Research paper

Pulse laser ablation characteristics of quartz diffusion plate and initiation of PETN
— Glass fragment size distribution and surface roughness dependence —

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Abstract
This paper describes an investigation of laser-induced initiation of PETN powder based on the pulse laser ablation of ground glass surface previously reported by our group. In this study, we have chosen diffusion plates made of quartz as a ground glass specimen with several different surface roughnesses instead of commercial ground glass adopted in the previous study. This is due to the fact that laser ablation characteristics usually depends on the material.

We have found that cloud of glass fragments is generated, when ground glass surface is laser ablated through the glass plate focused from rear side. In this study, we found further properties of this striking ablation phenomena; for example, (i) size distribution of produced glass particles have almost no dependence on the initial surface roughness, (ii) generated number of particles by ablation increases with increasing surface roughness, (iii) particle generation continues more than 10 μs after laser ablation, etc.

A series of initiation experiments of a small amount of PETN by pulse laser ablation of ground glass with different surface roughness has been performed. We studied the dependence of initiation sensitivity on the laser fluence. It is found that within tested conditions, threshold laser fluence is around 10 J cm^{-2}. For initiation experiments by laser fluence lower than this value, probability of initiation decreases, and initiation delay time after ablation elongates appreciably irrespective of the initial surface roughness, indicating that the initiation is governed almost solely by the magnitude of kinetic energy of generated glass particles.

Keywords: Ground glass, Pulse laser ablation, Ablation threshold, Energetic material, Laser initiation

1. Introduction
Initiation of high explosives by pulse laser has been studied by several authors [1)-6)]. Purpose of these studies is to provide alternative means of initiation of no danger against electrical noise. Laser initiation also has the possibility to give a reliable means of igniting or initiating very small amount of high energetic materials required for the space applications of MEMS (Micro Electro Mechanical System) devices. It is necessary to have a method for handling a very small amount of high energetic materials, since detonation and combustion characteristics changes with decreasing mass and size of materials.

We have been studying enhancement of laser pulse absorption by the intentionally roughened surface [7), 8)]. One of the main target of our study is the application to laser initiation. We found that if one side of a transparent substrate, e.g., PMMA (PolyMethyl Metacrylate) is intentionally roughened with water resistant paper

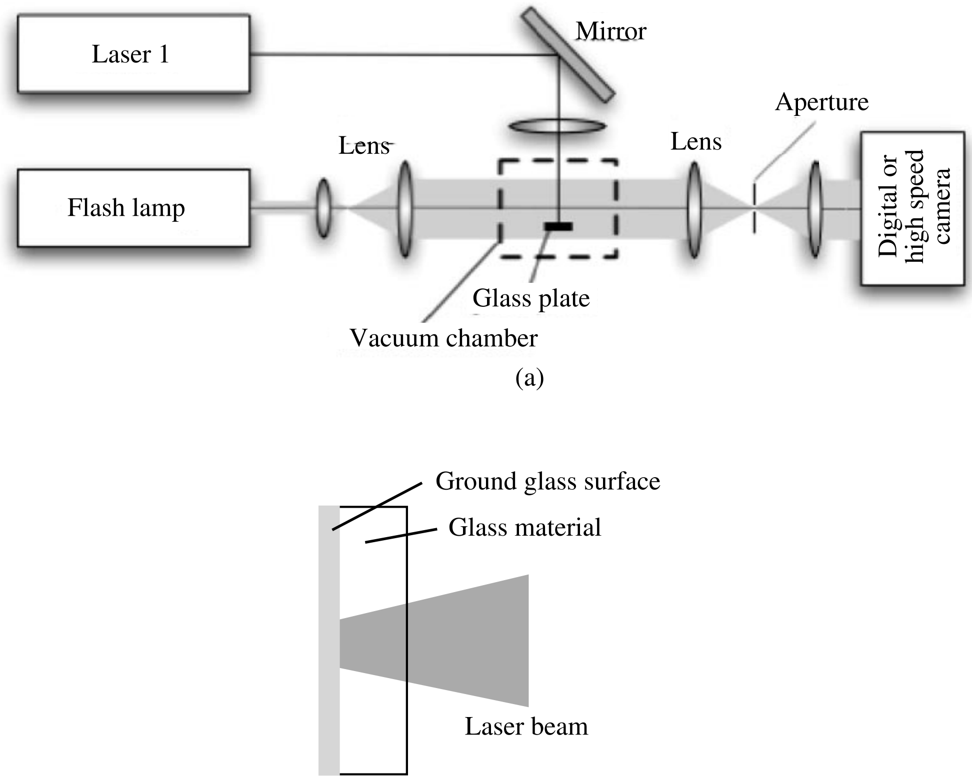


Fig. 1 Optical setup for laser shadowgraph or high speed framing camera observation. Vacuum chamber is installed only for ablation in vacuum experiments.

and then aluminized by vacuum evaporation method, appreciable enhancement of laser energy absorption was recognized [9)-11)]. A very intense air shock wave is produced by ablation [9)]. By utilizing high temperature high pressure state of the ablation plasma, initiation of PETN powder of very small amount has been studied by the authors [9)-11)]. In case of energetic material initiation, thin metal coating on the roughened surface has been made in order to further enhance the laser absorption. However, too thick metal layer leads to the appearance of granular structure behind the region of ablation-induced shock wave. This is explained by the insufficient laser energy to evaporate and ionize thicker layer to form plasma plume.

Recently, we have found that pulse laser ablation of ground glass surface is totally different from those of other rough surfaces [12)]. The phenomenon was successfully applied to the initiation of PETN (Pentaerithritoltetranitrate) powder. The phenomenon apparently depends on various parameters such as laser fluence, laser pulse duration, ambient medium, target material and surface roughness. Summaries of the findings of the laser ablation of ground glass surface include;

(i) Glass fragments have been produced only when pulse laser was focused through the glass material.

(ii) Shock wave precedes the glass particles when ground glass is ablated in air.

(iii) Maximum particle velocity is observed to be 1-1.5 km s^{-1}, although the velocity may depend on the other experimental conditions such as laser fluence.

In this study, further characteristics of pulse laser ablation of ground glass surface has been studied by capturing glass particles in vacuum ablation experiments. A series of experiments on laser initiation has been performed and initiation sensitivity has been measured by varying initial glass surface roughness and laser fluence as a parameter. We have used diffusion plates in initiation experiments, and no metal coating on the glass plates have been made in order to study the characteristics of laser ablation and initiation without influence of metal evaporation by ablation. Therefore, our aim of the present study is to provide scientific data for the present process to apply engineering applications.

2. Experimental

A cloud of glass fragments is produced by pulse laser ablation of ground glass, and motion of these small particles will be affected by the air shock wave preceding particle motion. We therefore observed the phenomena both in air and in vacuum. Experiments in vacuum were planned in order to eliminate the effects of air shock wave. Experiments in vacuum were performed in a small vacuum chamber connected to turbo molecular pump. For laser ablation, we have used an Nd:YAG (Neodium doped Yttorium Aluminum Garnet) laser of fundamental frequency and of 4 ns duration (New Wave Research). Laser energy of about 120 mJ per pulse was used. Laser beam is always focused onto the ground surface through the specimen, since ablation phenomena was found to be dependent on the beam irradiation direction.

In order to demonstrate the feasibility of initiation by the kinetic energy of glass particles, two high-speed photographic observations of the phenomena have been performed. Since we have already known that laser-induced initiation phenomenon is commonly associated with a finite delay time after laser energy deposition, accurate timing of the observation of reaction front cannot be predicted in advance. In order to know the timing of initiation, continuous time-resolved record is inevitable. For this purpose, image converter camera (IMACON 790) of the streak mode is used to record the light emission caused by the reaction of PETN.

Phenomena of both pulse laser ablation and initiation induced pressure waves in a medium adjacent to PETN were observed by high speed camera. High speed camera of Cordin 220 was used, which can record six frames by image-intesified CCD camera with 10 ns exposure time and arbitrary delay time. Optical layout of the experiment is shown schematically in Fig. 1(a). We have used a small flash lamp for the light source of the observation, and quasi-parallel illumination for the phenomena was accomplished due to the high sensitivity of the high-speed camera system. Flash lamp we have used is the very small one commercially available for camera flash type MFT3222 of Miyata Elevam Inc.. Since laser beam was focused through the glass plate to its ground surface, laser fluence must be less than the ablation threshold value of smooth glass surface, 20 J cm^{-2}.

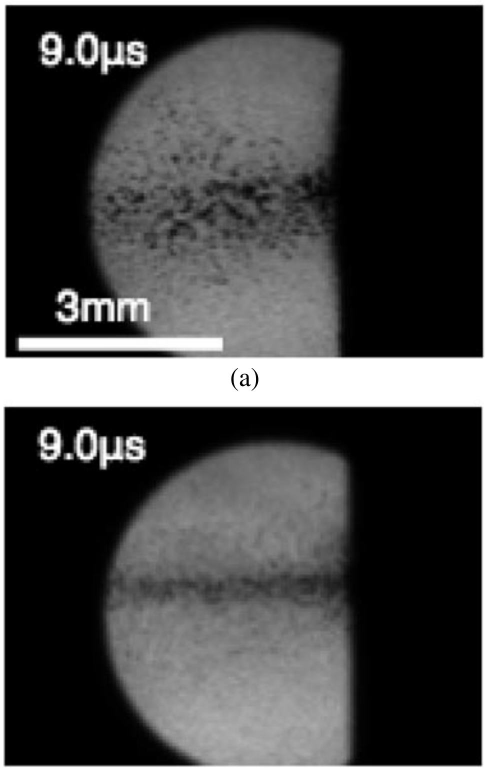
We have used diffusion plates with different surface roughness. Diffusion plate is a kind of optics to be used for diffusing light beam. We purchased commercially available diffusion plates from SIGMA KOKI Inc., which are labeled #240, #400, #800, and #1500 roughness levels. All the diffusion plates we have used are made of fused quartz with 25 mm in diameter and 2 mm in thickness. Diffusion plate having smaller number label indicates rougher surface plate. They have a roughened surface on one surface. This is important in this study, since laser beam should be focused through the glass material. Figure 1(b) shows the schematic illustration of diffusion plate with proper direction of laser irradiation. Microscopic and/or SEM inspection of the surface, however, shows that even rough surface has broad undulations of shorter to longer wavelengths. Therefore, all plates are composed at least of shorter wavelength structures.

3. Experimental results

3.1 Shadowgraph observation and ejected particle recovery

In the present study, it is found from high speed photography that particle generation lasts fairly long time after laser irradiation. Figure 2 shows some of the examples of this behavior. These two pictures are taken at 9 μs after ablation. One may see many particles are still emanating from the focused area for #240 and #800 diffusion plate. In laser ablation of most materials, ablation plume is produced roughly several tens of times the laser pulse duration. Particle generation process in this phenomenon is, therefore, considered to be very long compared with common ablation process. Ejected particles might have size distribution and the distribution depends on time. Information on the particle size distribution, however, cannot be obtained from high-speed photos.

SEM images suggest that glass fragments whose size is larger than geometrical roughness or mean wavelength of the original ground glass surface could be produced by laser ablation. Ejected particles may have their own size distribution. Parameters in this process are laser parameters such as laser fluence and initial surface roughness, etc. In order to obtain size distribution resulting from this process, we have performed a series of experiments by directly capturing particles. These experiments were performed in vacuum to avoid shock wave effects. Part of the particles are collected by a PMMA plate ahead of the ground glass plate, and residue of the particles are collected by another PMMA plate placed beneath the experimental setup. Glass fragments are found stuck on the surface of PMMA plate as shown in Fig. 3(a). Photograph of PMMA plate surface with stuck fragments was analyzed by image analysis program to identify the size of each particle. We have tried to estimate the particle diameter based on several methods, for example, by (i) the cross section of the particle photo, (ii) the length of the particle periphery, etc. Results obtained by different methods, however, gives almost similar size distribution. Size distributions obtained from PMMA plate ahead and from bottom plate are found



(a)

(b)

Fig. 2 High speed camera photos of laser ablation of optical diffusion plate at later delay time of 9.0 μs. Two examples of diffusion plates with different surface roughness, #240 (photo (a)) and #800 (photo (b)).
Laser energy, focused diameter and fluence is 0.38 J, 1.4 mm, and 25 J cm^{-2}, respectively.

to be slightly different, in that more large particles are collected at the bottom plate.

Figure 3(b) and (c) shows the particle size distribution of diffusion plate experiments of #240, #400, #800 and #1500 surface roughness. Distributions are obtained by averaging data of several experiments with the same conditions.

No qualitative differences in the form of the distribution function by each image analysis method were found. Particle size distribution shown in Fig. 3(b) is the sum of particles for both PMMA plates ahead and bottom of ground glass plate, and is the average of size distributions estimated by several methods

One may note immediately from Fig. 3 that size distribution is almost the same for each diffusion plate with different surface roughness. A peak of the number of fragments is seen at around 7-8 μm. For rougher plates of #240 and #400, another peak is seen around 25 μm. The most striking difference of the distribution is the number of particles produced by one laser ablation.

For the comparison of the difference in distribution for each diffusion plate, the distribution function is normalized by the maximum number of recovered particles and is shown in Fig. 3(c). From this figure, one may note that normalized size distribution function is quite similar irrespective of the surface roughness. This might be an example of a kind of a scaling law. This result can then be attributed to the scaling law of glass material fracture by instantaneous energy deposition. One may also note that size distribution function has two peaks, 7-8 μm and 25 μm. One of the possible explanation of this result is that 25 μm particles tend to be broken into 7-8 μm size particles. This means roughly 100 spherical particles of 7-8 μm from one 25 μm spherical particles. Although another peak at particle size less than 2 μm is possible, optical inspection of small particles less than several μm especially 2-1 μm contain significant error by optical microscope.

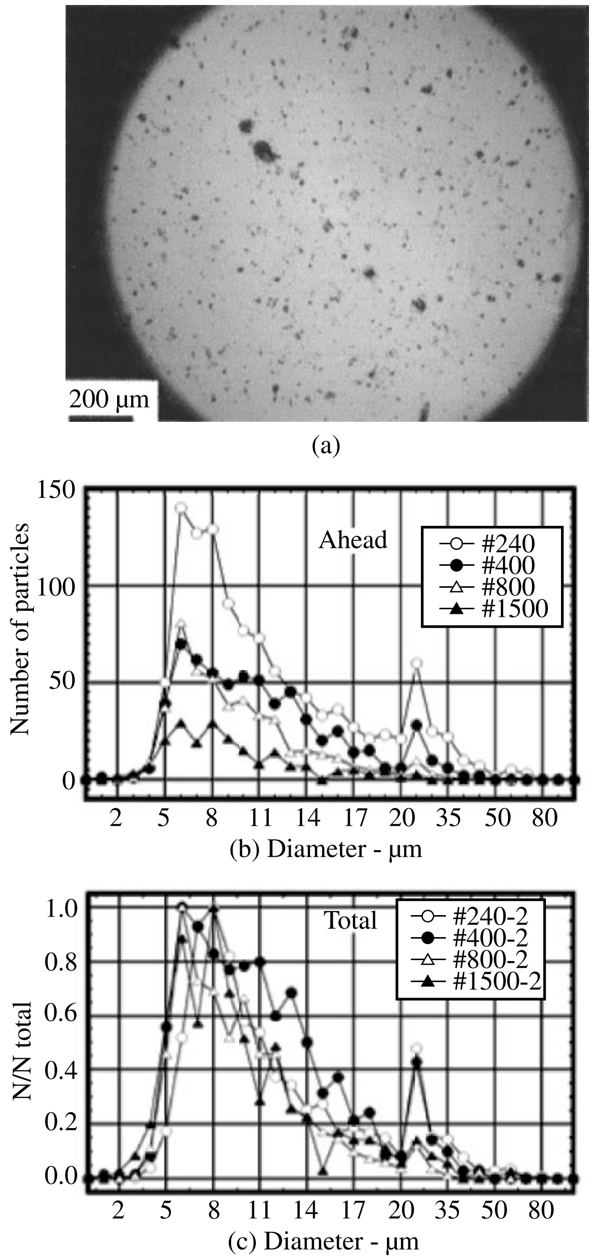


Fig. 3 Particle size distribution for each diffusion plate with different surface roughness. Figure (b) corresponds to size distribution for particles both at PMMA plate ahead of and beneath the target. Figure (c) shows particle size distribution for particles normalized by the peak value, respectively.

3.2 Initiation of PETN powder

Energy conversion of laser energy for ground glass ablation is totally different from that for thin metal layer on roughened surface. It is therefore expected that initiation mechanism of the present experiment is different from those of laser induced initiation of high explosives [1)-6)]. In case of ground glass ablation, part of the laser energy is converted to high velocity glass particle kinetic energy. Feasibility of initiating high explosive by this kinetic energy has been examined systematically by using diffusion plates. A diffusion plate with one ground surface was used and put on the PETN powder instead of using PMMA plate with a roughened surface. Similar scenario might be expected to the experiment of initiation of PETN grain by the collision with a laser-accelerated thin metal flyer by Watson et al [13)].

Based on the information on the timing of initiation, high-speed framing photography of the event was planned. In case of framing photography, we have observed the detonation of PETN powder by observing the high pressure stress wave in PMMA plate attached directly with PETN powder. Detonation of PETN is initiated by pulse laser ablation of ground glass plate which is attached to the PETN powder. Schematic illustration of the target assembly is given in Fig. 4. Figure 4 also shows typical framing photographs of stress wave propagation in PMMA. Two waves traveling in PMMA are seen in the figure. Preceding wave is expected to be induced by the pulse laser ablation, and successive wave is produced due to detonation of PETN powder. Estimated propagation velocities of these two stress waves are both faster than sound velocity of PMMA. Distance between two wave fronts represents a finite delay time of pulse laser ablation and induced detonation wave. Whether the reaction of PETN in this case is the detonation or the deflagration is examined by the comparison of the propagation velocity of reaction along the

PETN layer with the published data of detonation velocity of PETN of similar initial density. At least secondary stress wave in PMMA may have rather high stress level, since shock-compressed high density region after stress wave front is dark due to little light transmission by appreciable change in refractive index of PMMA by shock compression.

In the initiation experiments, the most important parameter is the laser fluence. Since diameter of laser focus for initiation does not have a large value, precision of the laser fluence measurement is almost determined by that of focused diameter measurement. In the present study, laser fluence was measured by inspecting the trace on the aluminum plate by microscope, which was laser ablated at the exact position of the PETN specimen. This procedure is repeated in each experiment. By adopting such a procedure, precision of measuring laser fluence increases appreciably to ensure the reproducible data acquisition.

Figure 5 shows examples of streak photographs taken by the image converter camera. Two typical examples are shown, (a) short delay initiation, and (b) longer delay detonation. The earliest intense flash in the figures is the one by emission caused by laser ablation of ground glass surface. Long streak of luminescence after some break in case (a) indicates emission caused by detonation of PETN powder. Bright region is seen to spread to the direction of camera slit gradually. Since the slope of this bright region corresponds to the propagation velocity of detonation, gradual change in slope indicates the acceleration of reaction front approaching the steady state detonation.

GO/NOGO results were identified by several criterions such as inspection of (i) streak photographs, (ii) broken specimen, (iii) explosion sound, and (iv) smell, etc.. The present criterion is found to be sufficient for judgement of the process at least in this experiment. Experimental results and conditions are summarized in Table 1.

Break in time between laser irradiation and onset of detonation emission is called here the initiation delay time τ. Initiation test results can be divided into three categories; (i) initiation with short delay time less than 1 μs, (ii) initiation with longer delay time larger than 1 μs, and (iii) no initiation. It is apparent that case (i) can be realized in case of higher laser fluence.

4. Discussion

In the present experiment, pulse laser is focused through transparent glass material to the ground surface. Therefore, laser fluence is less than the inside mterial breakdown threshold fluence and also the ablation threshold fluence of smooth glass surface. Laser ablation of ground glass surface takes place even when the laser fluence is less than both of these threshold fluence of glass material of about 20 J cm^{-2}. This is the effect of enhancement of absorption of surface by roughening. Ablation threshold of ground glass surface is at least less than half of that for smooth surface. We do not know, however, what amount of laser energy is absorbed at the ground surface. It is difficult to estimate the pressure generated by ground glass ablation, and then understanding whether only the generated plasma plume could initiate the PETN powder is not an easy task. It is known, however, that direct laser ablation of PETN powder through smooth glass plate does not lead to initiation even if laser fluence is close to the glass ablation threshold.

It is shown in this experiment that PETN powder can be ignited by laser ablation of ground glass surface without help of high temperature plasma. As a preliminary experiment, glass plate with no ground surface was used to cover

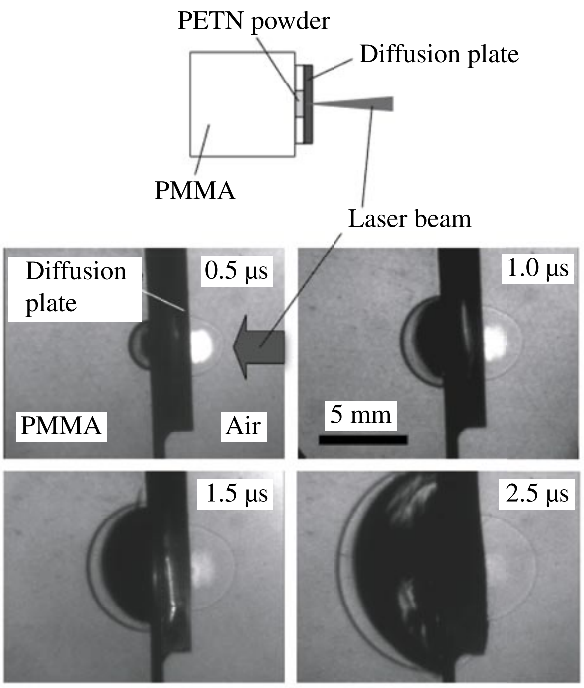


Fig. 4 High speed photograph of stress waves in PMMA attached to PETN powder induced by the detonation of PETN powder for the diffusion plate with surface roughness #800.

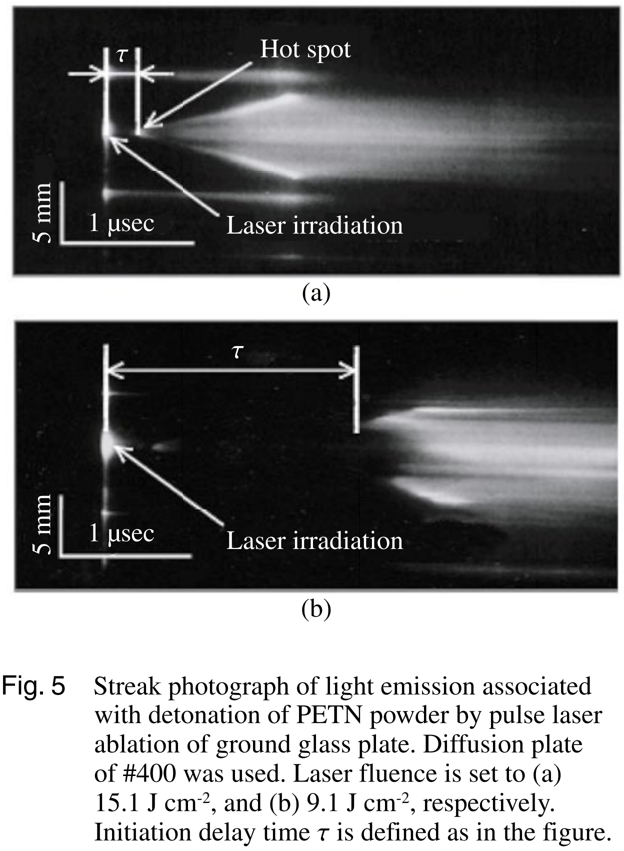


Fig. 5 Streak photograph of light emission associated with detonation of PETN powder by pulse laser ablation of ground glass plate. Diffusion plate of #400 was used. Laser fluence is set to (a) 15.1 J cm^{-2}, and (b) 9.1 J cm^{-2}, respectively. Initiation delay time τ is defined as in the figure.

Table 1 Experimental data for initiation of PETN powder by laser ablation.

Diffusion plate	PETN mass mg	Initial density g cm^{-3}	Laser energy mJ/pulse	Laser fluence J cm^{-2}	Delay time μs	GO/NOGO
#240	9.4	0.7	123	15.1	0.33	GO
#240	10.9	0.8	120	11.2	0.27	GO
#240	12.1	0.9	118	11.0	–[a]	GO
#240	13.3	0.9	120	10.1	0.33	GO
#240	9.4	0.7	124	9.2	–	GO
#240	9.7	0.7	114	9.0	0.70	GO
#240	10.7	0.8	119	9.0	∞	NOGO
#240	11.3	0.8	119	9.0	1.55	GO
#240	11.4	0.8	121	6.9	–	GO
#240	10.9	0.8	118	6.1	∞	NOGO
#240	10.1	0.7	114	5.1	∞	NOGO
#400	10.1	0.7	123	15.1	0.20	GO
#400	9.5	0.7	120	11.2	0.30	GO
#400	13.5	1.0	120	10.1	∞	NOGO
#400	12.3	0.9	120	10.1	∞	NOGO
#400	12.2	0.9	120	10.1	1.62	GO
#400	12.9	0.9	123	9.1	–	GO
#400	10.1	0.7	112	9.1	∞	NOGO
#400	8.9	0.6	112	9.1	1.87	GO
#400	11.8	0.8	119	7.0	1.32	GO
#400	11.2	0.8	118	6.1	1.60	GO
#400	12.2	0.9	114	5.1	∞	NOGO
#800	12.5	0.9	125	15.0	0.20	GO
#800	10.1	0.7	119	10.9	0.27	GO
#800	8.7	0.6	119	10.9	∞	NOGO
#800	14.4	1.0	119	10.0	1.32	GO
#800	12.7	0.9	122	9.1	∞	NOGO
#800	10.0	0.7	114	9.0	∞	NOGO
#800	14.0	1.0	119	9.0	0.33	GO
#800	9.5	0.7	119	9.0	0.34	GO
#1500	11.7	0.8	117	15.2	0.23	GO
#1500	12.1	0.9	117	10.9	0.30	GO
#1500	9.8	0.7	119	10.0	0.33	GO
#1500	11.1	0.8	119	9.0	∞	NOGO
#1500	10.8	0.8	119	9.0	0.30	GO
#1500	12.1	0.9	119	9.0	2.25	GO

[a] symbol "–" denotes the shot that initiation delay time cannnot obtained.

the PETN powder and tried to initiate PETN through glass plate. If laser beam is focused by a condenser lens of relatively short focal length, glass surface on an interface between PETN powder will be laser ablated to produce high temperature plasma. It is shown that PETN powder can be initiated only by the roughened surface. This means the initiation is induced not by the ablated plasma but by the collision of glass particles at the PETN powder grains. It is, therefore, evidenced that kinetic energy of glass particle cloud is large enough to ignite high explosive.

Dependence of the initiation delay time on the laser was also investigated. The results of a series of experiments are summarized in Fig. 6. From Fig. 6, it is shown that initiation by glass ablation was observed in the case of laser fluence 10 J cm^{-2} or higher. Threshold laser fluence for PETN initiation is found to be higher than the value for plasma-assisted initiation. Critical laser fluence for initiation of PETN by ablation of commerical ground glass published in ref. 12) is lower than that reported in this study. Difference of the experimental condition between previous and present study is (i) glass material, and (ii) aluminum coating on the ground surface. It is plausible that aluminum coating absorbs laser energy which passes through glass surface to produce plasma for initiation of PETN powder.

Kinetic energy of glass particles generated by pulse laser ablation should be a function of laser fluence, laser energy, surface roughness of the glass, loading density of PETN powder specimen, PETN powder grain size, etc. Although Watson et al have succeeded in igniting PETN powder by laser-accelerated metal flyer, they can ignite PETN powder

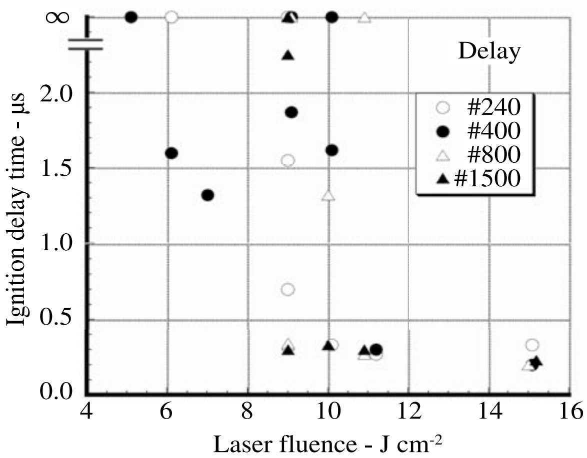


Fig. 6 Initiation delay time as a function of laser fluence. All data of Table 1 are plotted.

whose grain size is 1 μm. By their method, PETN powder of larger grain size of 100 μm cannot be ignited due to the short pressure duration induced by the thin metal flyer impact with the explosive grains. It is apparent that in this situation, PETN powder of larger grain size has less sensitive to pressure pulse of short duration.

In the present study, we used PETN specimen of the average grain size of about 100 μm, then it is required to deposit enough energy to ignite. In every streak photographs of initiation experiments including no initiation cases, a slight emission probably due to the generation of a hot spot is observed 200-300 ns after laser ablation. Location of the hot spot is always at the position of laser focus. This result suggests that impact of glass fragments with PETN grains have large enough energy to induce partial reaction near the impact area. Since streak camera looks the PETN specimen from rear side of the laser focus direction, detection of light emission indicates a local area hot enough to emit light.

According to the analysis of trapped glass particles, the only meaningful difference in the glass particle production by ground glass of different surface roughness is the absolute number of particles, and not the size or velocity of the particles. By the measure of deposited energy on the explosive specimen, larger energy is deposited by rougher glass surface than by smooth surface because of the difference in number of particles. Rougher surface glass produces more glass particles which makes more chance to initiate and evolve into the generation of hot spots. In other words, use of rougher surface glass seems to give more possibility to ignite PETN powders. In some sense, it is surprising in the experimental result that the possibility and delay time of initiation almost does not depend on surface roughness.

Threshold of laser initiation in the present experiment is given almost solely by the laser fluence. By a fixed value of the laser fluence, produced glass particles may have kinetic energy of some definite level. Result strongly suggests that initiation threshold is determined by the value of the kinetic energy of the particles of an average size of 7-8 μm, and not the number of particles, which should be the measure of the number of chances of initiation. One may note that the behavior of initiation near the threshold fluence is quite probabilistic, and almost no dependence on the other parameters such as loading density, surface roughness, etc.

In our experiments, we have used several kinds of diffusion plate of 2 mm thick. Thickness of glass plate should be another parameter to keep the elevated pressure by impact due to inertia. This may influence the delay time and/or go/nogo results. The extent of influence, however, is expected to be limited, if the kinetic energy of glass particles is a key parameter for initiation.

5. Conclusion

In this paper, we have shown that ablation of ground glass produces small glass fragments fairly long time of over 10 μs after laser ablation. Glass particles generated by ablation were collected and size distribution of them is derived. PETN powder is successfully ignited by pulse laser ablation of ground glass of several different surface roughness. From these experiments, produced glass particles have very similar size distribution irrespective of surface roughness, but increases the number with increasing roughness. Initiation threshold fluence is about 10 J cm^{-2} and no dependence on the initial surface roughness.

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粗さの異なる水晶拡散板のパルスレーザーアブレーション特性とPETNの起爆
－ガラス破片サイズ分布と表面粗さ依存性－

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この論文は我々のグループが既に報告したすりガラス面のパルスレーザーアブレーションによるPETN粉のレーザー誘起起爆の研究を記述したものである。裏面からガラス板を通してすりガラス面がレーザーによりアブレートされるとガラス破片群が発生することがわかった。この研究では，この際立ったアブレーション現象の更なる性質がわかってきた。たとえば，(i) 発生するガラス粒子のサイズ分布は初期の表面粗さにほとんど依存しない，(ii) 表面粗さが増すにつれてアブレーションによって発生する粒子の質量や数が増加する，(iii) 粒子発生はレーザーアブレーションから10 μ秒以上も持続する，等である。

異なる表面粗さを持つすりガラスのパルスレーザーアブレーションによる少量のPETNの一連の実験をおこなった。起爆感度のレーザーフルーエンス依存性について研究した。試験した条件の範囲内では，レーザーフルーエンスの閾値は10 J cm^{-2}である。この値より小さいフルーエンスによる実験では，起爆確率が減少し，アブレーションからの遅延時間は初期の表面粗さによらず長くなる。このことは起爆が発生するガラス粒子の運動エネルギーの大きさにのみ左右されることを示唆している。

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