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THE EFFECTS OF PRESSURE ON THE LASER INITIATION OF $TiH_x/KClO_4$ AND OTHER PYROTECHNICS

[redacted]

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ABSTRACT

The ignition thresholds of $TiH_x/KClO_4$, Mg/Teflon, and an Al-torch mix are measured as a function of gas pressure for ignition by 514.5 nm pulses from an argon ion laser. The $TiH_x/KClO_4$ system in argon gas has three distinct pressure regimes. The thresholds are very high below about 0.3 MPa, decrease rapidly between 0.3 and 1.5 MPa, and decrease gradually and become constant between 1.5 and 7 MPa. Mg/Teflon and the Al-torch mix also have decreasing thresholds with increasing gas pressures and decreasing pellet densities. Ignition pulse lengths between 0.25 and 20 milliseconds at a spot diameter of 65 microns in the $TiH_x/KClO_4$ system indicate that the thresholds are predominantly power, not energy, thresholds for pulses longer than one millisecond. The increase in threshold power below one millisecond is consistent with a calculated time to reach thermal equilibrium of 0.96 milliseconds. The effect of beam diameters between 40 and 175 microns on the $TiH_x/KClO_4$ thresholds is consistent with ignitions occurring at approximately the same temperature at the center of the beam. $TiH_x/KClO_4$ and $Zr/KClO_4$/Viton/Graphite pyrotechnics are also ignited in sealed devices through sapphire windows. Ignition thresholds are comparable to those on open surfaces in pressures of 0.1 to 0.5 MPa which suggests that samples first decompose and ignite under a pocket of pressure. Ignition failures in $TiH_x/KClO_4$ and in low density $Zr/KClO_4$/Viton/Graphite samples are attributed to increased porosity and/or gaps near the windows which allow reaction products to escape from the ignition zone.

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1. INTRODUCTION

The laser is proving to be a very useful energy source for studying the ignition of propellants, explosives, and pyrotechnics. The power, energy, time duration, and spot size of the laser beam on the sample surface can be readily varied and precisely and reproducibly measured. Ignition properties can unequivocally be related to material properties when such a precise ignition source is used.

One of the most important parameters in the ignition of many energetic materials is the gas pressure at the ignition zone. Significant pressure effects have been observed in the ignitions of the explosive HMX and pyrotechnic Holex No. 14 (Zr fuel) sealed in glass tubes under pressure[1], in the propellants TRX, N-S, PPC, and ANP[2], in the propellant ammonium perchlorate at pressures above[3] and below[4,5,6] atmospheric pressure, in the CO_2 laser ignition of the explosive RDX, HMX, PETN, Tetryl, HNS, and TNT[7], and in the argon laser initiation of $TiH_x/KClO_4$ pyrotechnics.[8] Most of these experiments heat the bulk sample in a molten bath[1] or furnace[3] or the whole sample surface with a rastered laser beam[4,5,6] and measure times to ignition of several hundred milliseconds to seconds as a function of pressure and heat input. Much more work has been done on pressure effects in the decomposition of energetic materials[9,10,11] than on ignition, but the decomposition process must be related to ignition.

It was previously observed qualitatively in the $TiH_x/KClO_4$ system that ignition times and thresholds decreased as the gas pressure was increased.[8] Those experiments have been extended to obtain quantitative measurements of the ignition thresholds as a function of pressure. $TiH_x/KClO_4$ and a $Zr/KClO_4$/Viton/Graphite pyrotechnic have also been laser initiated in windowed, sealed charge holders to measure ignition conditions under a more device-type configuration.

Another type of pressure effect on ignition is the pressing pressure used in forming a pellet. Changes in ignition thresholds for hot wire[12,13] and laser ignition[12,14,15] have been reported. We have observed significant pellet density effects in the ignition of free standing pellets of Mg/Teflon and the Al-torch mix and in the powders pressed in the sealed, windowed devices.

Many ignition effects dependent upon the gas pressure, sample density, power, spot size, and pulse width have been measured in the experiments reported here. Although a particular effect may have been studied in only one pyrotechnic, they should have a general applicability to most propellant, explosives, and pyrotechnics that have gas phase reactions.

2. EXPERIMENTAL

A simplified schematic of the laser initiation system is shown in Fig. 1. It has previously been described in detail.[8] The 514.5 nm line of the argon ion laser was used for all of the results reported here. The output beam of this laser has a Gaussian profile defined by Eqn. 1.

$$I(r) = \frac{2P}{\pi r_0^2} e^{-2r^2/r_0^2} , \qquad (1)$$

where I(r) is the radially intensity distribution, P the total beam power, and r_0 the Gaussian beam radius defined at the point where the intensity is e^{-2} of the center intensity.

The cw output of the laser is converted to an ignition pulse by the chopper-shutter combination and focused to a small spot on the sample by a lens. Ignition is monitored by the two filter-photodiode combinations with the interference filter passing only reflected laser light and the 560 nm filter passing the incandescence from ignition and burning. Various spots with Gaussian diameters between 40 and 175 microns were achieved with different focal length lenses and a beam expander before the focusing lenses. The focused diameters were measured by the Ronchi ruling technique[16] as previously described.[8]

For ignition experiments on the open surfaces of pellets, the sample chamber was first evacuated and then filled with argon, helium, or nitrogen. The chamber can be pressurized to 6.9 MPa.

The configuration used for ignition of powders in sealed charge holders is shown in Fig. 2. The charge holder consists of a stainless steel shell, a 3 mm diameter x 1 mm thick sapphire window, and a laser-welded closure disk. It is clamped into a holding block which is mounted in the sample chamber. In all charge holders, the closure disks sheared at their clamp points, and the sapphire window stayed intact after completion of the powder burn.

3. RESULTS AND DISCUSSION

3.1 OPEN SURFACE IGNITION OF $TiH_x/KClO_4$ UNDER PRESSURE

3.1.1 Pressure Effects on Ignition Thresholds

Samples of $TiH_x/KClO_4$ (x = 0.2, 0.65, and 1.65) with 33/67 fuel/oxidizer mass ratios were pressed into ceramic charge holders with an inner diameter of 4.27 mm and a length of 3.68 mm in two increments at a pressing pressure of 72 MPa. Resulting sample densities were 1.9, 2.1, and 2.0 gm/cm^3 for x = 0.2, 0.65, and 1.65, respectively.

Figure 3 shows typical data generated during an ignition experiment. The intensity of the reflected laser light begins to decrease within several microseconds after application of the pulse and milliseconds before ignition. This is an indication that there are chemical reactions occurring long before incandescence from ignition is detected.

The ignition thresholds of the three compositions were measured between 0.2 and 6.2 MPa in argon for 65 micron diameter, 3 millisecond pulses. The thresholds for $TiH_{1.65}/KClO_4$ were also measured in helium and nitrogen at two pressures for comparison. These threshold results are plotted in Fig. 4. A data point in Fig. 4 is typically the average from three ignitions whose threshold power was measured by approaching the threshold in 5 to 10 milliwatt increments. A fresh spot on the sample surface was used if no ignition occurred after a laser pulse. The typical spread in threshold values is approximately the size of the symbols in Fig. 4. The thresholds are plotted as power in the 3 millisecond pulses rather than energy because it was found that the thresholds are predominantly power dependent under these ignition conditions. There is an abrupt increase in the thresholds below about 0.3 MPa to a value larger than that available from our laser. The thresholds drop rapidly betweeen 0.3 and 1.5 MPa, and for x = 0.65 and 1.65 there is a further gradual decrease up to 3.5 MPa.

The decrease in thresholds with increasing pressure is similar to the effect of decreasing ignition times with pressure for a fixed ignition stimulus that have been observed in explosives[1] and propellants.[4,5,6] The pressure dependence of the ignition thresholds is also similar to the pressure dependence of the decomposition of many other energetic materials. Oyumi and Brill[10] defined three pressure zones − < 0.28 MPa, 0.14 to 1.4 MPa, and > 1.4 MPa − where distinct changes in decomposition products occur. These are the same pressure zones where distinct changes in the ignition thresholds of $TiH_x/KClO_4$ occur.

For $TiH_{1.65}/KClO_4$, the thresholds are slightly higher in nitrogen and much higher in helium compared to argon. The same dependence on these gases was found in ammonium perchlorate at subatmospheric pressures.[4]

The pressure-dependent burn rates of $TiH_x/KClO_4$ are reproduced in Fig. 5.[17] There is some correlation between the burn rates and ignition thresholds. $TiH_2/KClO_4$ has the highest burn rate and lowest thresholds below 1.7 MPa. The thresholds decrease and burn rates increase with pressure, but the burn rates are pressure dependent to much higher pressures than the ignition thresholds.

3.1.2 Pulse Width Effects

Pulse lengths were varied between 1 and 20 milliseconds for $TiH_2/KClO_4$ in 2.9 and 0.62 MPa of argon. Figure 6 shows the laser pulses and ignition incandescence for 1 and 3 milliseconds at 2.9 MPa and 10 milliseconds at 0.62 MPa. The indicated threshold power levels are all approximately the same for these three pulses. The ignition after about 1.5 milliseconds into the 3 millisecond pulse is typical of the timing in most samples at the higher pressures used. At the lower pressures, the ignitions can occur further into the longer pulses as illustrated by the lower two traces in Fig. 6. However, the applied power has to be right at the threshold; small increases in power cause large decreases in ignition times. Similar ignitions at longer times at the higher pressures probably would also occur, but it is difficult to adjust the applied power in small enough increments in this pressure regime where the thresholds are much lower. The ignitions occurred after the 1 millisecond and 10 millisecond pulses in Fig. 6 which seems to imply that once a decomposition has been started, the reactions between these products and/or between them and the powder will sustain the ignition.

Pulse lengths between 0.25 and 3 milliseconds were investigated in $TiH_{1.65}/KClO_4$ in 2.86 MPa of nitrogen and argon. The thresholds for this composition and the $TiH_2/KClO_4$ composition are summarized in Fig. 7. Note the slightly higher threshold in nitrogen over argon at the 1 millisecond pulse width. The trend is a gradual decrease in the threshold for pulses between 1 and 20 milliseconds and for a sharp increase in the threshold for pulses lengths shorter than 1 millisecond. Thus, the thresholds are predominantly power dependent for pulse lengths longer than 1 millisecond with a crossover to more energy dependence at pulse lengths shorter than 1 millisecond for the 65 micron diameter spots. These pulse width effects are the justification for plotting ignition thresholds as power thresholds in Fig. 4. A similar crossover between power and energy thresholds has been measured in $Mg/NaNO_3$.[7]

3.1.3 Spot Size Effects

The effect of Gaussian beam diameters between 40 and 175 microns on the threshold power and central power density for $TiH_{1.65}/KClO_4$ is plotted in Fig. 8. Larger powers are required with larger spot sizes, but the central power densities can be less at larger diameters. The central power densities increase rapidly at the smaller spot sizes and decrease more slowly at the larger spot sizes. Similar decreases in ignition power density with larger spot sizes have been observed for CO_2 laser ignition with a flat top beam profile[7] and in hot wire ignition with various wire diameters.[12,13]

3.2 OPEN SURFACES OF Mg/TEFLON AND Al-TORCH MIX UNDER PRESSURE

The laser ignitions of free standing pellets of Mg/Teflon and an Al-torch mix were also measured under pressure. Since consolidated pellets of these materials can be formed and the pellet density should remain constant, the effect of pellet pressing pressure and chamber gas pressure could both be determined. Table 1 lists the results of these measurements. The pellets with the lower pressing pressure have the lower ignition thresholds for a fixed gas pressure. For a fixed pellet density, increasing the gas pressure also decreases the ignition thresholds for these materials.

Table 1

IGNITION THRESHOLDS OF Mg/TEFLON AND Al-TORCH MIX

514.5 nm, 65 micron diameter, 20 millisecond pulses in nitrogen

Sample	Pressing Pressure (MPa)	Gas Pressure (MPa)	Threshold (Watts)
Mg/Teflon	34.5	1.55	0.29
	82.7	1.55	>1.6
	82.7	5.64	1.2
Al-torch	6.9	1.52	1.1
	82.7	1.52	>1.6
	6.9	5.67	0.38
	82.7	5.67	0.75

3.3 $TiH_x/KClO_4$ AND Zr/$KClO_4$/VITON/GRAPHITE IN SEALED DEVICES

3.3.1 Typical Ignitions

$TiH_2/KClO_4$, $TiH_{1.65}/KClO_4$, and Zr/$KClO_4$/Viton/Graphite (38/56/5/1 mass ratio) pyrotechnics were pressed at 69 MPa into the windowed charge holders of Fig. 2. Typical ignition and burning incandescence is shown in Fig. 9 for $TiH_2/KClO_4$ for ignition by 20 millisecond pulses; data for the other two pyrotechnics is similar. A major difference in the incandescence from the sealed devices and the open pellets is the time duration of the incandescence; it is on the order of a few hundred microseconds in the windowed devices compared to many milliseconds in the open pellets. This time effect is due to the predominance of a convective burning mechanism in the sealed systems and a conductive burning mechanism in the open pellets.[17]

The ignition thresholds in the windowed devices were also found to be predominantly power thresholds for the pulse lengths between 2 and 20 milliseconds. Most samples ignited within about 2 milliseconds. The power had to be right at threshold (within about 2-3 milliwatts) for the ignition to occur at longer times into the laser pulse. The middle trace in Fig. 9 is an example of ignition after about 10 milliseconds, and the upper trace shows how a small increase in power can greatly reduce the ignition time and the ignition energy.

Measurements of the ignition thresholds in the windowed devices are summarized in Table 2 for Gaussian beam diameters of 60 and 125 microns. Spot size effects similar to those on open surfaces are also observed; larger spot sizes require more power but less power density.

Table 2

THRESHOLD POWERS IN SEALED, WINDOWED DEVICES

514.5 nm, 20 millisecond pulses

Sample	Beam Diameter (microns)	Threshold (Watts)	Central Power Density (KW/cm^2)
$Zr/KClO_4/$ Viton/Graphite	60	0.20	14.1
	125	0.47	7.7
$TiH_2/KClO_4$	60	0.40	28.2
	125	0.72	11.7
$TiH_{1.65}/KClO_4$	60	0.57	40.3
	125	1.16	18.9

3.3.2 Pellet Density Effects in Windowed Devices

A comparison between ignitions at pellet densities of 2.4 and 2.1 gm/cm^3 for the Zr-fuel pyrotechnic is shown in Fig. 10. The threshold power and ignition times are increased considerably in the lower density samples. Several incandescence spikes from quenched ignitions occur before sustained ignition.

Ignition failures have been observed in $TiH_{1.65}/KClO_4$ powders which may be attributed to a powder relaxation with a density decrease and/or gaps that developed between the window and powder surface. Figure 11 shows the pellet surface through the window with two positions of failed ignition. It can be seen that reaction products were driven away from the ignition points into a gap between the window that was preexisting or created by the pressure generated during decomposition. This sample had incandescence spikes and quenches similar to those in the upper trace of Fig. 10. The lack of enough confinement at the ignition point caused quenching of the ignition.

A decrease in ignition sensitivity with decreasing sample density has also been observed in laser ignition[14,15] and hot wire ignition[12] of explosives.

3.4 IGNITION PROCESSES

These experiments on the open surfaces under pressure and in confined powders at the window may be compared to infer process that are occurring during ignition. The change in the intensity of the reflected laser light shown in Fig. 3 and the visually observed darkening of the sample implies that the powder's chemical composition, optical constants, and thermal properties are probably changing long before ignition occurs.

The temperature under the focused beam must reach a minimum value to start the ignition process although the ignition temperature may decrease with increasing pressure.[3] The temperature rise produced by a Gaussian beam has been calculated.[18,19,20] If the optical and thermal properties of a material can be considered constant (which may be a valid approximation after the sample "darkening" has reached completion), Lax[18] has shown that the temperature distribution T in the sample with attenuation constant α can be written as

$$T = T_{max} N(R, Z, W), \qquad (2)$$

where $R = r/r_o$, $Z = z/r_o$, $W = \alpha r_o$, and z is the coordinate normal to the surface. T_{max} is the temperature that is generated at the beam center (R =0) on the surface (Z = 0) for large attenuation ($W \rightarrow \infty$). N(R,Z,W) is a complex function that must be evaluated numerically, but Lax shows that

$$\lim_{W \rightarrow \infty} N(0,0,W) = 1. \qquad (3)$$

N is less than one for other values of R, Z, and W. Lax further shows that

$$T_{max} = \frac{P}{\sqrt{2\pi} K r_o}, \qquad (4)$$

where K is the sample thermal conductivity, P the laser power, and r_o the Gaussian radius.

Equations 2 through 4 explain the spot size and pressing density dependences observed in these ignition experiments. If a fixed temperature is required to begin the ignition process, the power should increase linearly with beam radius for infinite W. For finite W, T_{max} is weighted by N. Calculated values of N[18] show that N tends to become smaller as r_o decreases. Thus, the trend of threshold power with spot size shown in Fig. 8 is the trend that would be expected if ignition started at approximately the same temperature at the center of the Gaussian beams of different diameters.

Donaldson[21] and Pierce and Leith[22] have used values of 0.9 and 0.8 W/m-°K for the thermal conductivity in the modeling of the $TiH_x/KClO_4$ systems. If we use 0.9 and assume that 70% of the incident laser power is absorbed by the pellets, Eqn. 4 gives values of T_{max} between 810 and 1,240 °K for the data shown in Fig. 8. These temperatures would be reduced by N(0,0,W) and the cooling effects of the high pressure gas. We do not calculate N(0,0,W) because of our present lack of an estimate for α. These calculated values of T_{max} do give a somewhat reasonable estimate for an upper bound on the ignition temperature.

Lax[19] also defines the time required to reach thermal equilibrium as

$$T_{equil} = \frac{C r_o^2}{2K}, \qquad (5)$$

where C is the specific heat. Using C = 1.63 x 10^6 J/m^3-°K and K = 0.9 W/m-°K,[21] Eqn. 5 yields a T_{equil} of 0.96 milliseconds for a Gaussian diameter of 65 microns. Thus, if the pellet is considered as a semi-infinite cylinder with respect to the size of the laser beam, pulse lengths longer than about 1 millisecond would not increase the temperature at the center of the beam. One millisecond is precisely where there is an abrupt increase in threshold power for the data plotted in Fig. 7. The gradually decrease in threshold powers for pulses longer than 1 millisecond may be attributed to deviations from the semi-infinite cylinder approximation.

However, heating an energetic material to a certain temperature only begins one step in the ignition process. The temperature produces decomposition products, and these products must be confined at the ignition point for the ignition to become self-sustaining. For open surfaces, the gas pressure provides the confinement. A fixed gas pressure will also fix any pressure-dependent gas phase reaction kinetics. Therefore, with open surface ignition at a fixed pressure the ignition thresholds should be expected to decrease as the sample density decreases. A lower density sample will have a lower thermal conductivity, and less power is needed to reach the same temperature as defined by Eqn. 4 and measured in Table 1.

For sealed devices that are ignited by a laser or a hot wire, the powder and header must provide the confinement at the ignition point. The incandescence spikes in the low density sample of Fig. 10 and the dispersed products in Fig. 11 can be interpreted as reaction products forming but being dispersed from the ignition point through pores or gaps. The low density incandescence trace in Fig. 10 is probably the result of the ignition process starting, products diffusing away but increasing the pressure in the device, heat flowing away from the inert solid reaction products at the laser beam and igniting surrounding material, and repeating the process until the pressure is high enough to lower the ignition threshold to the applied power. Munger and Woods[23] has also been able to quench ignitions in hot wire devices with pressure vents under the bridgewire. To prevent ignition quenches, the pellet porosity must be low enough so that the decomposition products can form a pocket of pressure at the ignition point, confine the reactions, and sustain ignition. There should be an optimum powder density for minimum ignition thresholds in sealed devices; there will probably be a trade-off between increasing the density to provide better confinement and decreasing the density to create a higher temperature for a given power.

The necessity of confinement also implies that there are chemical reactions occurring in the gas phase among the decomposition products that are probably pressure dependent.[3,9,10,11] The reactions between them or between them and the condensed phase near the ignition point is a critical factor in determining whether the ignition will be self-sustaining. The observation of no incandescence after input power has stopped and before ignition incandescence also implies that creation and confinement of the decomposition products start a chemical reaction that evolves into ignition. Ignitions have even been observed 3.5 milliseconds after application of 15 nanosecond laser pulses.[15]

4. CONCLUSIONS

The use of lasers as ignition sources provides the advantages of being able to precisely measure and adjust many ignition parameters. The importance of gas pressure, sample density, spot size, pulse length, and confinement on ignition has been shown. A comparison of our results on different materials and the results of others suggests that the observed effects should have general applicability to most propellants, explosives, and pyrotechnics, particularly those with gas phase or gas-condensed phase reactions involving decomposition products.

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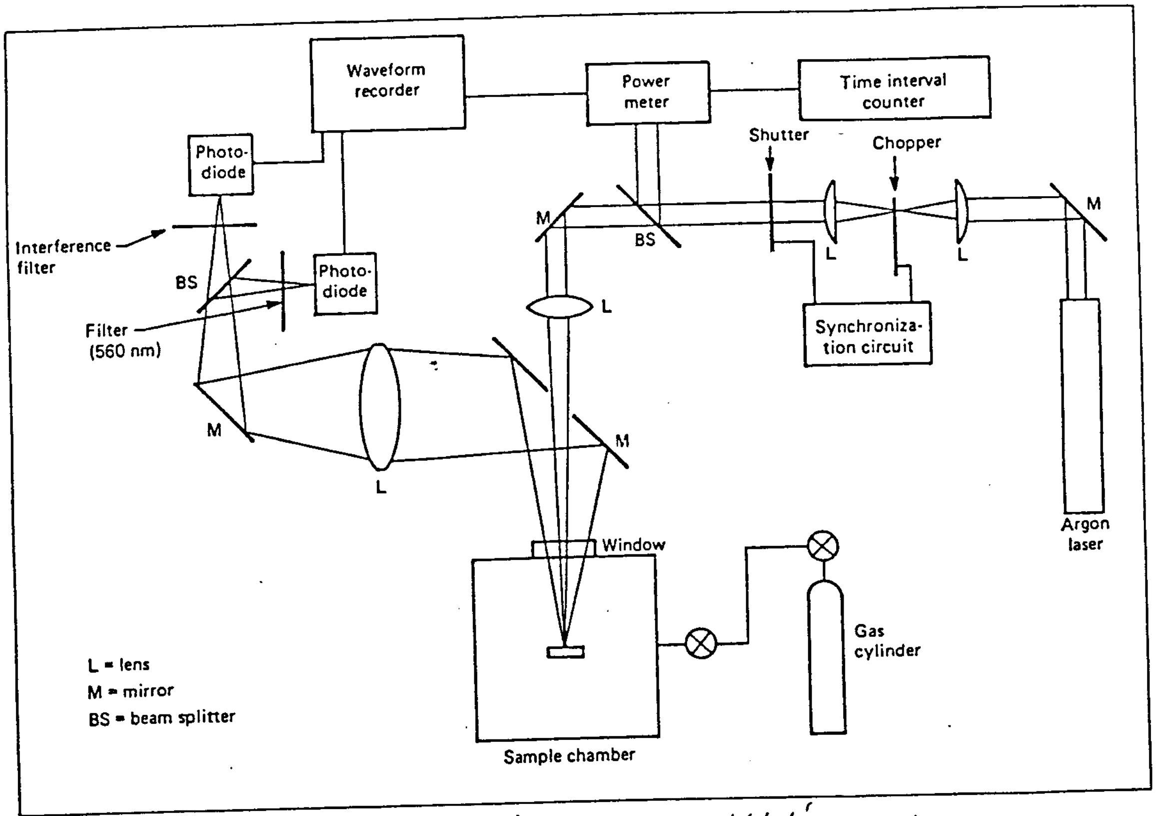

Figure 1. Simplified schematic of laser initiation system.

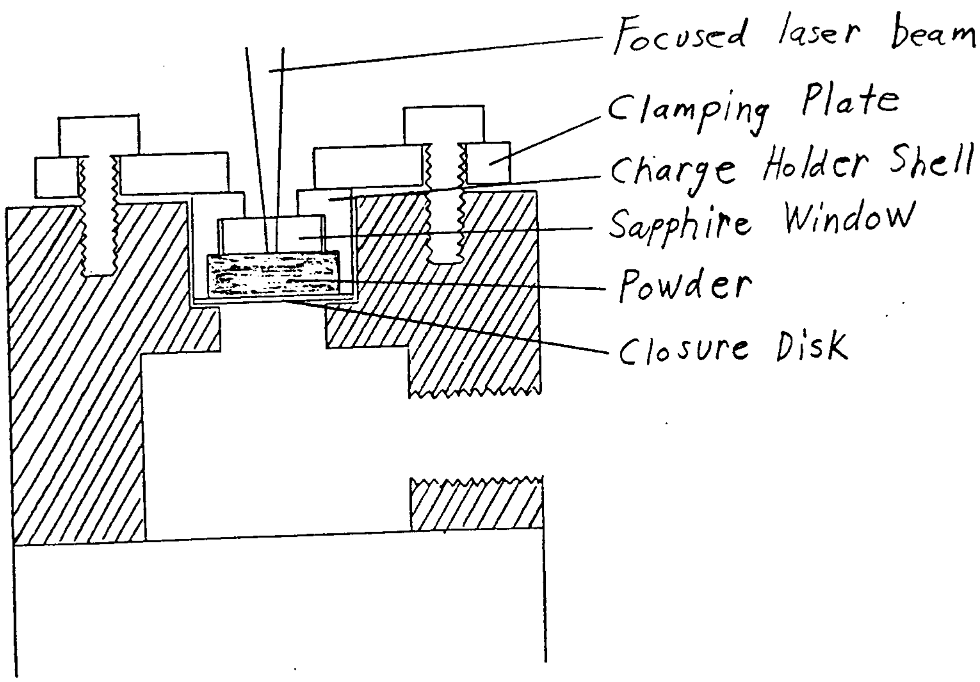

Figure 2. Details of the windowed charge holder and its mounting in the sample chamber.

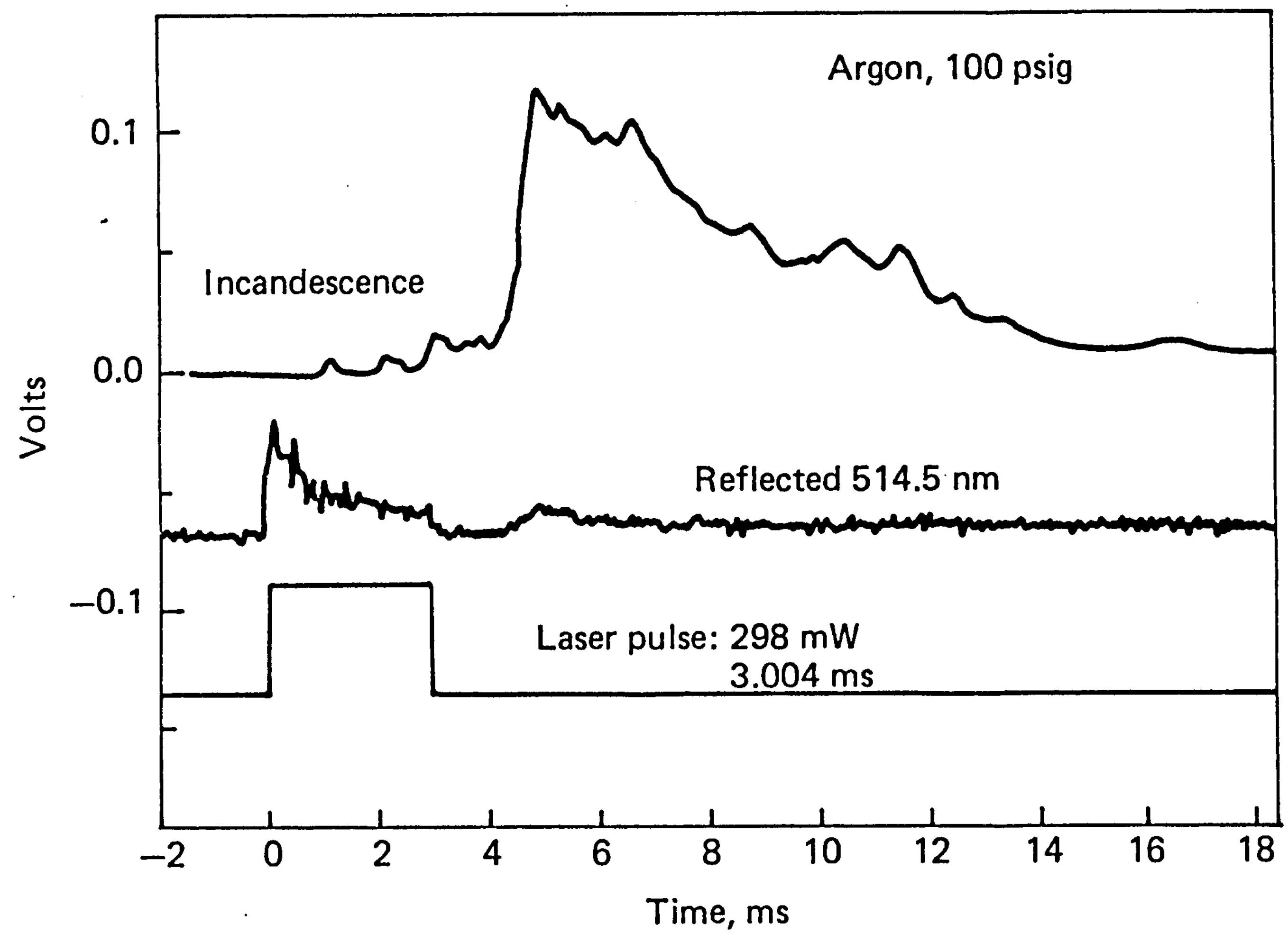

Figure 3. Typical waveforms generated in the system of Fig. 1 during laser ignition of $TiH_{.65}/KClO_4$ in 0.79 MPa of argon.

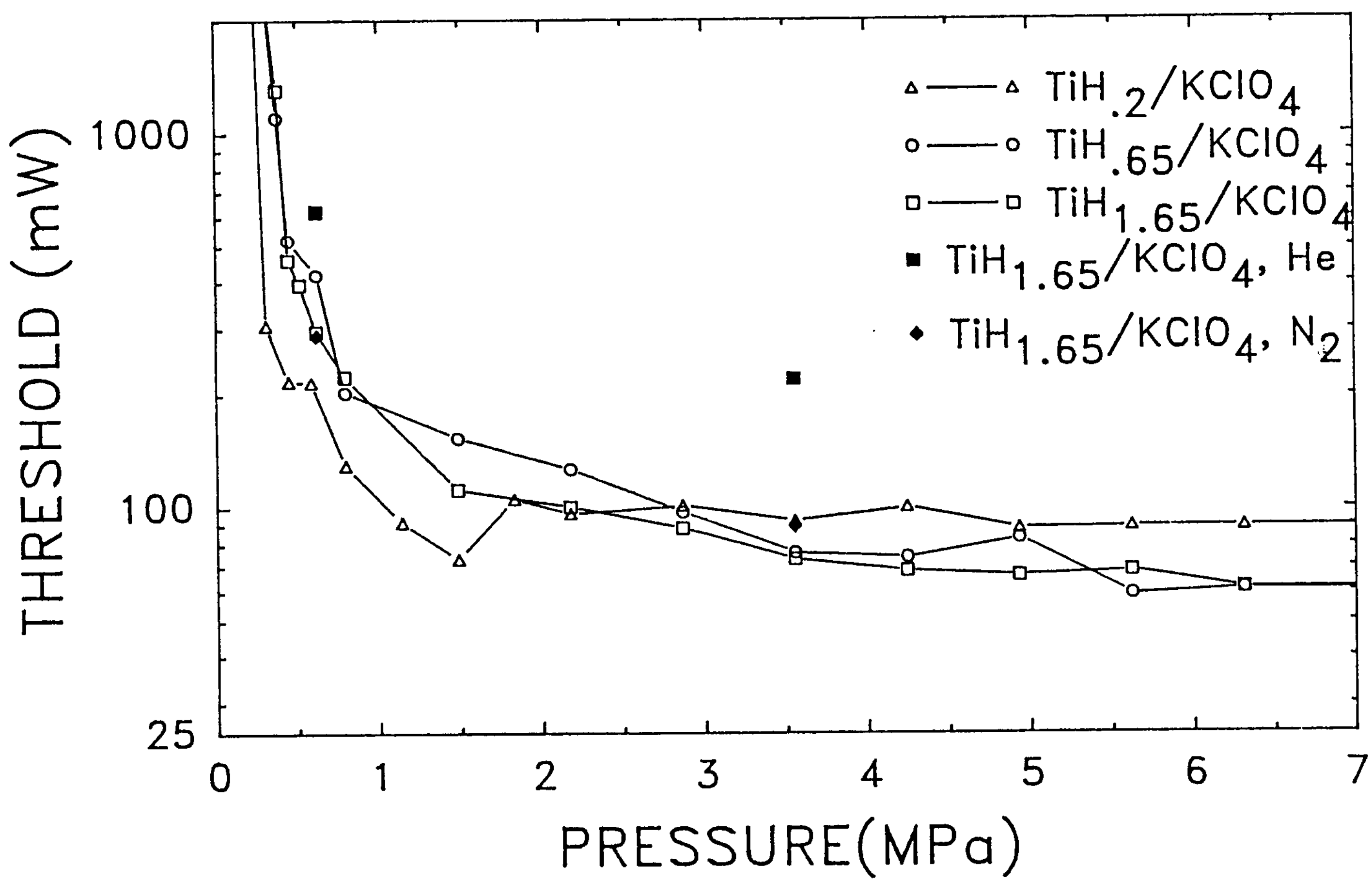

Figure 4. Ignition thresholds of $TiH_x/KClO_4$ as a function of pressure in argon. Thresholds for $TiH_{1.65}/KClO_4$ at 0.62 and 2.86 MPa in helium and nitrogen are also plotted. Laser pulse length is 3 milliseconds.

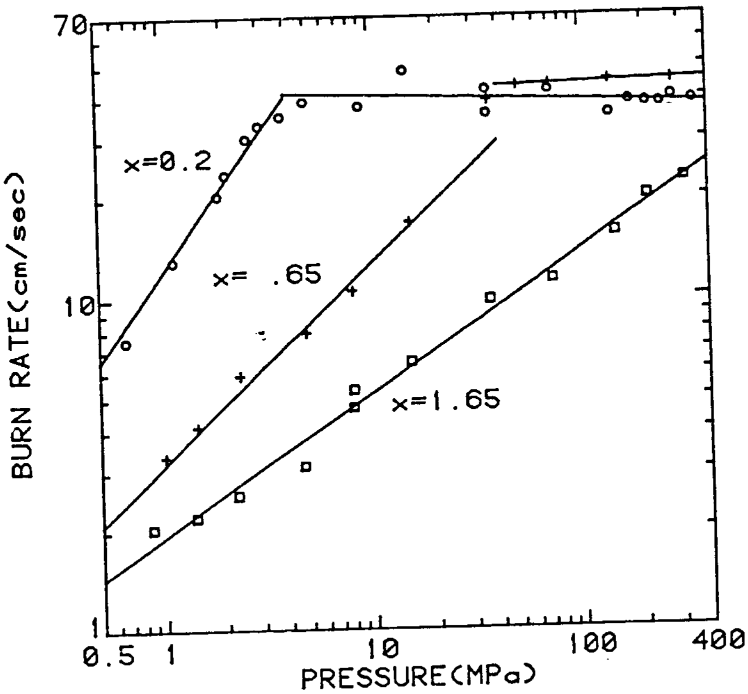

Figure 5. Burn rates of $TiH_x/KClO_4$ in nitrogen.[17]

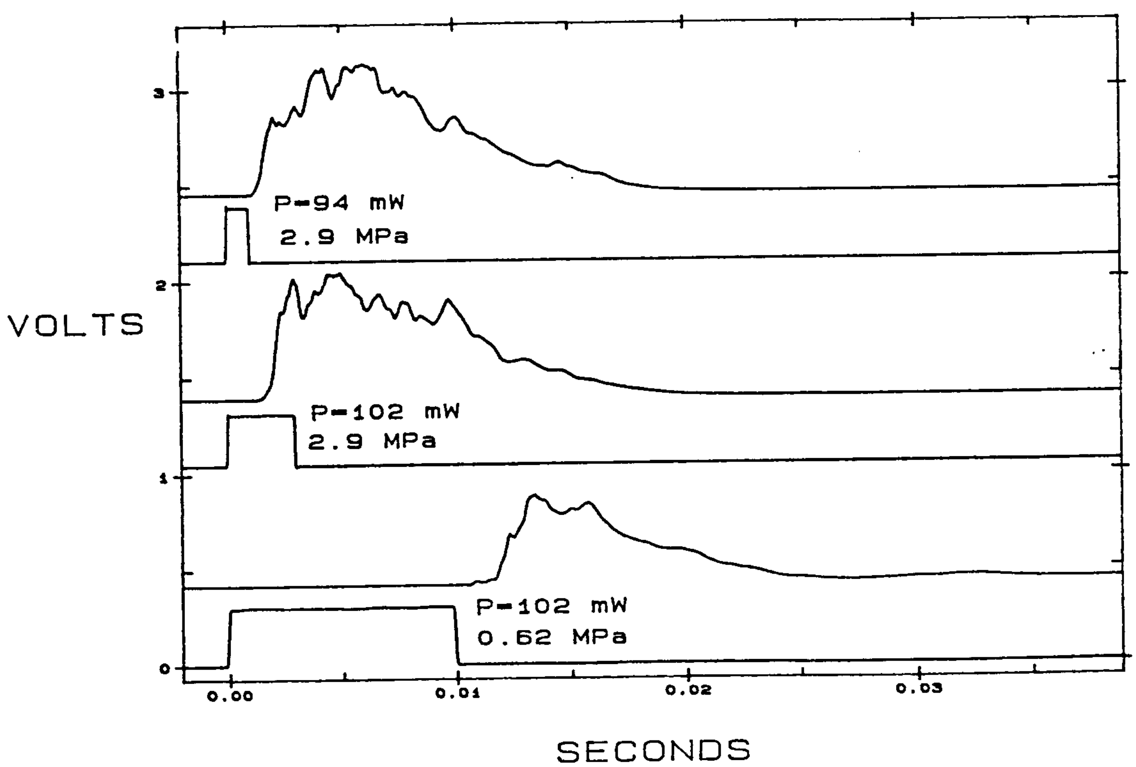

Figure 6. The effect of pulse width on ignition of $TiH_{.2}/KClO_4$ in 2.9 and 0.62 MPa of argon. P= threshold power. The traces are the laser pulses and incandescence.

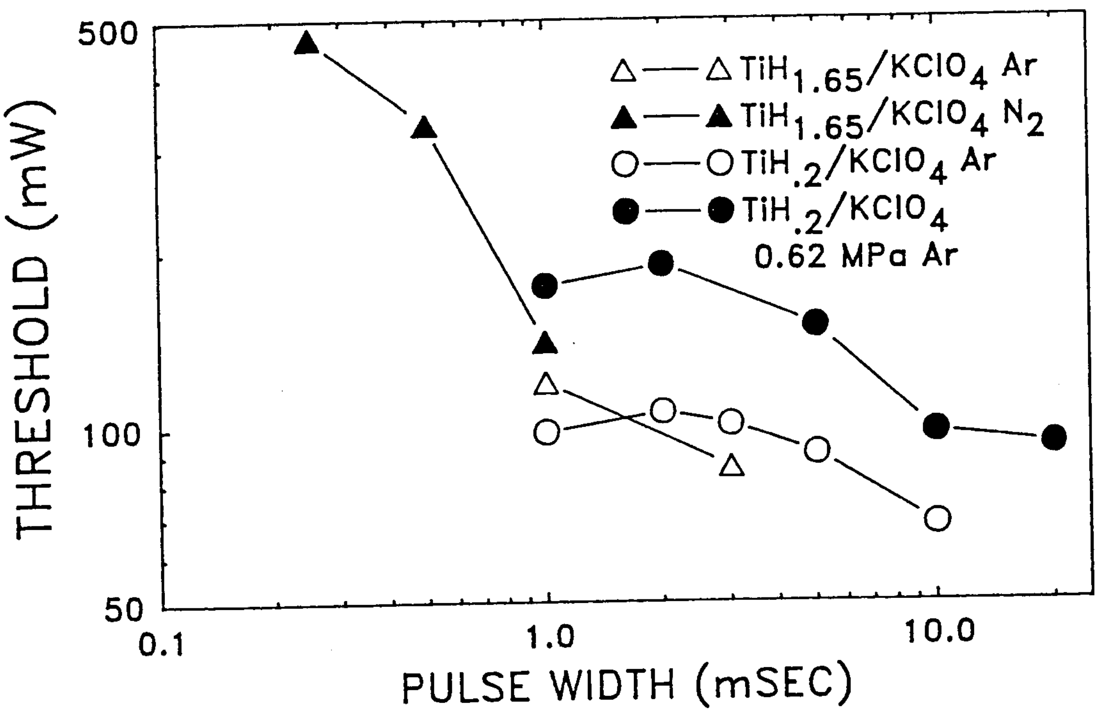

Figure 7. The effect of pulse width on the threshold power. All thresholds were measured at 2.86 MPa except the one $TiH_{.2}/KClO_4$ set at 0.62 MPa of argon.

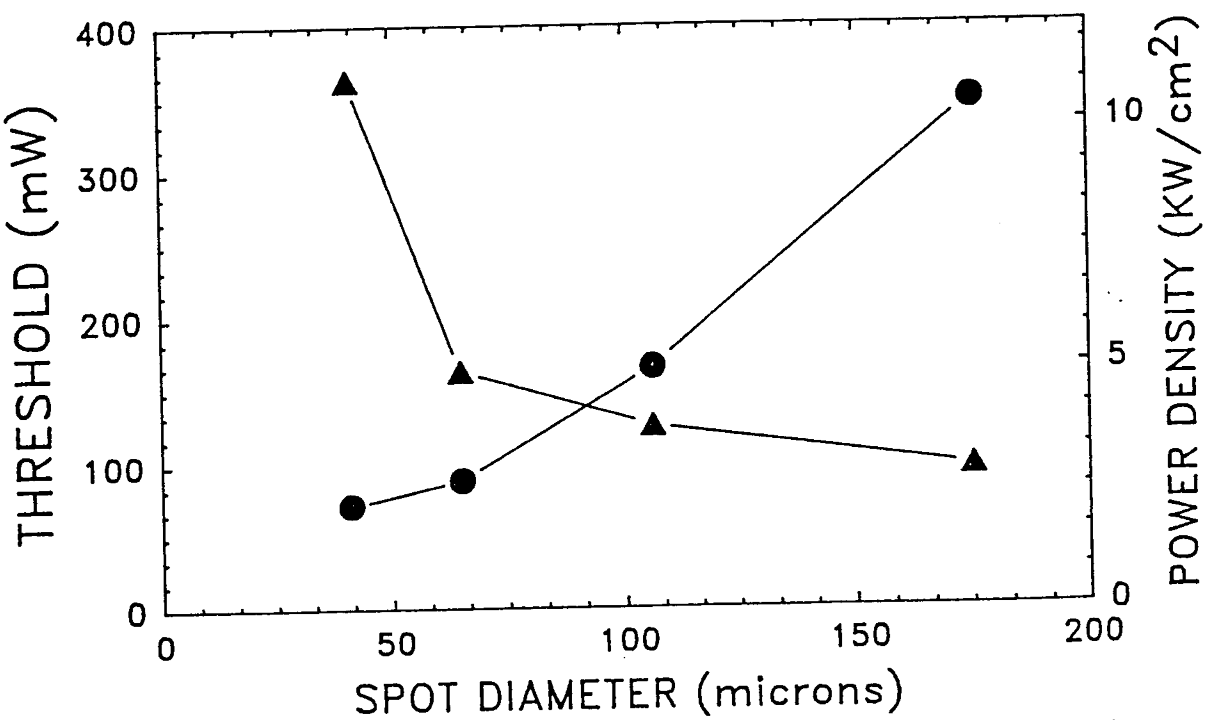

Figure 8. The effect of the focused Gaussian beam diameter on the threshold power and central power density for ignition of $TiH_{1.65}/KClO_4$ in 2.86 MPa of argon.

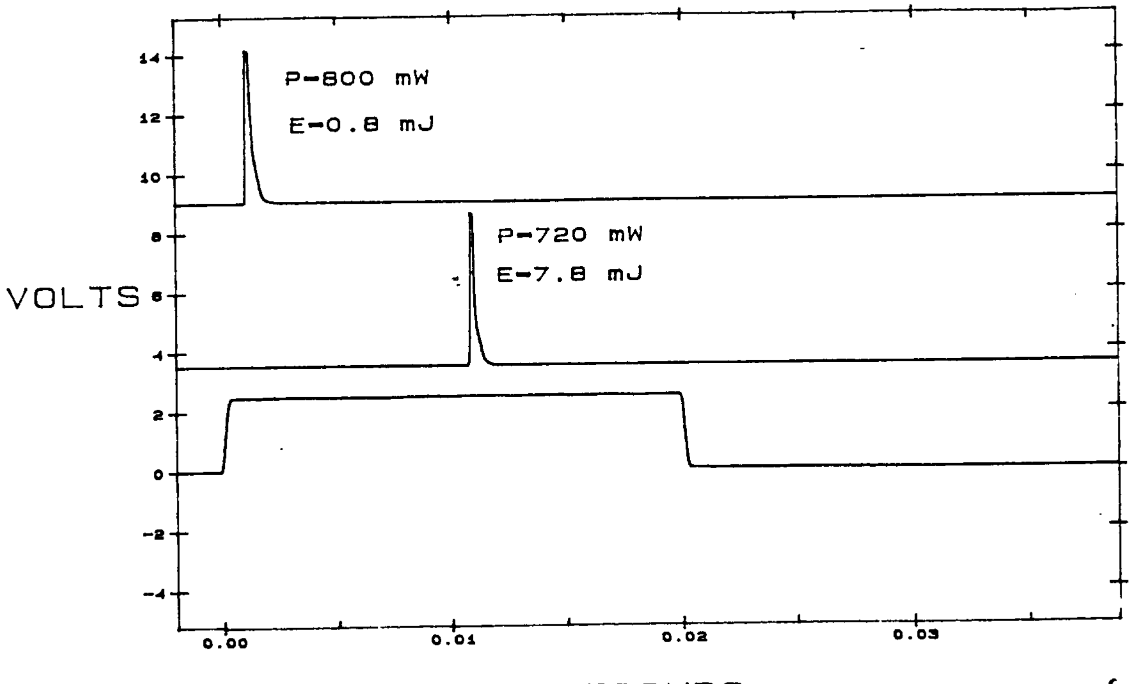

Figure 9. Typical incandescence traces for ignition of $TiH_2/KClO_4$ in the sealed device of Fig. 2. Lower trace is 20 millisecond, 125 micron diameter laser pulse. P = laser power, E = applied energy up to ignition.

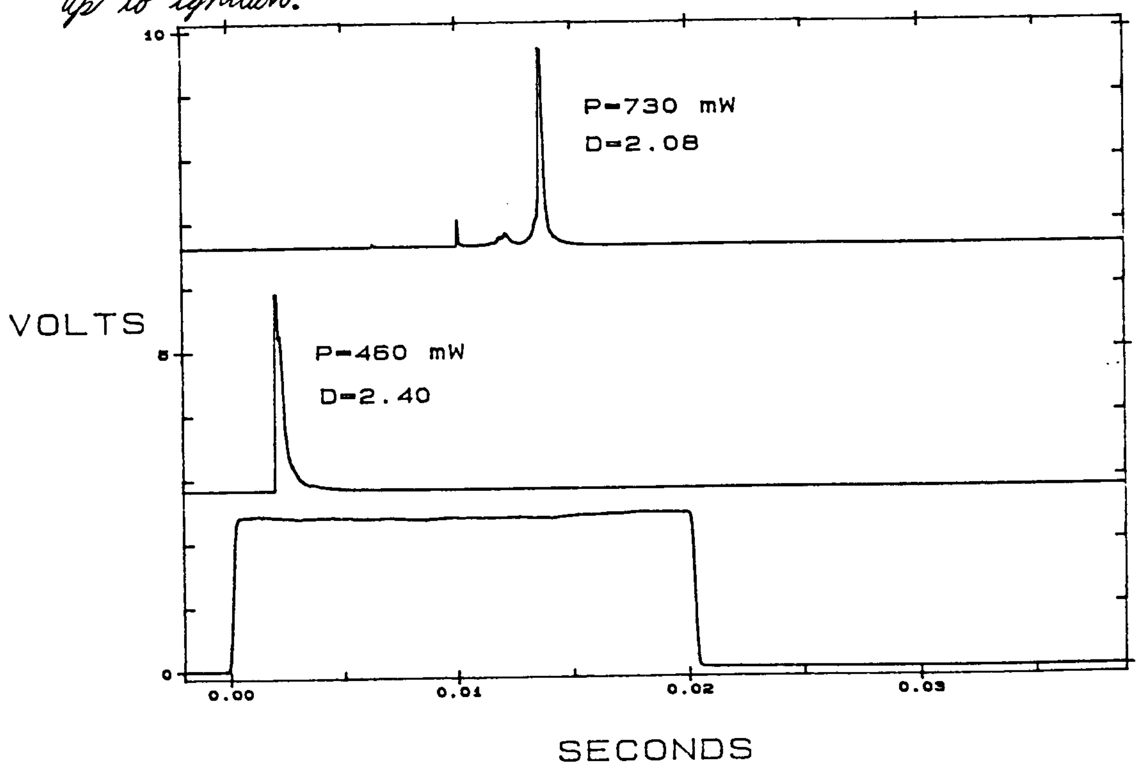

Figure 10. Ignitions near threshold of the $Zr/KClO_4$/Viton/Graphite pyrotechnic at densities D of 2.08 and 2.40 gm/cm^3 in the sealed device of Fig. 2.

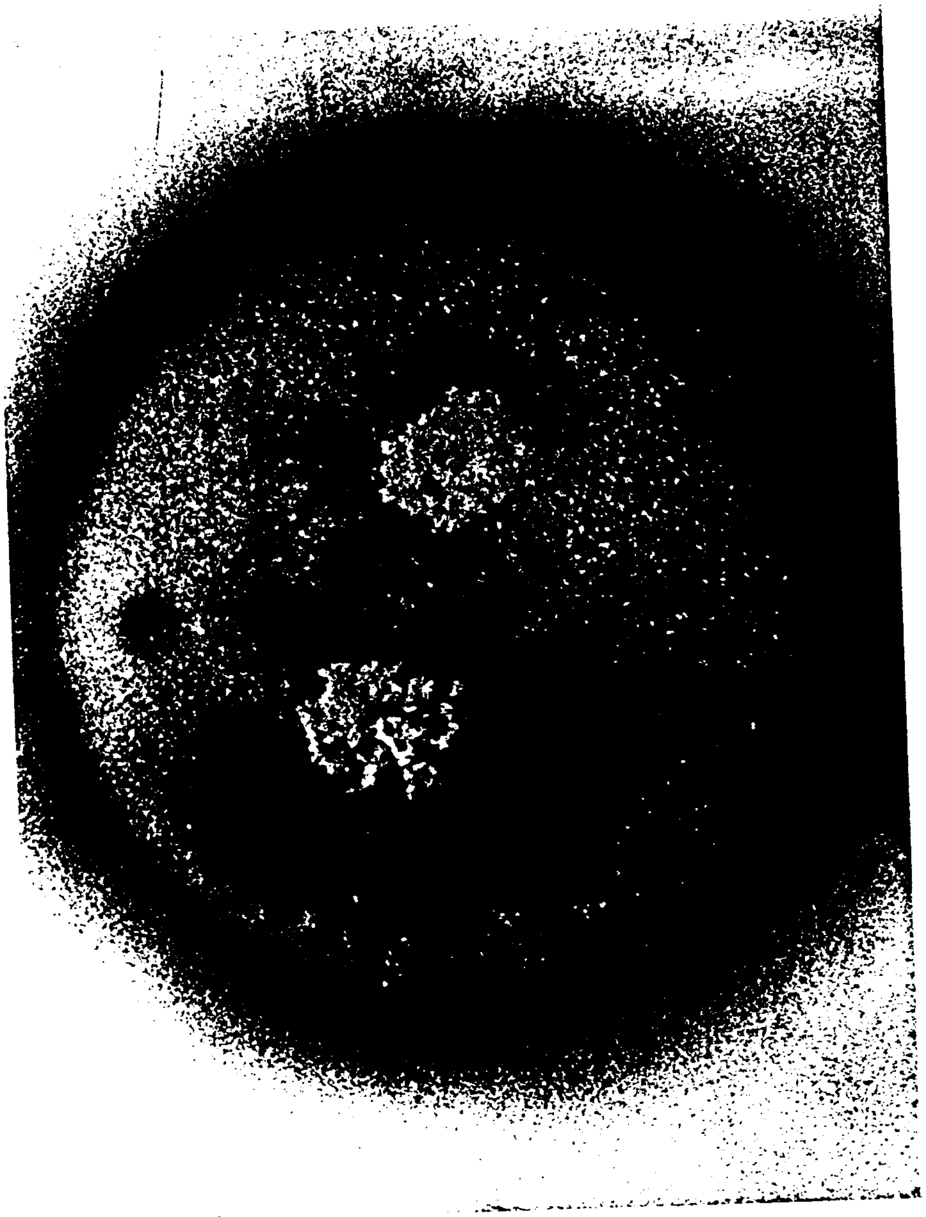

Figure 11. Ignition failures at two points of $TiH_{1.65}/KClO_4$ in the windowed device of Fig. 2. View is of the powder surface at the interior window surface.