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**Visualization and Measurement of the Burning Surface of
Wire-Embedded Energetic Materials,
Part I: JA2 and Pentolite**

by John J. Ritter, Zachary Wingard, Tony Canami, and Andrew McBain

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14. ABSTRACT To increase the burning rates of minimum-smoke propellant formulations that are less sensitive than current standards, the Army is investigating gains that are achievable by embedding propellant with thermally conductive wires. The mechanisms that lead to increased burning rates in wired propellant are not fully understood; therefore, grain design optimization is a significant technical challenge. As part of an effort to gain insight into the mechanisms underlying the phenomenon, and thereby accelerate development of the technology, an experimental technique was developed that enables the burning surface of a wire-embedded strand to be visualized throughout the course of a burn rate measurement. Revealing details of the process that are hidden to standard burn rate measurement techniques, the U.S. Army Research Laboratory is developing a state-of-the-art computational fluid dynamics model for simulating the performance of rocket motors with wire-embedded propellant grains.				
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Contents

List of Figures	iv
List of Tables	iv
Acknowledgments	v
1. Introduction	1
2. Experimental Methods	4
2.1 Test Article Fabrication.....	4
2.2 Measurement Techniques.....	5
3. Results	6
3.1 Pentolite.....	6
3.2 JA2.....	9
4. Other Observations	13
5. Summary	13
6. References	14
Appendix A. Pentolite Melt Casting Hardware Drawing	17
Appendix B. Measured Burning Rate Data for Pentolite Experiments	19
List of Symbols, Abbreviations, and Acronyms	25
Distribution List	26

List of Figures

Figure 1. (left) Photograph of internal conical burning surface of translucent solid propellant composition embedded with 0.8-mm-diameter silver wire. (right) Schematic of wire embedded propellant combustion.	2
Figure 2. Pentolite cast into glass tube.....	4
Figure 3. Windowed test article: JA2 strip embedded with a silver wire.	5
Figure 4. Schematic of strand burner facility (left), windowed strand burner (middle), and strand burner control panel (right).	6
Figure 5. Pentolite burning in glass tube.	7
Figure 6. Pentolite burning at 3.50 MPa: rates with and without an embedded 0.010-in. silver wire.	8
Figure 7. JA2 burning samples at 6.90 MPa; baseline, no wire (top left), 0.002-in. silver (Ag) wire (top right), 0.005-in. Ag wire (bottom left), and 0.010-in. Ag wire (bottom right).....	9
Figure 8. Distance measurements taken at the outer edge and centerline.	10
Figure 9. JA2 burning rates at 3.45 MPa.	11
Figure 10. JA2 burning rates at pressure near 5.18 MPa.	12
Figure 11. JA2 burning rates at 6.90 MPa.	12

List of Tables

Table 1. Pentolite burning rate data.	8
Table 2. JA2 burn rate data.	11

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1. Introduction

The U.S. Army Missile Research, Development and Engineering Center (AMRDEC) is working to develop nitrate ester-based (minimum smoke) propellant formulations that are less sensitive to threats than current standards. However, reduced sensitivities are usually achieved at the expense of burning rate, which, given standard center perf and end-burning grain designs, limits the range of applications for which they are suitable (1). Of the many techniques that have been successfully employed to increase burning rates, increasing the thermal diffusivity of grains by embedding them with metal wires (or fibers) appears to be the approach that is the most compatible with the objective. Reported as early as 1955 (2) and fielded in the 1960s (e.g., Redeye and Stinger missile systems), this approach is nevertheless a challenge to implement reliably. Casting grains without breaking wires is difficult.

Given the risks related to manufacturing wire-embedded propellant grains, performance increases need to be significant to justify an attempt to field the technology. However, the limited understanding of the process that exists today makes it hard to predict the technology's full potential. It is known that metal wires produce localized conductive heat transfer from the combustion zone into the uncombusted propellant, generating conically burning surfaces and changes in surface topology and burning rate. The extent of heat transfer is a function of the wire's thermophysical properties and geometry (1-7). However, only empirical models of the process have been developed to date, and their extensibility is very limited. As a result, AMRDEC has had to rely on experimental (trial and error) testing of various wire-propellant combinations to establish burning rates, then use a model such as the one depicted in figure 1 as a basis for grain design. Results have been mixed, and the parameter space that can be probed via this approach is limited. (Parameters include the wires' thermophysical properties and diameter(s), their number, spacing and orientation within the grain, and the thermophysical and chemical kinetics properties of the propellant formulation.) As such, it is not clear that the full potential of the technique can be established or how it might best be approached.

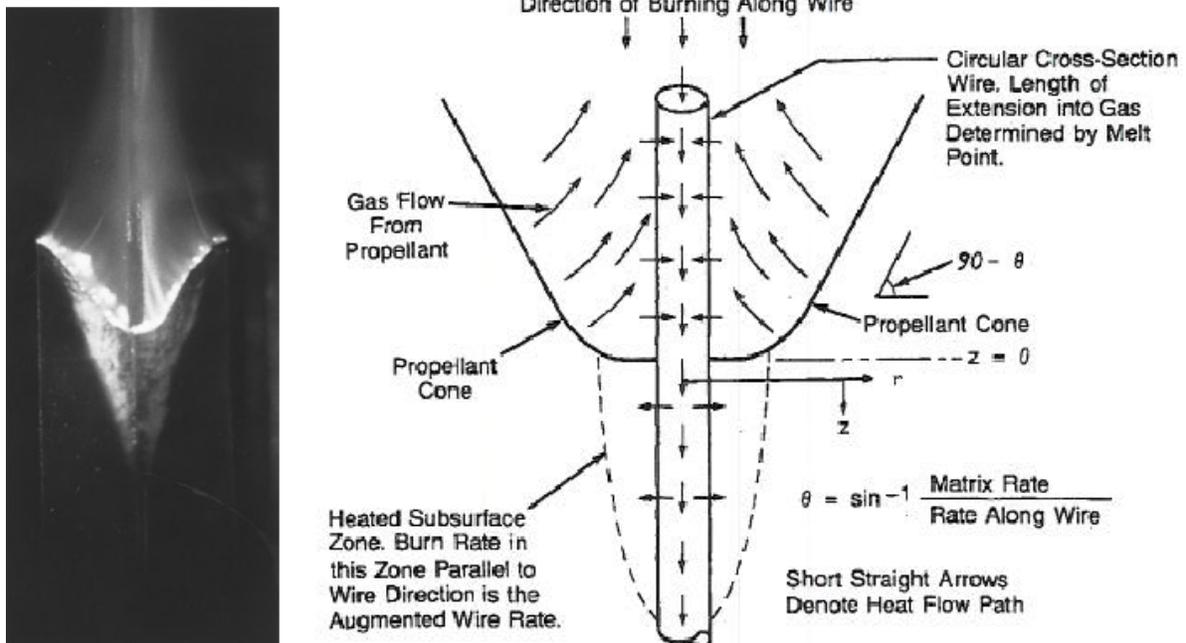


Figure 1. (left) Photograph of internal conical burning surface of translucent solid propellant composition embedded with 0.8-mm-diameter silver wire (4). (right) Schematic of wire embedded propellant combustion (5). (Source: Kubota, N.; Ichida, M. *Combustion Processes of Propellants With Embedded Metal Wires*; and King, M. K. *Analytical Modeling of Effects of Wires on Solid Motor Ballistics*. Reprinted by permission of the American Institute of Aeronautics and Astronautics, Inc.)

Seeking to address this issue, the U.S. Army Research Laboratory (ARL) is developing a state-of-the-art computational fluid dynamics (CFD) model of the process (8). As part of the effort, experimental data that can be employed for model validation are being sought. In particular, our goal is to directly observe burning surfaces produced by various propellant-wire configurations. Such data are not produced by the Crawford-style strand burner that AMRDEC has been employing for its effort (9). Measuring the time between the openings of two break wires that are a known distance apart, the Crawford-style strand burner technique produces data that have limited value for CFD model validation.

Experimental results along the lines of what we are pursuing have been published by Kubota and Ichida (4). They reported results for seven different translucent nitrate ester-based formulations and three formulations containing ammonium perchlorate. The formulations were embedded with a silver, copper, or iron wire. A range of wire diameters was employed. To fabricate test articles, Kubota and Ichida cast the formulations into strands, cured them, cut them in half, placed a conductive wire between them, and then welded the halves back together by applying acetone to the contacting surfaces. The assemblies were then placed in an oven at 40 °C for 72 h to remove the acetone and homogenize the propellant. Strands were combusted in an inert (nitrogen) atmosphere at pressures ranging from 7 to 50 atm (100 to 750 psi). The translucent nature of the formulations enabled flame fronts to be imaged. However, only a few results related to surface topology were presented. Those that were presented confirm the expectation

that the surface topology evolves with time and therefore has the potential to undermine results obtained from a standard Crawford-style strand burner. Moreover, they indicate that, even at steady state, the topology depicted in figure 1 is (at best) only valid for a limited radial distance (r) from the wire. Without knowledge of the actual topology for all r , the prediction of mass/gas generation rates could be problematic.

Recognizing the need for experimental data that characterize the surface topology and regression rate of wire-embedded AMRDEC formulations, ARL sought to conduct such experiments. However, the cost to ship nonstandard formulations and/or test articles between AMRDEC and ARL was prohibitive. We also found that the start-up cost for making the formulations at ARL and fabricating them into test articles would be prohibitive (10). Therefore, the first step of the effort involved identifying energetic materials that were chemically similar to AMRDEC's formulations and that could be readily fabricated into test articles. ARL's ability to model the systems' ignition and combustion with an existing chemical kinetics mechanism was also a consideration.

Driven primarily by fabrication considerations, experiments were developed and conducted with Pentolite. Composed of trinitrotoluene (TNT) and pentaerythritol tetranitrate (PETN), it was melt cast into glass tubes with or without a silver wire strung along the centerline. The resulting items were ignited at one end in a windowed chamber, and events were recorded with a high-speed video camera. However, the results were not encouraging. Pentolite is opaque, and though it was found that light diffusing through unburned material gave some indication of differences in the surface as a function of experimental parameters, there was not sufficient resolution for the images to be of use for model validation.

Based on the experience gained with wire-embedded Pentolite, an alternate sample configuration was devised and tested. In it, the wire was sandwiched between JA2 and a polycarbonate window. This configuration proved to allow direct observation of the burning surface throughout the course of an experiment. JA2 is a standard gun propellant composed of nitrate esters (nitrocellulose [NC], nitroglycerin [NG], and diethylene glycol dinitrate [DEGDN]) and is therefore chemically similar to minimum-smoke rocket propellants. Stock in hand, it was also attractive from a model development standpoint because a chemical kinetics mechanism with a demonstrated ability to model its burning rate as a function of pressure was available (11, 12).

This report provides details of strand configurations that were devised, fabricated, and tested. Results of the experiments conducted with Pentolite and JA2 are summarized. They demonstrate the need for and utility of the windowed strand assembly that was developed. The results obtained for JA2 provide a basis for model validation.

2. Experimental Methods

2.1 Test Article Fabrication

Our initial approach to assembling test articles was similar to the one employed by Kubota and Ichida (4). That is, strips of energetic material were cut from stock pieces, a wire was centered between them, and they were glued together with an adhesive. In our case, the adhesive was made by dissolving slivers of the host propellant in ethyl acetate. Assemblies were held in compression while the adhesive cured. Two different assembly types were fabricated. One was made with 0.100-in.-thick sheets of JA2. The other was made from 0.50-in.-diameter sticks of M9. M9 is a standard double-base gun propellant that is 57.75 weight-percent (wt%) NC and 40 wt% NG. All assemblies contained flaws (introduced mainly by the fabrication process) that prevented proper end burning. Thus, the results were not useful, and alternate fabrication techniques were sought.

Given the difficulties encountered in embedding wires into nitrate ester-based propellants, we decided to consider other energetic material types for use in developing the experimental approach and settled on Pentolite (10). Although it is considered an explosive rather than a propellant, being 50 wt% PETN, which is a nitrate ester, it has chemical similarities to minimum-smoke propellants. Moreover, ARL has extensive experience in casting it. To fabricate wire-embedded strands of Pentolite, a length of wire was tautly suspended along the center of a glass tube, and molten material poured into the tube. (See appendix A for details.) The glass tube was included to prevent flame propagation down the strand's side wall, and it proved to be effective in this role if it did not break. The articles were nominally 0.34 inches in diameter, 1.9 in. high, and had a mass of 4.8 g (figure 2). Ten samples were fabricated. Five had no wire, and five had a 0.010-in.-diameter silver wire (Alfa-Aesar, soft-tempered, 99.9% [metals basis]).



Figure 2. Pentolite cast into glass tube.

The third approach was developed from branch discussions about the experimental difficulties (13). It involved placing a wire along the surface of a 0.100-in.-thick JA2 strip and sandwiching the two components between a pair of 0.200-in.-thick transparent polycarbonate windows. Compression applied via six screw-nut pairs positioned around the windows' edges pressed the wire into the propellant (figure 3). Following compression, the exposed sides of the JA2 were smeared with Dow Corning Molykote 33 medium-consistency grease to prevent flame propagation down them.

For the experiments reported here, the JA2 sheet was cut to a nominal width of 0.4 in. and a height of 3.5 in. The samples were embedded with a single silver wire. Wires with a 0.002-, 0.005-, or 0.010-in. diameter were employed. To establish baseline burning rates, articles without an embedded wire were also fabricated. To test if air gaps were introduced by the embedding process, assemblies with a 0.008-in.-diameter nylon wire were fabricated and tested.



Figure 3. Windowed test article: JA2 strip embedded with a silver wire.

2.2 Measurement Techniques

All experiments were conducted in ARL's low-pressure strand burner (14, 15) (figure 4). The apparatus includes a windowed chamber capable of being pressurized up to 10 MPa. It also includes a ballast tank that adds considerably to the system's overall volume, minimizing pressure increases due to propellant combustion. Nitrogen was employed as the pressurizing gas. Pressure was measured with both a Setra Systems pressure transducer and a Heise mechanical dial gauge. The desired chamber pressure for each experiment was established just prior to ignition. Ignition was achieved by running current through a nichrome wire placed on top of the sample. Events were recorded with a Phantom high-speed camera. Images were acquired at 60 frames per second (fps). Exposures were 1 μ s. To prevent smoke and soot buildup from obscuring the camera's view, a slow, steady stream of nitrogen was flowed through the chamber over the course of each experiment.

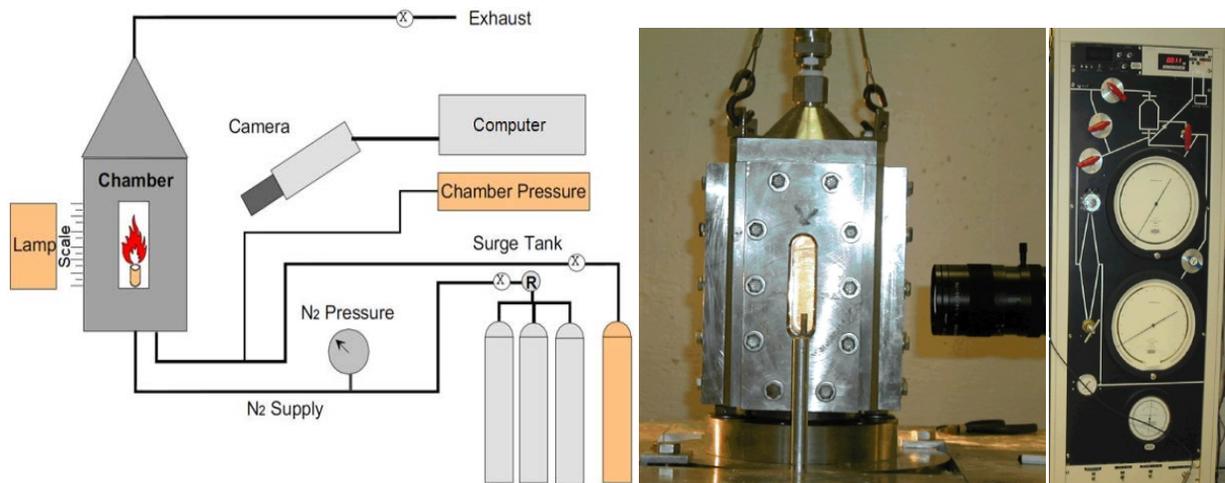


Figure 4. Schematic of strand burner facility (left), windowed strand burner (middle), and strand burner control panel (right).

3. Results

3.1 Pentolite

Experiments with Pentolite-filled test articles were conducted at pressures ranging from 2.0 to 6.2 MPa. Figure 5 shows a representative image of a nonwired sample's burning. The actual flame front is obscured by soot formation on the glass tube. Images obtained from experiments with wired samples were similar. The most noticeable difference between the two cases was the amount of light observed diffusing from unburned portions of the sample, with the wire-embedded strands producing more. We assume the difference is attributable to the formation of a conical burning surface in the wired embedded samples, but Pentolite's opacity prevented direct visualization of the internal surface.

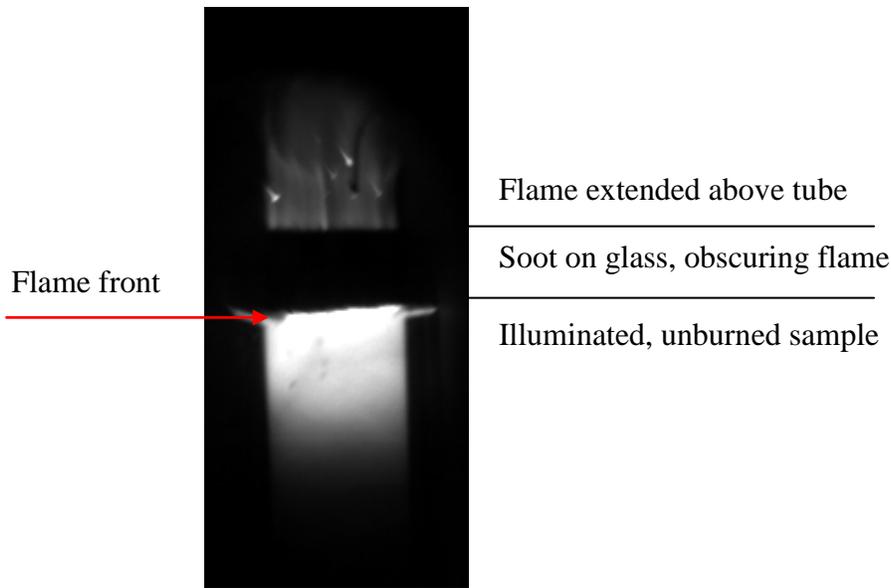


Figure 5. Pentolite burning in glass tube.

Although the imaging results were disappointing from the standpoint of characterizing surface topologies, the recordings still proved to be instructive. Employing the bottom line of soot formation as a marker of the flame front's position, we measured it as a function of time to establish burning rates. Figure 6 shows representative examples of data acquired for strands with and without an embedded wire. Other examples are provided in appendix B, and table 1 provides a reduction of the data. For strands without an embedded wire, derived (linear) burning rates were constant over the entire duration of each trial. In trials with wired samples, on the other hand, an initial phase in which the strand burned at essentially the same rate as the unwired strand was followed by acceleration to a second, higher rate. As such, these results demonstrate the possibility that a measurement based solely on knowledge of the surface's position at two different times (as it is in standard Crawford-type measurements) could lead to poor predictions for the gas generation rates of wire-embedded grains.

Pentolite Burning at 3.50 MPa

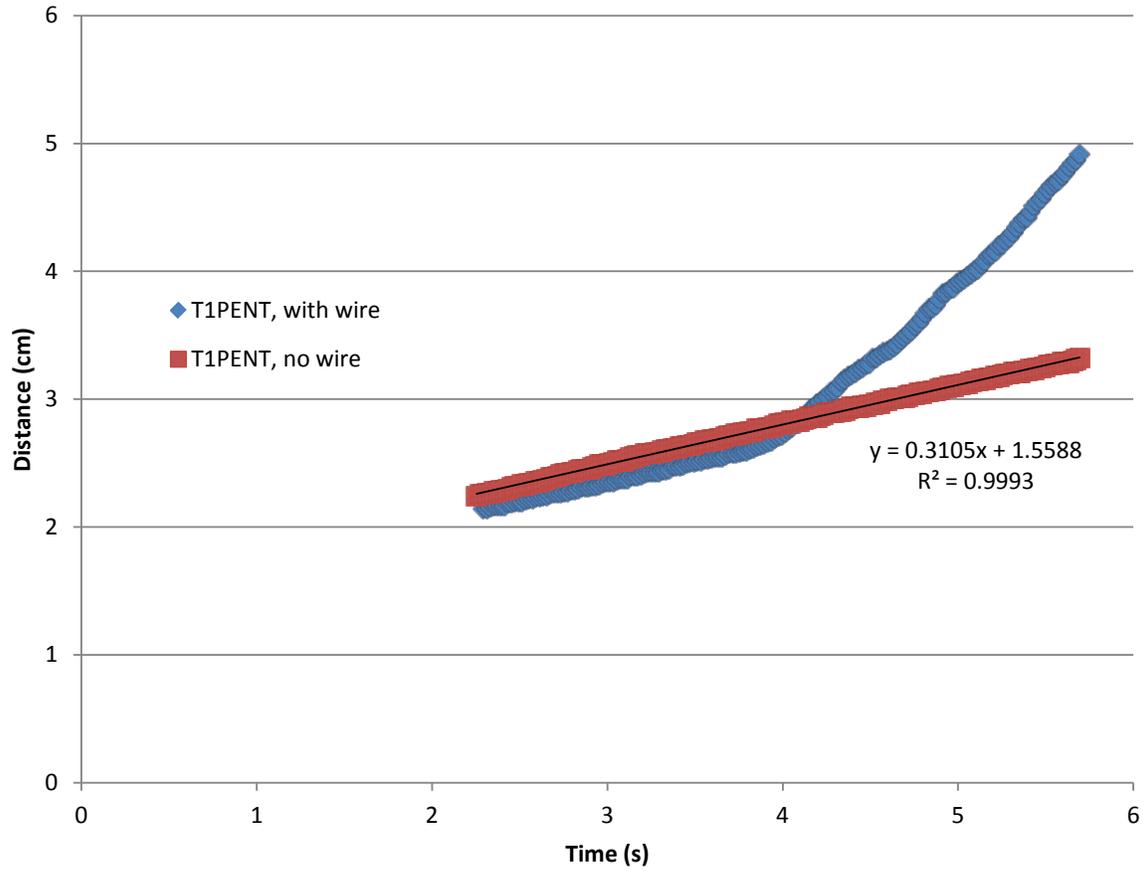


Figure 6. Pentolite burning at 3.50 MPa: rates with and without an embedded 0.010-in. silver wire.

Table 1. Pentolite burning rate data.

Trial	Pressure (MPa)	Burning Rate (cm/s)	R ²	Wire?	Phase	% Increase
1	2.00	0.160	0.9974	Y	1	—
—	2.00	0.447	0.9965	Y	2	179
2	3.50	0.301	0.9999	N	—	—
—	3.50	0.299	0.9999	N	—	—
3	3.50	0.305	0.9972	Y	1	—
—	3.50	1.327	0.9846	Y	2	335
4	3.50	0.303	0.9988	Y	1	—
—	3.50	1.265	0.9950	Y	2	218
5	4.85	0.412	0.9999	N	—	—
6	6.20	0.517	0.9997	N	—	—
7	6.22	0.488	0.9981	Y	1	—
—	6.22	a	—	Y	—	—

^aThe sample failed catastrophically prior to transitioning to a second phase.

3.2 JA2

Experiments with the windowed JA2 assemblies were performed at 3.45, 5.40, and 6.93 MPa. Representative images acquired from various tests are shown in figure 7. Similar to the nonwired Pentolite samples, the nonwired JA2 samples showed good end-burning behavior. This behavior was also observed for the sample embedded with a 0.008-in. nylon wire, suggesting that air gaps were not introduced by the embedding process.

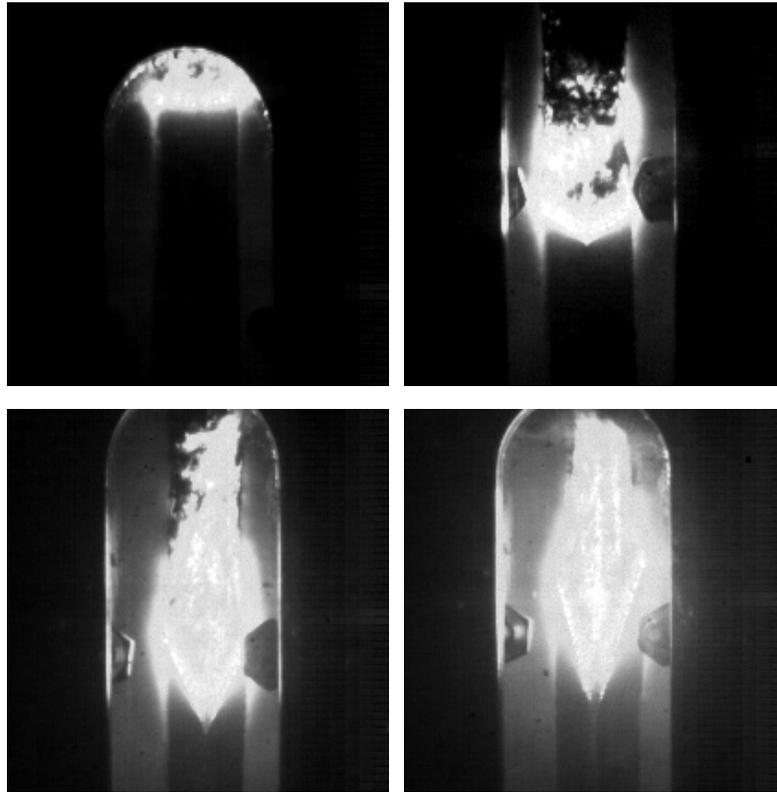


Figure 7. JA2 burning samples at 6.90 MPa; baseline, no wire (top left), 0.002-in. silver (Ag) wire (top right), 0.005-in. Ag wire (bottom left), and 0.010-in. Ag wire (bottom right).

For samples embedded with a silver wire, conically shaped burning surfaces were clearly observed, and marked differences in their shape were found within the range of the parameter space that was investigated. Interestingly, in contrast to the tests with wire-embedded Pentolite, where the data indicated that a considerable amount of time elapsed before full transition to a higher burning rate, the images showed that the surfaces in these tests quickly transitioned to their shape at steady state, and the burn rate was constant throughout most of each trial. An example of this behavior is illustrated by the results shown in figure 8. Until about 0.2 s, the rate of regression at the centerline was higher than the rate of regression of the edge. However, after 0.2 s, the two rates were effectively the same.

JA2 with 0.010 in Ag wire, 6.93 MPa

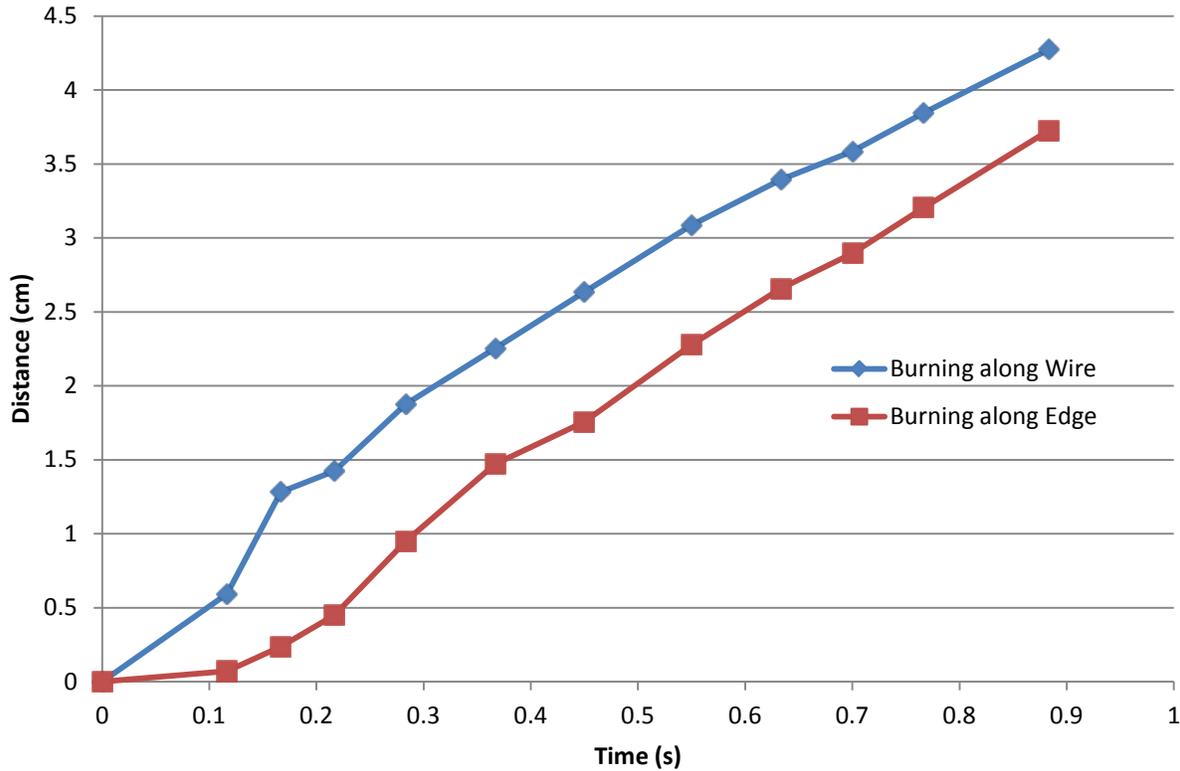


Figure 8. Distance measurements taken at the outer edge and centerline.

Based on this finding, a linear burning rate for each test condition was calculated simply by measuring the position of the centerline as a function of time. Table 2 and figures 9–11 show the results. They indicate that at the three (nominal) pressures at which the tests were conducted, the burning rate increased with the wire’s diameter; the fastest rates at each pressure were produced with the 0.010-in. wire. In addition, we observed that the burn rate increase was higher at the highest pressure tested. At 3.45 MPa, the burning rate increased 110%. At 6.93 MPa, the burning rate increased 350%. A volumetric burning rate (VBR) increase was also calculated from the data. To calculate the VBR, the linear burning rate is divided by $\sin(\theta)$.

Table 2 also includes cone angle values, θ (defined in figure 1), that were measured in the images and calculated based on the ratio of measured rate to the rate measured for the nonwired sample at the same pressure. Measured values fluctuated 2° – 3° based on when the measurement was taken in the burning event. The 3.45-MPa, 0.005-in. wire event fluctuated throughout the entire burning event; therefore, the value reported is a range of the highest and lowest measured angles. Overall, the measured and calculated values are in good agreement.

Table 2. JA2 burn rate data.

	P (MPa)	Burning Rate ^a (cm/s)	R ²	θ (calculated) (°)	θ (measured) (°)	Burning Rate Increase (%)	VBR Increase (%)
No wire	3.45	0.549	0.9968	90	90	—	—
0.002-in. wire	3.45	0.635	0.9996	59.8	68	16	34
0.005-in. wire	3.45	0.759	0.9977	46.3	45–60	38	91
0.010-in. wire	3.45	1.156	0.9989	28.4	30	111	344
No wire	5.40	0.889	0.9991	—	—	—	—
0.002-in. wire	4.80	0.762	0.9965	—	—	-14	—
0.005-in. wire	5.05	0.935	0.9988	—	—	5	—
0.010-in. wire	5.22	1.720	0.9995	—	—	93	—
No wire	6.93	1.057	0.9999	90	90	—	—
0.002-in. wire	6.87	1.481	0.9981	45.5	43	40	96
0.005-in. wire	6.92	2.588	0.9981	24.1	25	144	500
0.010-in. wire	6.93	4.740	0.9784	12.9	15	348	2007

^aMeasured at the centerline

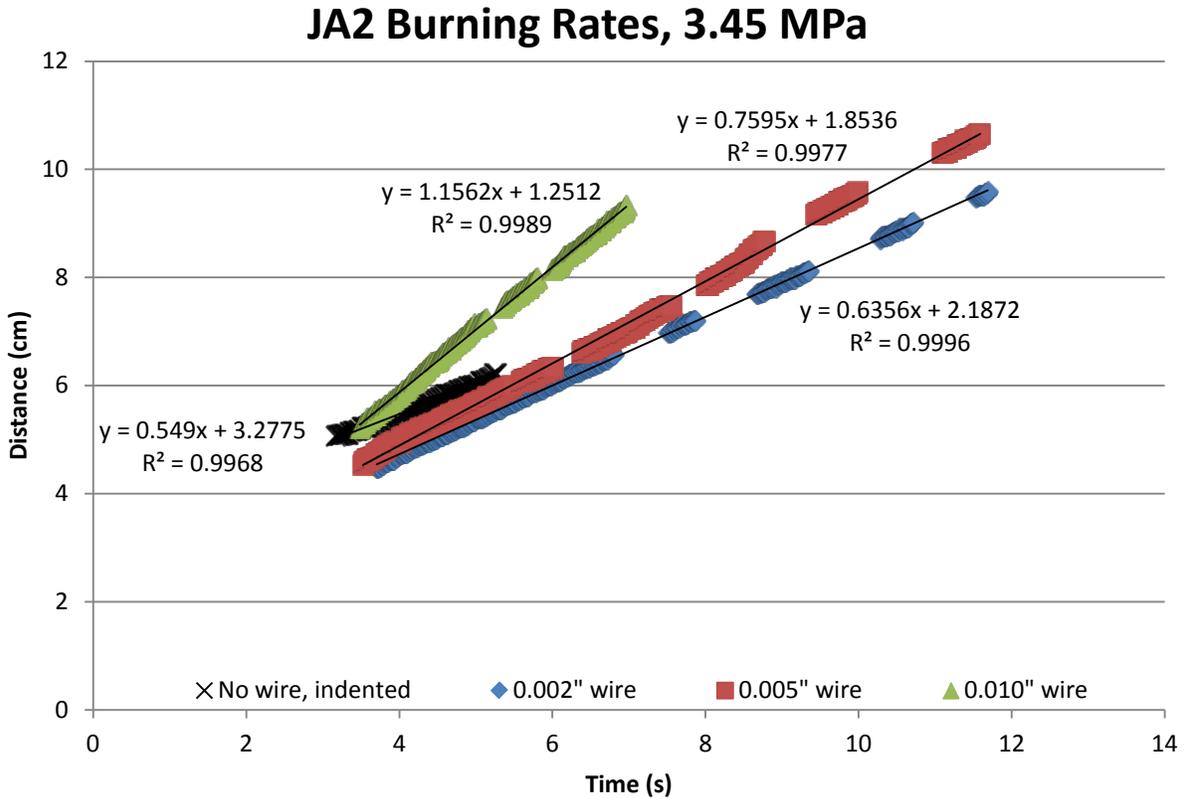


Figure 9. JA2 burning rates at 3.45 MPa.

JA2 Burning Rates, Nominal 5.18 MPa

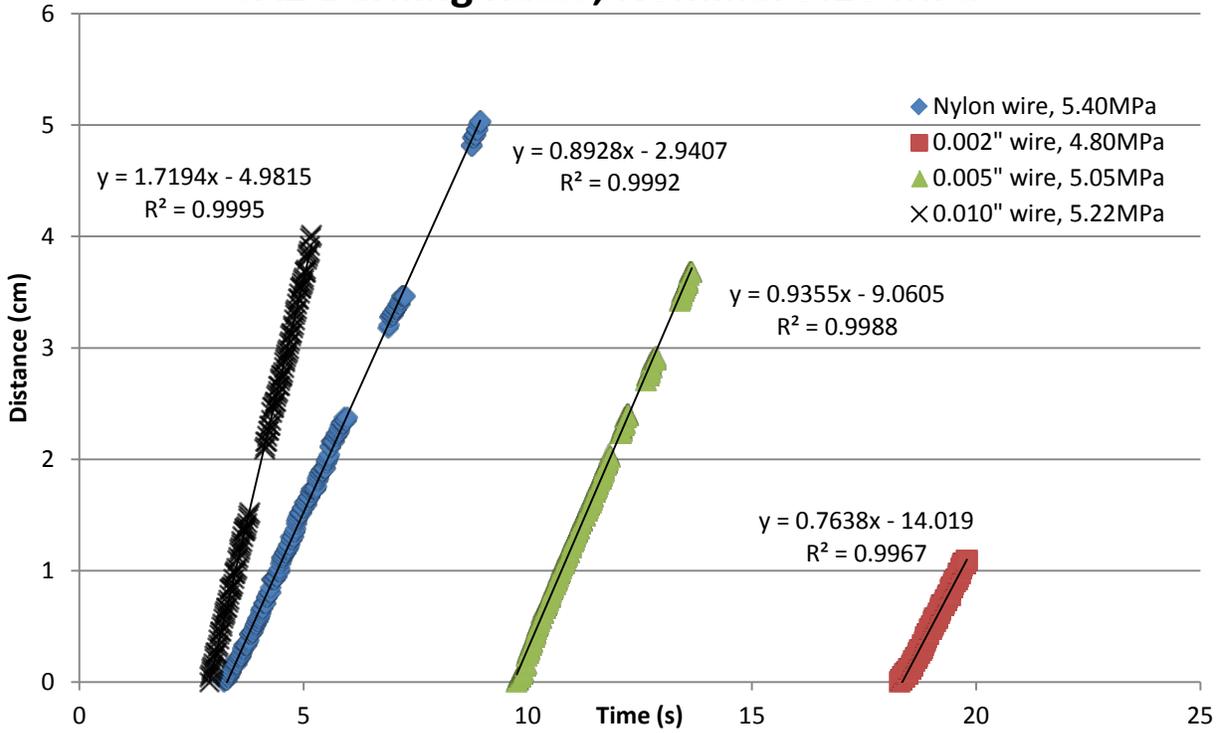


Figure 10. JA2 burning rates at pressure near 5.18 MPa.

JA2 Burning Rates, 6.90 MPa

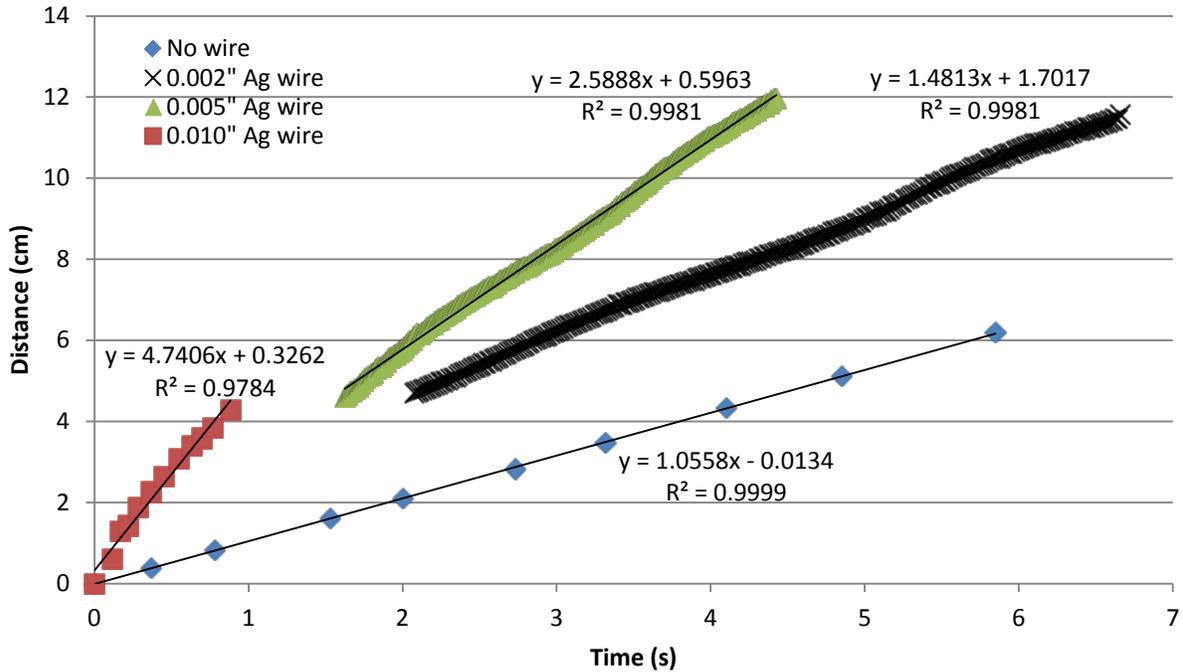


Figure 11. JA2 burning rates at 6.90 MPa.

4. Other Observations

Beyond enabling direct observation of the burning surface, regardless of sample opacity, the windowed strand configuration has several advantages over the other configurations that were investigated. It also allows the parameter space of any sufficiently pliable material to be studied without having to design and construct elaborate casting hardware, and a wide range of sample dimensions can be accommodated. These valuable attributes should facilitate future testing of AMRDEC formulations.

The results also testify to the considerable range of burning rate modification that can be achieved with this approach. The parameter space investigated in the current study is only a tiny fraction of the nearly limitless combinations of wire material, diameter, shape, and spatial arrangement that could be employed. As such, the technology should help motor developers meet performance objectives with propellant formulations whose burning rates have been compromised in the interest of achieving lower sensitivities. In addition, it is clear that meeting those objectives will be greatly facilitated by having models that can make reliable predictions for the performance of proposed designs.

5. Summary

Techniques for imaging the burning surface of wire-embedded minimum-smoke propellants were developed and tested. Test articles fabricated with Pentolite and JA2 were employed for the effort. Embedded with a silver wire, the energetic material was ignited at one end in a windowed chamber, and events were recorded with a high-speed video camera. Results obtained with the Pentolite samples revealed the possibility that burning rates measured via a standard Crawford style (two-point break wire) technique could be in error. They also showed that when embedded in the interior of an opaque material, one's ability to characterize the evolution of the surface's topology is seriously compromised. Being a potential impediment to the acquisition of data relevant to minimum-smoke formulations, this issue prompted the development and testing of a design in which the wire was embedded at the propellant's surface, and the surface sealed with a polycarbonate window. It was found to enable direct observation of the burning surface of JA2, with videographs revealing in great detail the evolution of the surface from ignition to a final steady-state form. Results for a variety of wire diameters and pressures were acquired, and they can be employed to validate a state-of-the-art CFD model that ARL is developing to accelerate the development of wire-embedded minimum-smoke propellant technology.

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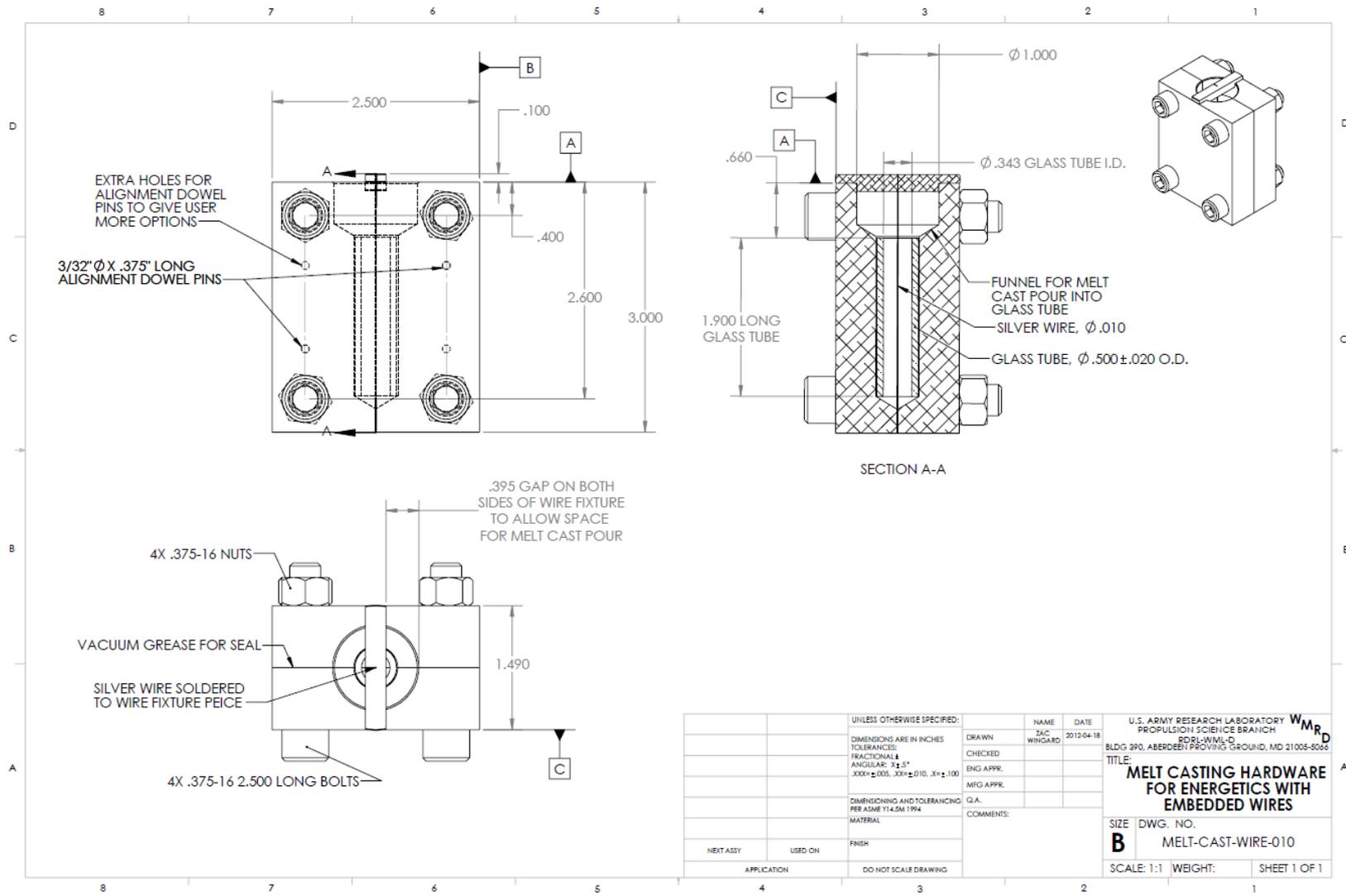
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Appendix A. Pentolite Melt Casting Hardware Drawing

This appendix appears in its original form, without editorial change.

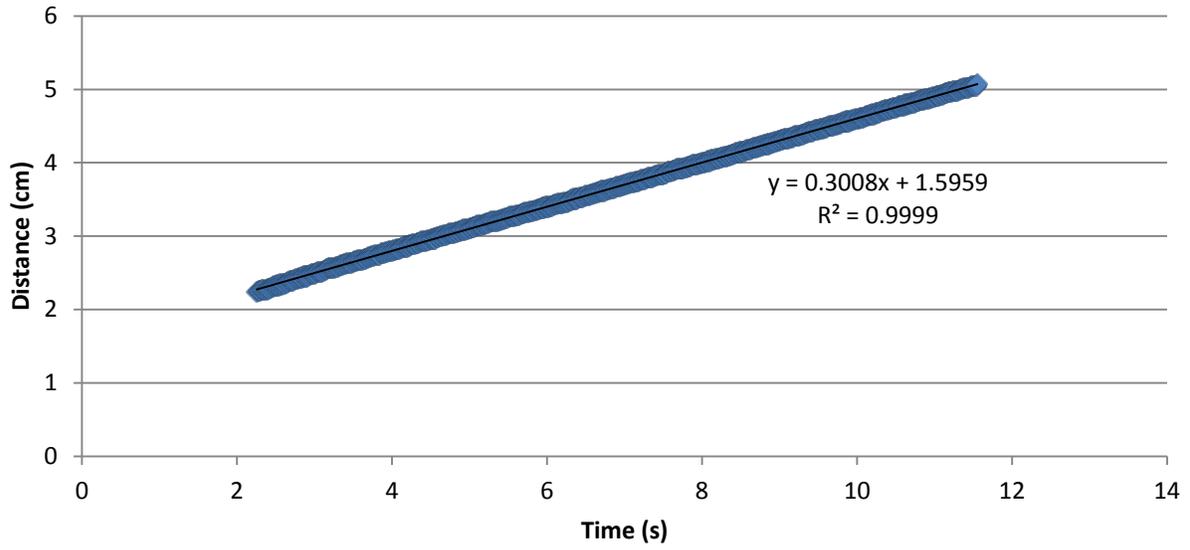


UNLESS OTHERWISE SPECIFIED:		NAME	DATE	U.S. ARMY RESEARCH LABORATORY PROPULSION SCIENCE BRANCH BDR-OWNL-D BLDG 390, ABERDEEN PROVING GROUND, MD 21005-5066
DIMENSIONS ARE IN INCHES TOLERANCES: FRACTIONAL: \pm ANGULAR: \pm 8" XXX \pm .005, .XXX \pm .010, .X \pm .100		ZAC WINGARD	2012 04 18	
DIMENSIONING AND TOLERANCING PER ASME Y14.5M 1994		DRAWN		TITLE: MELT CASTING HARDWARE FOR ENERGETICS WITH EMBEDDED WIRES
MATERIAL		CHECKED		
NEXT ASSY		ENG APPR.		
USED ON		MFG APPR.		SIZE DWG. NO. B MELT-CAST-WIRE-010
APPLICATION		FINISH		SCALE: 1:1 WEIGHT: SHEET 1 OF 1
		COMMENTS:		

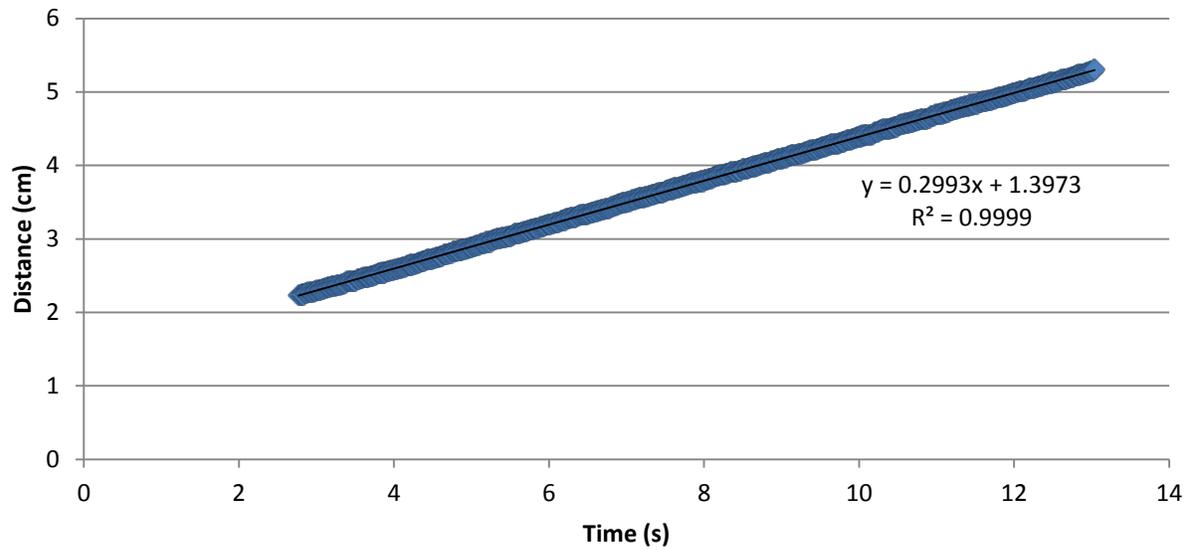
Appendix B. Measured Burning Rate Data for Pentolite Experiments

This appendix appears in its original form, without editorial change.

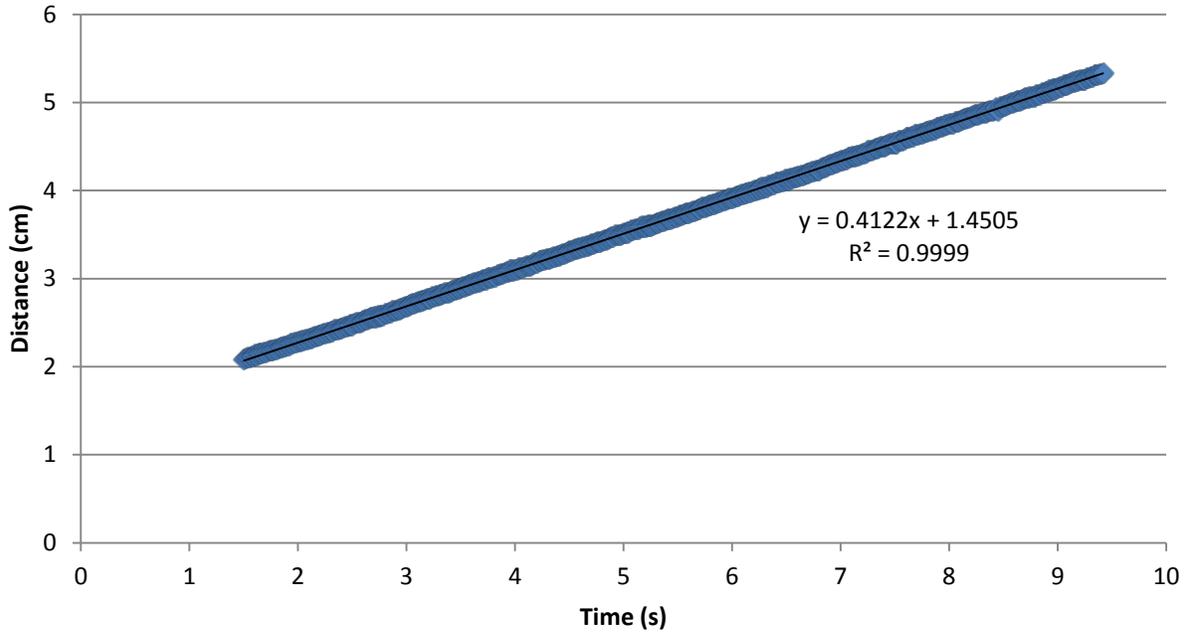
Pentolite no wire, 3.50 MPa shot 1



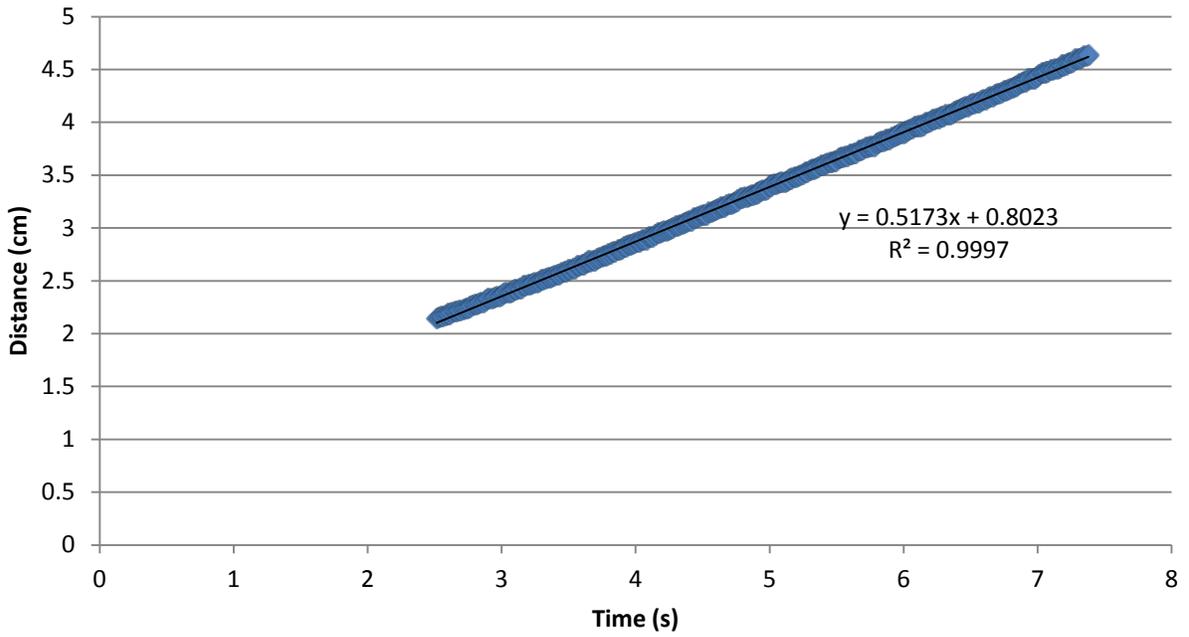
Pentolite no wire, 3.50 MPa shot 2



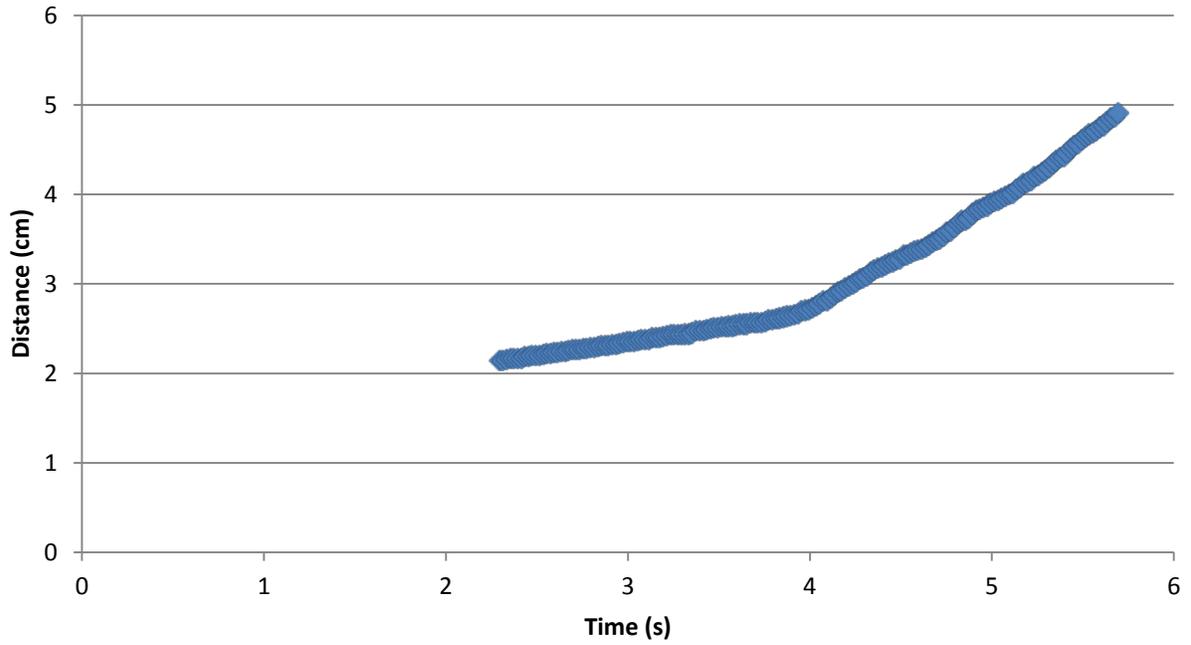
Pentolite no wire, 4.85 MPa



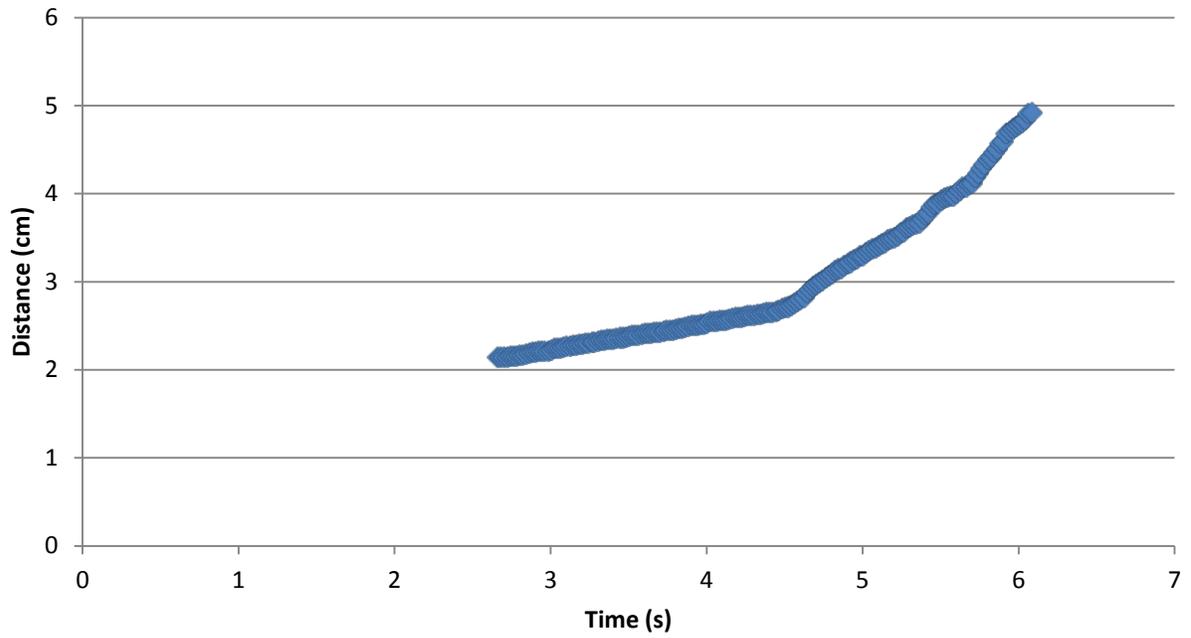
Pentolite no wire, 6.20 MPa



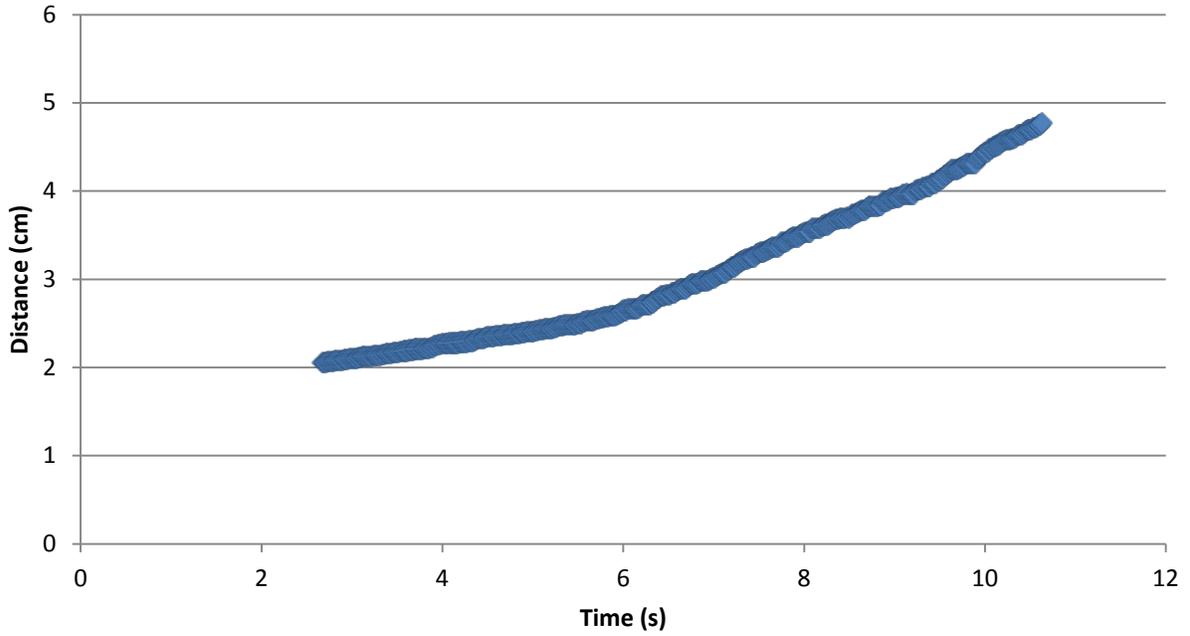
Pentolite w/wire, 3.50 MPa shot 1



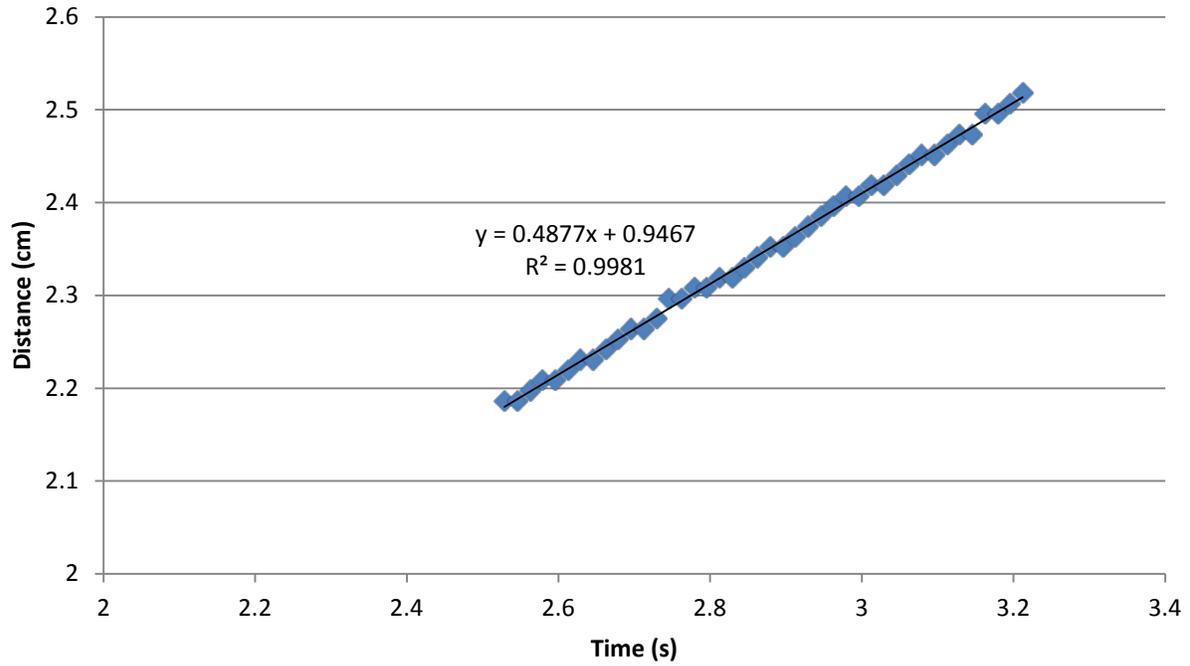
Pentolite w/wire, 3.50 MPa shot 2



Pentolite w/wire, 2.00 MPa



Pentolite w/wire, 6.22 MPa, prior to tube failure



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List of Symbols, Abbreviations, and Acronyms

θ	theta; cone angle of flame
Ag	silver
AMRDEC	U.S. Army Missile Research, Development, and Engineering Center
ARL	U.S. Army Research Laboratory
cm	centimeter
cm/s	centimeter per second
DEGDN	diethylene glycol dinitrate
fps	frames per second
g	gram
h	hours
in.	inch
P	pressure
psi	pounds per square inch
MPa	megapascal
N ₂	nitrogen
NC	nitrocellulose
NG	nitroglycerin
R ²	coefficient of determination
s	second
TNT	trinitrotoluene
μ s	microsecond

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RDRL WMM C
P KASTE