

## **Initial Testing of a Prototype Laser Ignition Chamber**

**by Jennifer L. Gottfried, Chase A. Munson, Joe Colburn, and  
Richard A. Beyer**

**ARL-TR-6862**

**March 2014**

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**Weapons and Materials Research Directorate, ARL**

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## 1. Introduction

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Laser ignition or initiation of energetic materials has been an active area of investigation for more than four decades based on its potential advantages over traditional ignition schemes, including improving system reliability and safety, simplifying the ignition train through elimination of the primer, and facilitating the ignition of insensitive munitions (see, e.g., 1–9). Methods for laser ignition include direct initiation through thermal heating (with or without impurities that strongly absorb laser radiation), laser-induced microplasma/spark initiation, photochemical ignition, and indirect initiation via laser-driven flyer plate impact or through a shocked thin film.

The Laser Ignition in Guns, Howitzers, and Tanks (LIGHT) program at the U.S. Army Research Laboratory (ARL) began in the early 1990s (10). As part of this program, laser beam parameters, such as pulse width, pulse wavelength, pulse duration, pulse energy, and repetition rate, have been investigated for the ignition of many energetic materials, including igniter materials such as black powder, nitrocellulose, M44 propellant (44% nitroglycerin, 52% nitrocellulose), and numerous other propellants (11–34). Lasers used for these studies included Nd:glass lasers (up to 30 J at 1.054  $\mu\text{m}$  with variable pulse widths from 100  $\mu\text{s}$  to 10 ms), Nd:YAG lasers (up to 1 J at 1.064  $\mu\text{m}$ , 100- $\mu\text{s}$  pulse width; 10 J at 1.064  $\mu\text{m}$ , 5-ms pulse width; or 355 nm/266 nm with a 10-ns pulse width), a continuous-wave (CW) CO<sub>2</sub> laser (10.6  $\mu\text{m}$ , 10–100 W), and CW diode lasers (980 nm, 60 W or 910 nm, 100 W). It was observed that some propellants which do not ignite in air with certain lasers were effectively ignited when enclosed in a laser ignition chamber (14).

Laser ignition is dependent on energy density rather than total energy, thus it is important to ensure the spatial uniformity of the laser (e.g., no hot spots that might be difficult to reproduce). Other considerations for the laser-material interaction include the absorption coefficient of the energetic material at the laser wavelength (i.e., surface reflectivity), the composition and extent of gas generation during combustion, the thermal diffusivity of the material, and the flame-spread to adjacent grains or energetic crystals.

Based on previous experiments at ARL, a modified laser ignition chamber has been designed to enable the measurement of pressure-time traces for new types of insensitive and nonconventional energetic materials currently under investigation for military applications. The results from initial testing of the prototype chamber with black powder are presented in this report.

The combustion and flame-spread of black powder (a mixture of 12% sulfur, 16% charcoal, and 72% sodium nitrate) ignited by a pulsed laser was first studied in 1976 (35), followed by numerous subsequent studies (10, 12, 20, 27, 30, 36–40). Black powder has been shown to be extremely amenable to laser ignition over a wide range of pulse energies and pulse widths (12).

Reliable ignition of black powder was achieved using near-infrared laser pulses with widths greater than 1 ms and several joules of energy. It was found that larger pulse widths resulted in increased ignition delays with lower ignition thresholds, while increasing the laser energy decreased the ignition delay; tightly focusing the laser resulted in a substantial increase in ignition delay times or lack of sustained combustion (10). Although most laser ignition studies concentrate on near-infrared laser wavelengths due to cost and portability concerns, ignition of black powder at a wide range of laser wavelengths is likely possible because of its broad absorption spectrum (41), especially compared to other energetic materials. For this experiment, a CW ultraviolet (UV) laser was selected to ignite the black powder based on its availability and portability.

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## 2. Experimental

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### 2.1 Chamber Design

A schematic of the modified three-piece laser ignition chamber constructed from stainless steel is shown in figure 1. The first piece is designed to hold the window used to transmit the laser radiation. The center piece holds the energetic material in a chamber with a volume of 503 mm<sup>3</sup> (0.0307 in<sup>3</sup>) and contains a port for a pressure transducer. The third piece secures a polyethylene terephthalate (Mylar,\* type A) diaphragm to the center piece. A rapid increase in pressure from the gasification process occurring during black powder combustion leads to rupture of the Mylar diaphragm; the Mylar blowout disk serves as a laboratory safety feature to prevent overpressurization, which could damage the pressure gauge or window. Figure 2 shows photographs of the assembled laser ignition chamber.

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\*Mylar is a registered trademark of Dupont Teijin Films for its brand of polyester film.

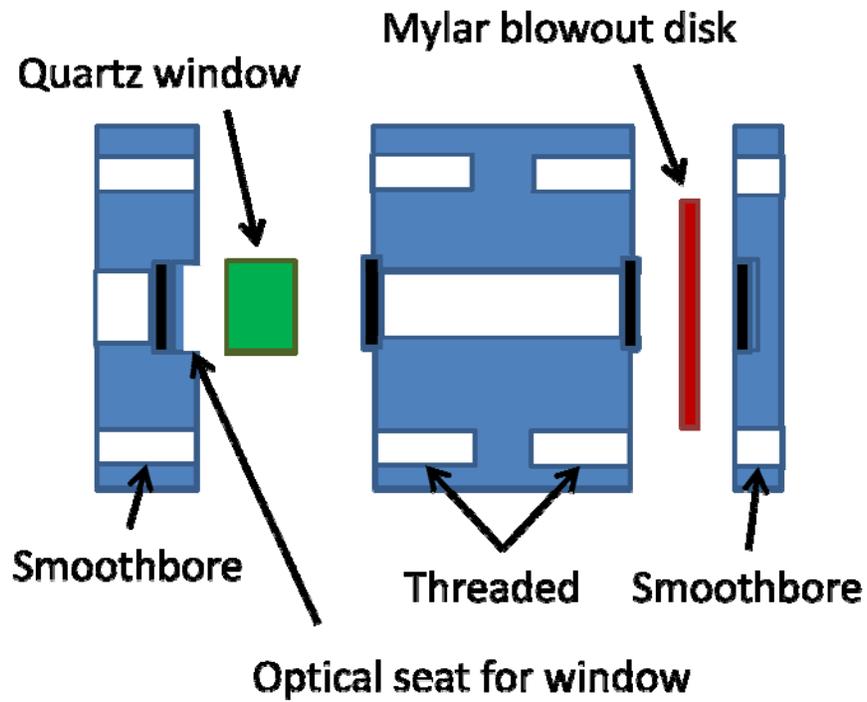


Figure 1. Schematic of the three-piece laser ignition chamber. The pressure port is located on the side of the center piece.

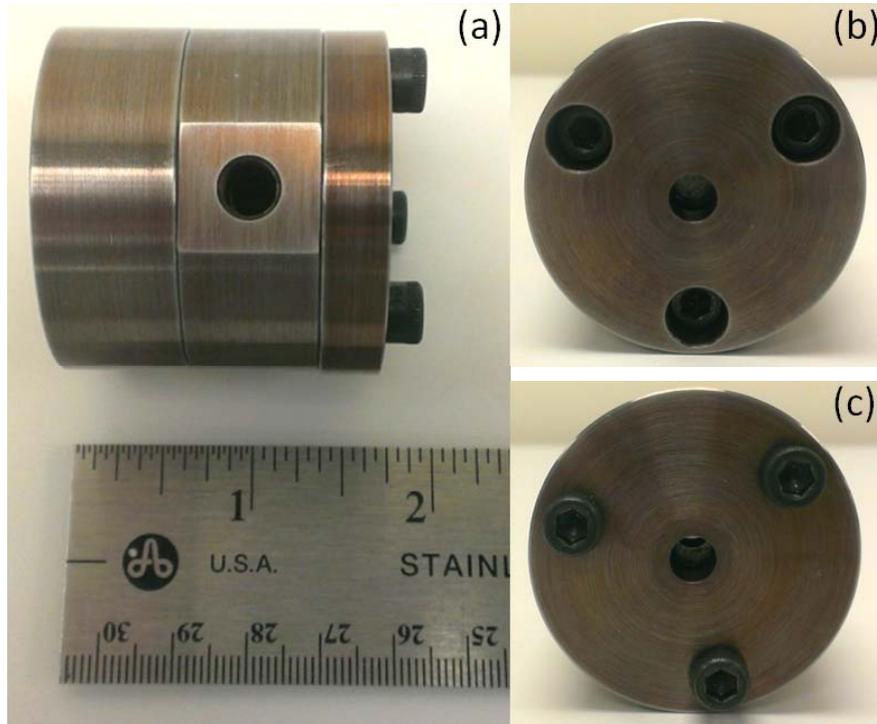


Figure 2. Photographs of the laser ignition chamber, including (a) the side view with threaded pressure transducer port, (b) front view of optical window, and (c) rear view of blowout disk port.

## 2.2 Optical Windows

Optical window selection was constrained by commercial availability—we did not want to pursue custom manufacturing—and our experimental requirements; specifically, optical windows needed to (1) be highly transparent at a wide range of laser ignition source wavelengths (from the UV into the infrared) and (2) possess sufficient mechanical strength to withstand estimated internal chamber pressures. Previous laser ignition experiments used a Mylar film for both a blowout disk and the optical window. Other commercially available windows under consideration for use in our optical cell were composed of fused silica or sapphire, 12.7 mm in diameter, and had varying thicknesses (sapphire, 3 mm; fused silica, 6.4 and 9.5 mm).

### 2.2.1 Window Optical Transparency

Laser radiation not transmitted through an optical window is either reflected/scattered from the window surface (typically a very small component) or absorbed into the material matrix. Absorption of laser energy into the material may produce optical damage leading to further reductions in transparency. In our experiments, damage to the optical windows could lead to insufficient energy for laser ignition within the chamber and an increase in the potential for misfires.

In figure 3, we show optical transmission profiles of three potential window materials: UV-fused silica, sapphire, and Mylar (type A). While both fused silica and sapphire exhibit relatively high transmission (85%–95%) over the visible to near-infrared regions of the spectrum, fused silica consistently outperforms sapphire by about 10% greater transmission despite being twice as thick (absorbance increases with transmission path length). Type A Mylar has 0% transmission in the UV and significantly poorer transmission throughout the visible and infrared regions; thus, it was excluded from further consideration as an optical window. Windows examined in this work—76- $\mu\text{m}$ -thick Mylar, 3-mm-thick sapphire, and 6.4- and 9.5-mm-thick fused silica—are thinner than those used in this figure, so we can expect that their optical transparency performance should be similar to or better than those displayed here.

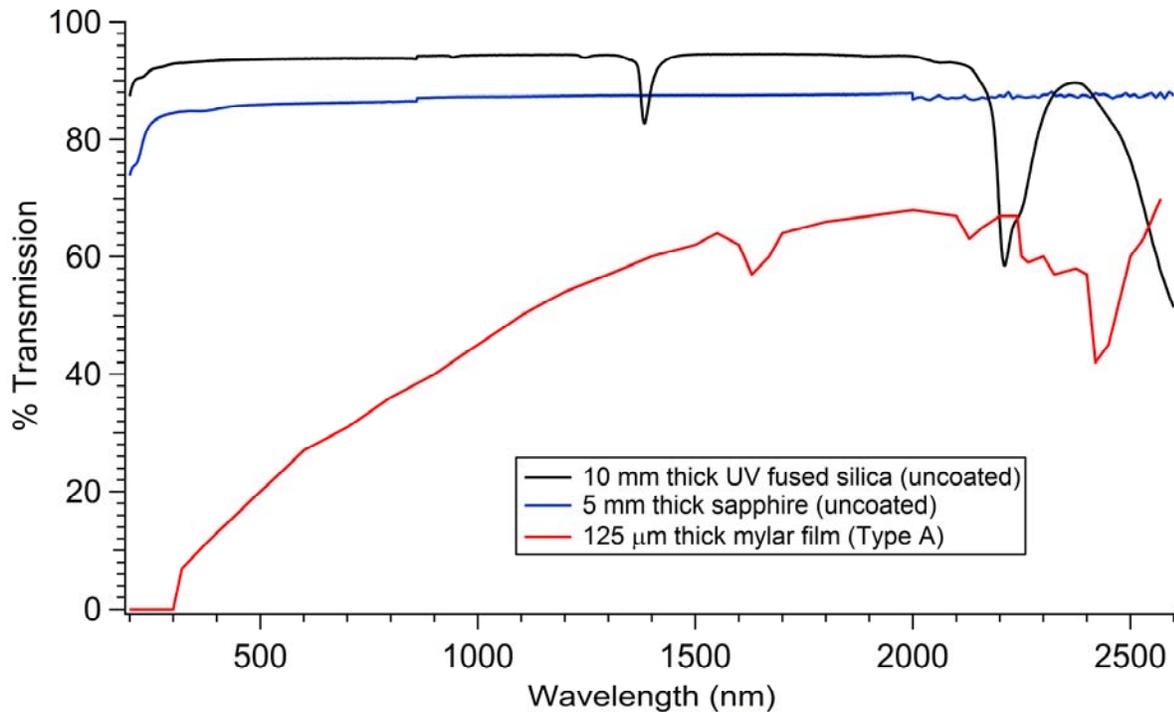


Figure 3. Optical transmission profiles of fused silica (42), sapphire (43), and Mylar (44).

### 2.2.2 Window Mechanical Strength

To ensure that the optical test cell performed as designed (i.e., controlled vessel failure through the Mylar burst disk), we wished to select optical windows whose anticipated failure loads exceed that of the burst disk by a factor of at least 2–3. For the dynamic range we expect to observe, 0–4000 psi, it was anticipated that a minimum of approximately 10,000 psi is required to allow us to safely collect the desired data.

Estimation of the failure loads on fused silica and sapphire optical windows was performed using equations developed for calculating uniform loads on flat plates (45). These equations make several assumptions: (1) the plate is flat, uniform, and isotropic, (2) the thickness is less than one-fourth the shortest transverse dimension, (3) the applied force is perpendicular to the plate, and (4) the plate is not stressed beyond the elastic limit.

Not all of our commercially available windows adhere to these assumptions; specifically, the 6.4- and 9.5-mm fused silica windows exceed the thickness-to-diameter ratio defined in assumption 2. For thicker specimens such as these, Roark provides theoretical expressions for failure loads placed on a short column structure (45). These equations are more complex, including terms for failure due to normal loads and bending fracture. Additionally, they require fitting to experimental test data, not readily available for optical materials. Because we do not have the required experimental data and our optical window is rigidly constrained by the cell

design, we have chosen to use the less rigorous flat plate formulas (45) to approximate our failure loads despite not meeting all the assumptions.

Brittle fracture in a flat plate occurs when the maximum tensile stress,  $\sigma_{\max}$ , is equal to the ultimate tensile strength of the material. In a flat plate,  $\sigma_{\max}$  may be related to the center moment,  $M_C$ , and plate thickness,  $t$ :

$$\sigma_{\max} = \frac{6M_C}{t^2} \quad (1)$$

For a simply supported plate (plate rotation and longitudinal displacement are allowed) under a uniform load  $q$ , the center moment  $M_C$  is

$$M_C = \frac{qa^2(3 + \nu)}{16} \quad (2)$$

where  $a$  is the plate radius and  $\nu$  is Poisson's ratio.

The maximum load  $q_{\max}$  for the simply supported plate is found by rearranging equations 1 and 2:

$$q_{\max} = \frac{8\sigma_{\max}t^2}{3a^2(3 + \nu)} \quad (3)$$

For a fixed plate, where only longitudinal displacement of the plate is allowed,  $M_C$  becomes

$$M_C = \frac{qa^2(1 + \nu)}{16} \quad (4)$$

and

$$q_{\max} = \frac{8\sigma_{\max}t^2}{3a^2(1 + \nu)} \quad (5)$$

We evaluated our 12.7-mm-diameter sapphire and fused silica windows using equations 3 and 5. Values used for  $\sigma_{\max}$  and  $\nu$  were 48 MPa and 0.17 for fused silica (46) and 400 MPa and 0.30 for sapphire (47). Results for these optical windows are listed in table 1.

Table 1. Maximum theoretical load for several windows evaluated in this study.

Material (Thickness)	Maximum Theoretical Load MPa (psi)	
	Simple Supported	Fixed Edge
Sapphire (3 mm)	72 (10,000)	183 (27,000)
Fused Silica (6.4 mm)	41 (5,900)	111 (16,000)
Fused Silica (9.5 mm)	90 (13,000)	244 (35,000)

Fixed edge attachment is never truly attained in realistic scenarios. Our optical cell design exceeds the constraint defined by Roark of simple support (45). Thus, we can assume simple supported as a minimal case with the actual sustainable loads somewhere between the simple supported and fixed edge cases.

### **2.2.3 Window Selection**

Final material selection was based upon considerations of optical transmission and the ability to withstand anticipated pressures in our test cell. To maintain the desired pressure load and safety margin requires either the 3-mm sapphire or the 9.5-mm fused silica window. From an optical transmission perspective, fused silica exhibits greater optical transmission than sapphire, even at the greater thickness. Also considered was previous experimental evidence demonstrating a low damage threshold of sapphire windows toward Nd:glass laser emission (17)—a laser considered for use in future experiments with this experimental apparatus. Based on these considerations, we chose to use the 9.5-mm fused silica windows with our optical test cell.

## **2.3 Range Procedure**

A 15-kpsi Kistler-type 601B1 transducer (maximum pressure without damage = 18 kpsi) with a dual-mode amplifier (type 5010) was used for the pressure measurements. A small amount of silicon grease was added to the tip of the pressure transducer to protect the sensor from heat damage, then the transducer was secured in the port with a mounting adaptor; a brass seal was used to ensure the tip of the pressure gauge did not extend into the ignition chamber. With the first two pieces of the chamber assembled, the ignition chamber was filled with the energetic material through the Mylar blowout disk port. Prior to each test shot, 270 mg of class-3 black powder with irregularly shaped grains (typical maximum dimensions 1.2–2.4 mm in diameter) was added to the laser ignition chamber. Although 270 mg was sufficient to fill the entire volume of the laser ignition chamber for the first test shot, subsequent loading with the same mass of black powder resulted in different fill volumes because of the random grain geometry. The final piece of the chamber was assembled after sandwiching the desired number of Mylar sheets between the center section and the third piece.

The loaded ignition chamber was secured in a vice clamp in between the laser and a cardboard box used to catch material ejected when the Mylar diaphragm burst; a laser beam block was placed in the box to prevent the cardboard from catching on fire in case the laser beam passed completely through the ignition chamber. A second beam block was placed between the ignition chamber and laser to prevent accidental ignition of the black powder; this block was removed just prior to exiting the range in preparation for firing the laser.

The unfocused CW laser (approximately 2 mm) was centered through the fused silica window; a button in a locked box outside the range was used to trigger the laser. While the button was depressed, the laser (Opto Engine LLC, model MPL-N-355) provided 125 mW of 355-nm light,

which was sufficient to ignite paper after a few seconds of exposure. For each shot, the laser was fired until the report from the Mylar blowout disk was heard (typically 2–7 s).

As in previous experiments (20), a threshold of 250 psi was selected for triggering the oscilloscope (indicating onset of ignition). The flame-spread was measured as the time from 250 to 700 psi. The combustion by-products were exhausted from the room prior to entering the range. After each shot, the ignition chamber was disassembled and thoroughly cleaned to remove the post-combustion residue from the fused silica window and the inside of the chamber. The undesirable post-combustion residue is one of the disadvantages of black powder, as noted in Chang et al. (27).

The first shot was used to establish the laser pulse duration needed to ignite the black powder (no reaction occurred following the 1- and 5-s pulses; the Mylar burst after a sustained 7-s laser pulse). The pressure signal was not recorded for the first shot because of a grounding error with the oscilloscope. Out of a total of eight shots, two misfires occurred (no pressure rise after the laser was fired for 30 s). When a misfire occurred, the range was kept clear for a waiting period of 15 min. The black powder was then discarded, since it is known that the surfaces of laser-irradiated propellants exhibit chemical and physical changes (48). In the case of both misfires during this testing, a large grain of black powder had gotten stuck on the fused silica window with the silicon grease used on the pressure gauge (presumably transferred during the chamber fill). Based on our visual observations, the laser had melted a small amount of material on the surface of the grain facing the window, which was insufficient to ignite the bulk material.

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### 3. Results

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A previous study on the laser ignition of less than 100 mg of black powder noted that a 0.006-in-thick Mylar sheet resulted in a peak pressure release near 2000 psi (20). For this experiment, we investigated the effect of varying the Mylar diaphragm thickness on the pressure-time behavior of the black powder. Figure 4 presents pressure-time traces for a series of tests with varying Mylar sheet thicknesses. Time zero corresponds to a pressure of 250 psi when the scope was triggered.

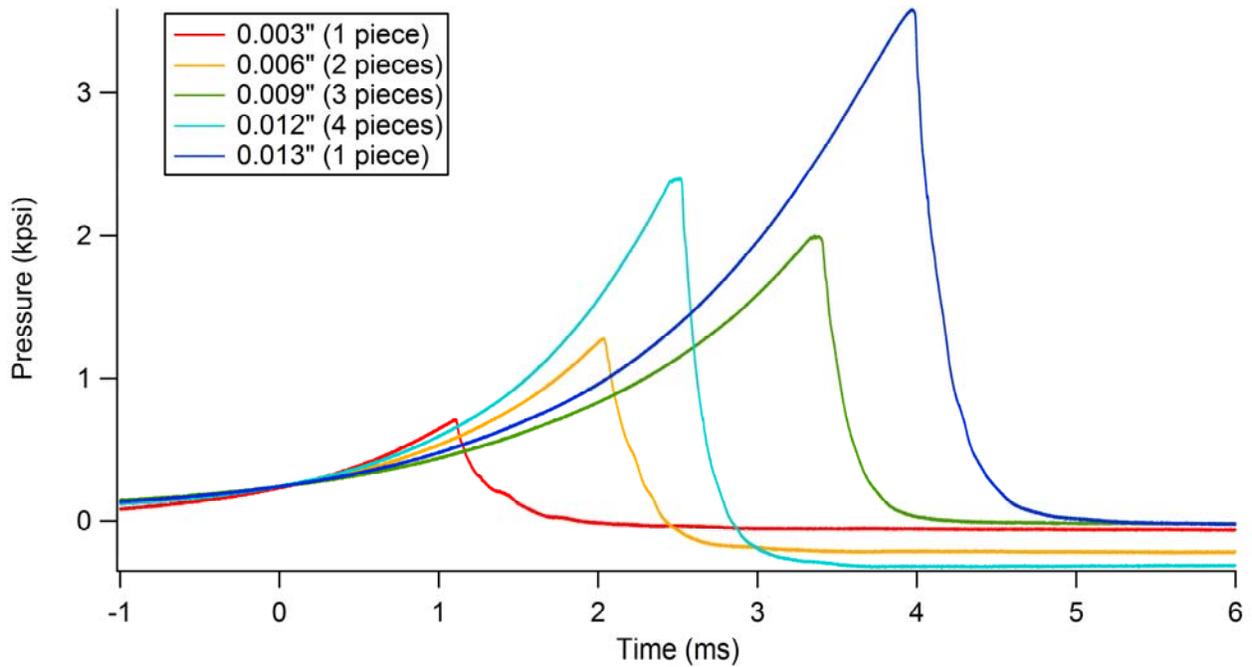


Figure 4. Pressure release from the laser ignition of black powder using Mylar sheets of varying thickness.

Although the ignition behavior of black powder is generally reproducible, random variations in the laser-material coupling have been observed (20). A previous study observed that the flame-spread for black powder was approximately 0.9 ms, with no dependence on laser energy (20). Here, the flame-spread for the five shots varied between 1.1 and 1.7 ms (figure 5); within the limited data set (one shot at each Mylar thickness) there was no correlation between flame-spread and blowout window thickness. Variations in the loading density due to the irregular grain size likely account for the different flame-spread times.

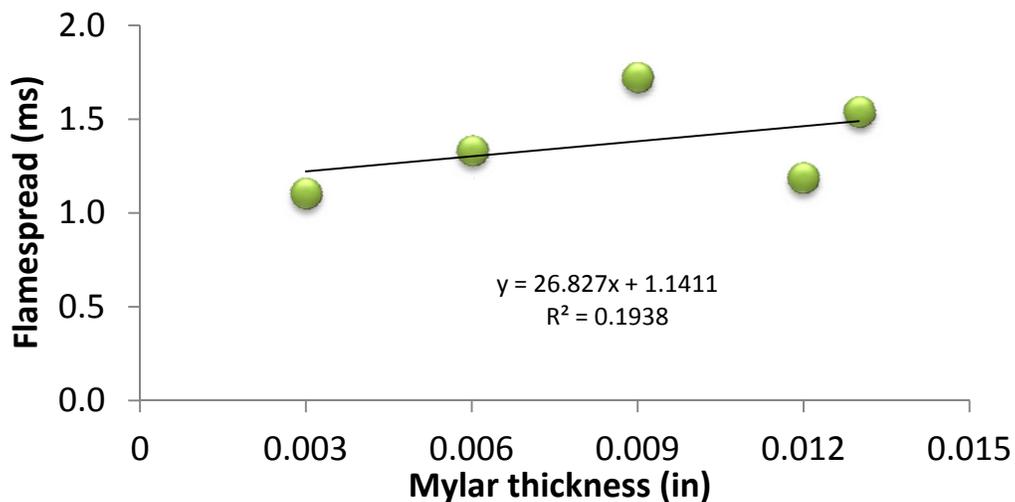


Figure 5. Flame-spread (ms) vs. Mylar sheet thickness (in).

For the four Mylar blowout disks consisting of one to four sheets of 0.003-in-thick (76- $\mu\text{m}$ -thick) Mylar, the peak pressure was linearly related to the total Mylar thickness (figure 6). The single 0.013-in (330- $\mu\text{m}$ ) Mylar sheet had a significantly higher peak pressure. Since the 0.003-in Mylar sheets were more flexible, the 0.012-in Mylar diaphragm consisting of the thinner sheets was able to stretch more during the pressure rise accompanying the combustion, expanding the volume of the ignition chamber and decreasing the peak pressure (compared with the 0.013-in single piece of less flexible Mylar). Figure 7 compares the two Mylar diaphragms post-blowout.

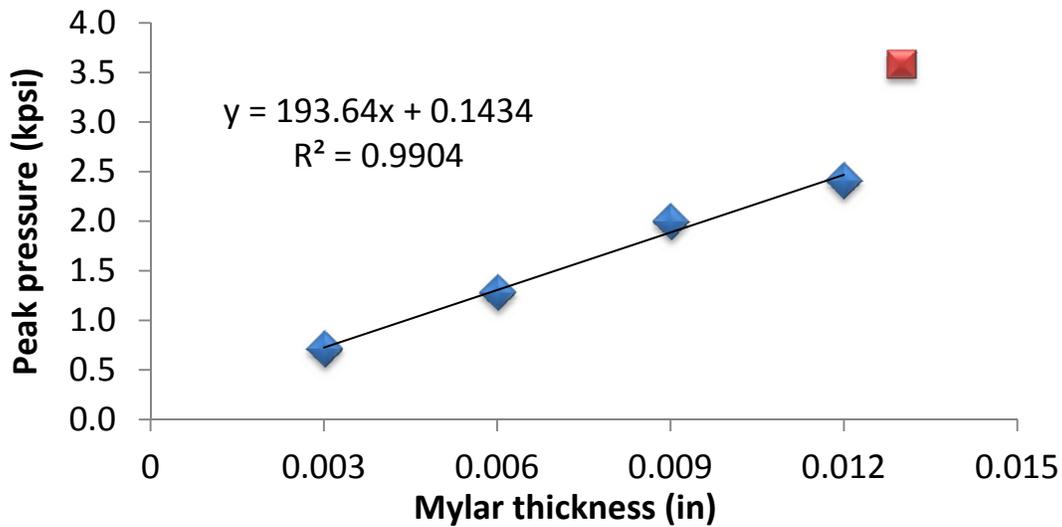


Figure 6. Peak pressure vs. Mylar sheet thickness (blue diamonds: 0.003-in-thick Mylar sheets; red square: 0.013-in Mylar sheet).

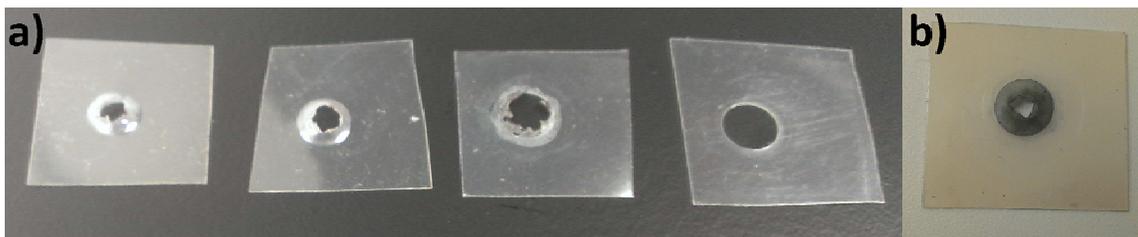


Figure 7. Mylar blowout sheets after laser ignition: (a) four clear 0.003-in sheets stacked together and (b) one white 0.013-in sheet with black powder residue.

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## 4. Conclusions

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Using the prototype laser ignition chamber, we were able to successfully measure the pressure-time behavior of the ignited black powder. It was demonstrated that an unfocused CW UV laser can be used to ignite black powder, although the time to ignition is on the order of seconds rather than milliseconds (as required for gun igniter applications). In addition, the linear relationship between Mylar diaphragm thickness and maximum peak pressure was observed. For future testing, the laser ignition chamber will be used to determine the most efficient ignition source (e.g., laser wavelength, pulse duration) for nonconventional energetic materials currently in development at ARL.

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## List of Symbols, Abbreviations, and Acronyms

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ARL	U.S. Army Research Laboratory
CW	continuous wave
LIGHT	Laser Ignition in Guns, Howitzers, and Tanks
UV	ultraviolet

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