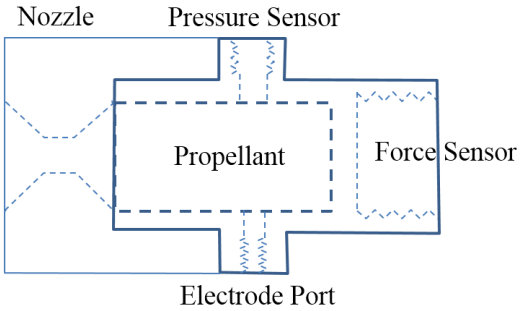

Figure 5: Schematic of the test chamber and sensor interfaces.

While this simple design was cost effective and easy to manufacture, there were concerns from the test group when a prototype was presented. Although the threaded parts facilitated assembly, they produced risks as pinch points if the energetic contaminated the threads. The support board also contained threads which could also be contaminated from reaction products even if complete reaction was achieved. To simulate a rocket motor, the test chamber was affixed to the support board horizontally but the torque on the end could result in erroneous readings as the system used a simple force fixture which is only capable of measurements in one dimension. This required the test chamber to be completely level with the gauge.

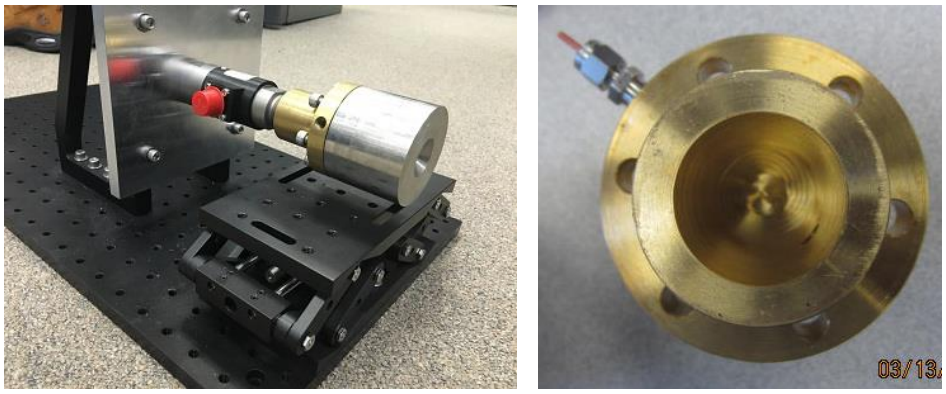

Figure 6: The in-house thrust fixture was easy to manufacture but had safety and data collection concerns.

A second thrust fixture design was developed and manufactured with the assistance of a contractor. Pinch points, threads, and horizontal loads were avoided. As the contractor had more manufacturing capability, the option to modify the electrode design was added as a requirement. Separation of the energetic material and instrumentation was achieved by manufacturing a test chamber that is separate from the support fixture as in the first design, allowing for several chambers to be prepared for testing at one time. The support fixture contained the electrical connections and ports for capturing temperature, thrust, and pressure.

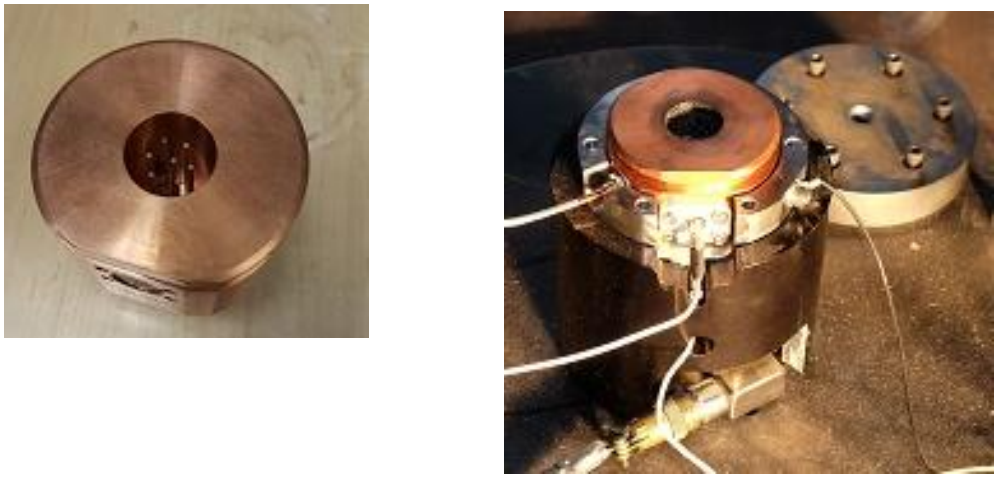

Figure 7: The propellant is loaded into the test chamber which contains several electrodes (left) and is seated in the support fixture. (right).

RESULTS AND DISCUSSION

Screening Test

The use of glass slides for the screening tests was convenient for observation. Regular video was able to capture the location and progression of igntion. Testing was conducted in a hood to minimize exposure to reaction gases. The power supplies and cameras were on a counter adjacent to the hood. In the figure below, the ignition point can be clearly seen, occurring on or adjacent to one of the stainless steel wires. The material appears to burn along the path of the wire as the gases are able to escape through a path that is opened from the burning propellant as shown in the screenshots below.

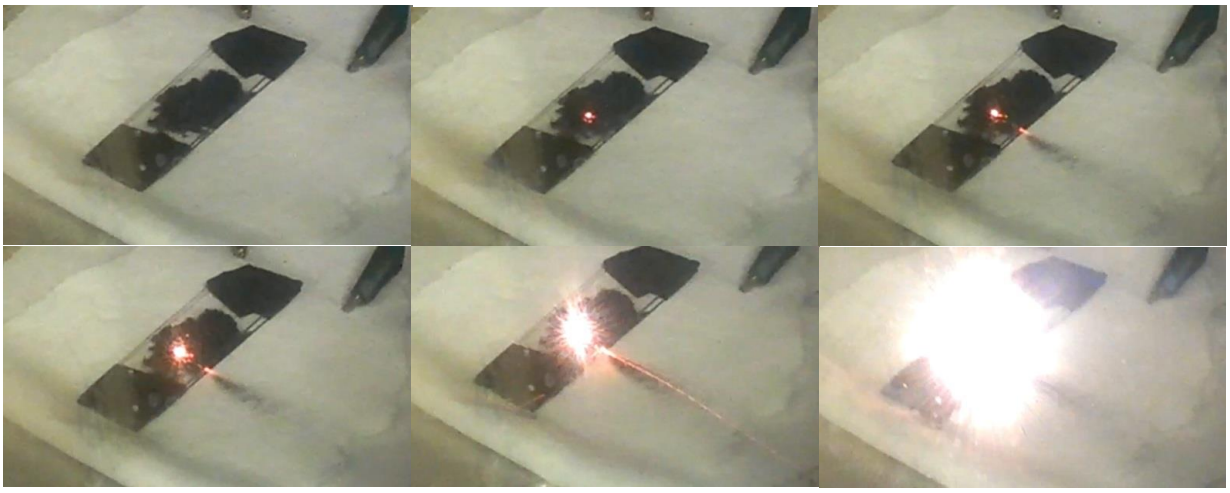

Figure 8: Screenshots of the reaction progression from a screening test

Most reactions occurred in under a minute. There were several samples where flickering was observed along the electrodes but additional exposure to power failed to result in ignition. Since the wire gauge was small and the reaction appeared to occur along the wires, the small contact area between the sample and electrodes became a concern. For some electrically initiated materials, the amount of contact is a factor for igntion[2]. Although larger wires could be used, transitioning from glass to metal slides would eliminate the need to use wires as the slides themselves would serve as electrodes.

Figure 9: Copper slides used for a screening test

The use of metal slides presented some challenges but many issues were quickly resolved. If the sample was cast onto the slides unevenly, it was possible for the slides to touch which would create a short circuit. It was also important to not stack one slide directly over each other since it would be nearly impossible to connect each one to the power supply. For testing, the metal also prevented observation of the reaction. Previously, the ignition location or the potential for ignition could be observed but any indication of reaction with metals was the presence of flame. This was overcome by using an IR camera to detect temperature changes. Although rapid, heat can be seen building up where the electrical tape is present. Shortly after, local temperatures rise to 95°C, the sample ignites.

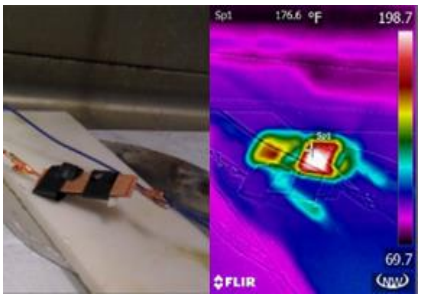

Figure 10: Image from IR camera indicating impending reaction

Cylinder Test
The cylinder test allowed a larger quantity of material to be evaluated. In this configuration, attempts to quench are possible. Material was hand loaded into the center cavity. The number of copper strips were varied.

Several formulations were evaluated in this configuration. Of those that ignited, quenching was also attempted. It should be noted that when quenching was achieved, the reaction receded for a few seconds before power was applied again. This is possibly an effect only achieved on the small scale. Although this test contains more material than the slides, it is still a small amount of material compared to the amounts used in an actual system. Energetics will self-sustain ignition at elevated temperatures and pressures. With these small quantities, quenching may be possible because the localized heating affects a very small area so secondary thermal reactions do not occur; pressure is also maintained at ambient. Propellants have an increased and sustained burn rate at elevated temperatures.

When testing, the cylinders were secured horizontally with respect to the open ends as shown in the figure below. The maximum power output the system is capable of putting out is 220W. Most ignition events only draw a maximum of 25W.

Figure 11: The cylinder is secured horizontally in the test chamber (left) and retrieved nearly intact (right)

To evaluate the effect of cylinder height, a single formulation was used for several tests. The majority of the samples ignited when subjected to power. Significant flame generation was observed from both open ends. The flat discs burned easily. The cylinders that were 25.4mm and 31.8mm in height had less consistent results. While some samples did not ignite, others experienced significant ignition delays. If a sample did not ignite after one minute of exposure, the test was stopped. For this particular formulation, once the material started to burn, the reaction could not be quenched. However, the current process of quenching involves visual observation of flame followed by manually shutting off the power supplies. The entire process takes a minimum of 3s which is enough time for most of the material to be consumed. A power supply that can be programmed to pulse or terminate the delivery of power would be suitable for evaluating the ability to quench a particular formulation.

Subscale Thrust Fixture
Scaling up to the subscale thrust fixture to collect quantifiable information about ECEMs was exciting. However, the complexity of the pre-test and testing operations increased. Although samples were easily hand loaded for the screening and cylinder tests, the viscosity of the formulations and the presence of several electrodes in the test chamber made hand loading incredibly challenging. The material was packed into the test chamber by hand as best as possible. To remove the voids that were likely in the bulk material, the test chamber was placed under a vacuum bell for at least ten minutes. In most samples, the material bloomed out of the fixture indicating that significant air pockets were present. The material was brought back to atmospheric and then pressed into the chamber again. Vacuum was pulled a second time. Additional blooming was observed but the material was pressed in a final time until the surface was flush with the test chamber.

Testing was carried out in a blast chamber. The instrumentation and video was set up the day before and checked again before the actual test. The response of the thrust, pressure, and temperature gauges were verified as well. The goal of the first test was to ignite the system and demonstrate that the data acquisition system functioned as intended.

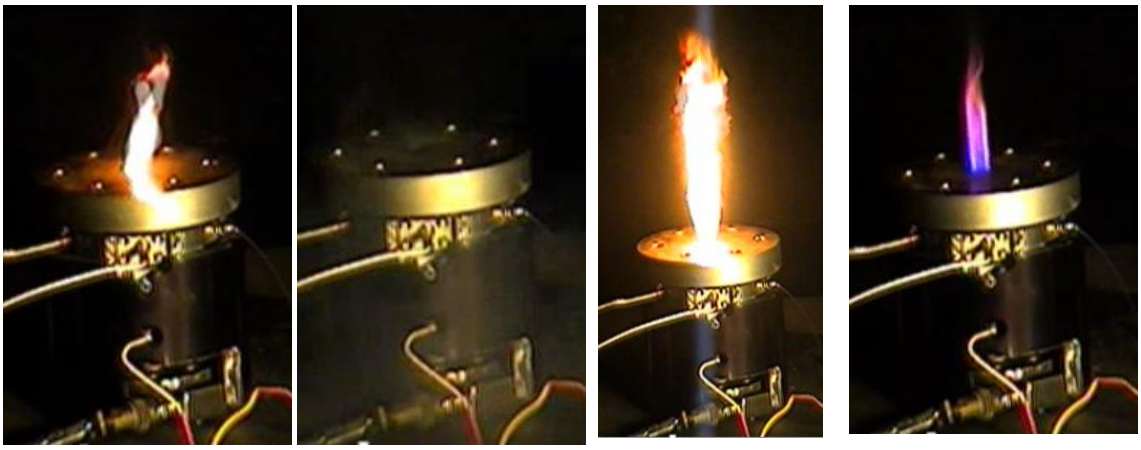

Figure 12: Video stills from regular video of a successful igntion

The video from the first successful test showed the sample sputtering in terms of flame generation. Even though the power delivered to the material was constant, the sample would ignite and extinguish itself several times before producing a consistent, bright flame. As there was no attempt to control the output, the presence of voids was the likely cause of the initial ignition inconsistencies. Post-test inspection of the fixture indicated that most of the material was consumed.

The data acquisition system managed to capture data for the thrust. Due to the location of the temperature and pressure gauges, there were no significant readings for those variables. The thermal mass of the test chamber and the short reaction time does not allow the thermal energy to propagate quickly and heat up the exterior of the test chamber, where the thermocouple is located. The pressure port was also located on the side of the test chamber and is blocked by propellant during the start of the test. As the material starts to burn, even when the pressure port is exposed, the path of least resistance for the gases is straight through the nozzle. For this burn reaction, two peaks in the thrust were observed. One correlates to the initial burn and the second appears when the reaction is self-sustaining.

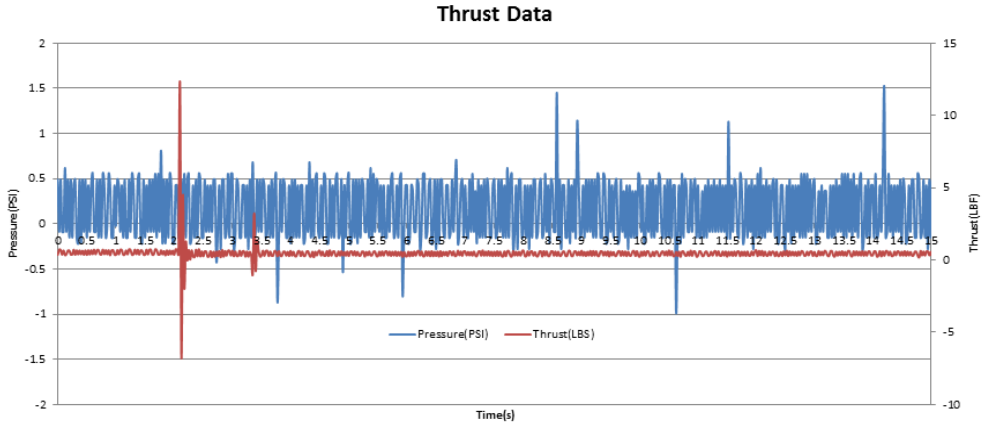

Figure 13: Thrust and pressure data

A second, higher energy formulation was eventually evaluated in this system with different results. Visual observation in real time yielded very little as the reaction unfolded in less than a second. High speed video was not used due to the long burn times typically exhibited by this material. Regular video would reveal that a significant amount of flame and gas was generated in a short period of time. The pressurization was so rapid that the nozzle plate was thrown off and broke into several pieces. The sleeve that the test chamber was seated in expanded and could not be removed from the support structure. Unburnt propellant was found scattered over the floor.

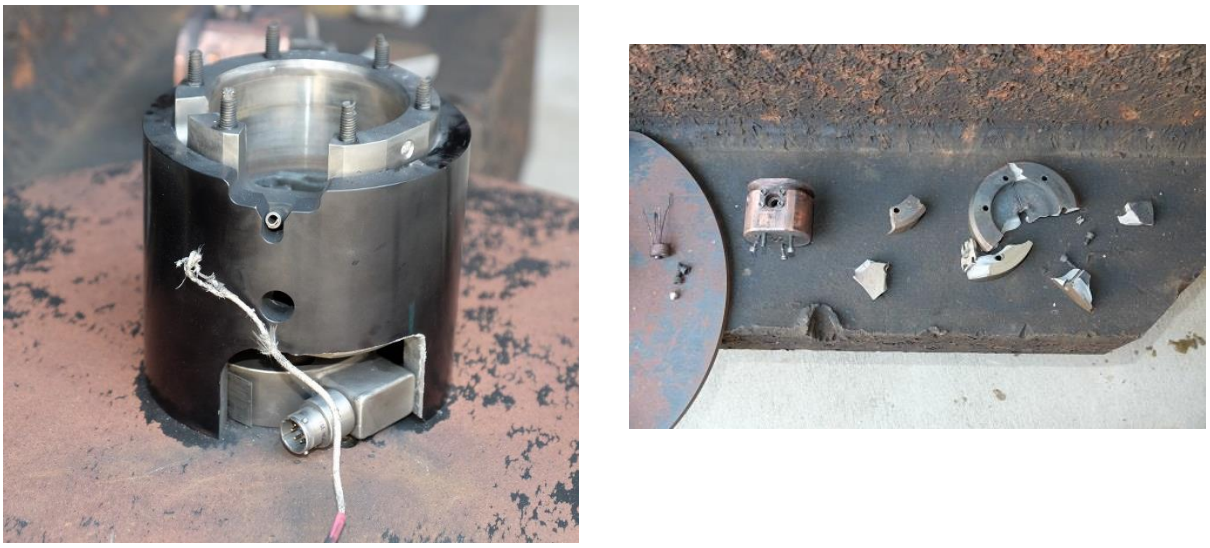

Figure 14: (left) The bolts securing the nozzle plate sheared off (right) and the nozzle plate was damaged when it was thrown into the walls of the blast chamber.

The pressure and thrust data also indicate that this reaction was more vigorous and energetic than previous iterations. Both gauges were maxed out and could not be reused.

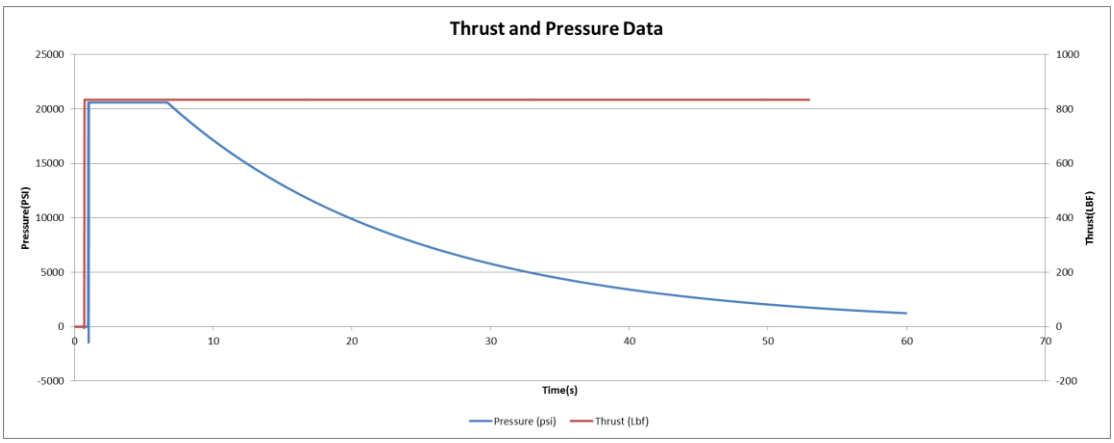

Figure 15: Thrust (red) and pressure (blue) profiles for a higher energy formulation

The violence of this test destroyed most of the hardware needed for testing. A redesign of the system to prevent such overpressurization and gauges with higher range will be considered for the next iteration.

CONCLUSION

Three tests were developed to evaluate the response of ECEMs at various scales. The first test is a screening tool and as such is simple in design, requiring little hardware, and can be carried out quickly. It is useful from a safety perspective (smaller quantities) and for when ingredients are scarce. The utility of the cylinder test is less clear. While samples that did ignite with the screening test did not always react with the cylinder test, the increase in material needed for the cylinder test is about five times as much as the screening test while it is an order of magnitude great between the cylinder test and the subscale thrust fixture. The fixtures also require a bit of work to produce since the copper tape needs to be added by hand. This test may benefit if the cavity can be made larger. However, there is an energetic limit when using the laboratory test chamber. Beyond that, the samples need to be evaluated in the blast chamber as is the case with the subscale thrust fixture. The subscale thrust test is the most important test out of the three but more preparation is required. Although the design has shown that data can be collected, the utility of the data is questionable until better casting methods are developed. The presence of voids produces an erratic burn so the true output of the material cannot be determined.

One of the goals of the program has been to develop a material that can be quenched with the removal of electricity. Quenching is achievable with the small quantities used in the screening tests but it has been more difficult to achieve with a larger quantity of material. The frequency with which the reactions can be stopped with the cylinder test is drastically decreased. Quenching the reaction with the subscale thrust fixture has not been attempted because there are challenges with igniting the material as is. Once the material loading issue has been resolved, different power profiles can be used to measure the sample output as a function of electrical input.

FUTURE WORK

Future efforts will continue to focus on material loading and redesigning the subscale test fixture. The quality of the data hinges on the quality of the same. Vacuum casting, pressure casting, or an injection molding process will be considered to improve sample preparation. Updates to the subscale thrust fixture are also necessary. Since the temperature sensor did not provide any data due to the thermal mass, it does not need to be included in the next iteration. The pressure sensor should also be relocated so that it is more in line with nozzle plate and can measure the gases as they are exiting the test fixture. Finally, a mechanism to prevent overpressurization of the system needs to be included to prevent damage to the overall fixture. With these changes, testing can be done to enhance understanding of ECEMs and transition the material to a larger scale of testing.