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Time-dependent photothermal characterization on damage of fused silica induced by pulsed 355-nm laser with high repetition rate*

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Time-dependent damage to fused silica induced by high frequency ultraviolet laser is investigated. Photothermal spectroscopy (PTS) and optical microscopy (OM) are utilized to characterize the evolution of damage pits with irradiation time. Experimental results describe that in the pre-damage stage of fused silica sample irradiated by 355-nm laser, the photothermal spectrum signal undergoes a process from scratch to metamorphism due to the absorption of laser energy by defects. During the visible damage stage of fused silica sample, the photothermal spectrum signal decreases gradually from the maximum value because of the aggravation of the damage and the splashing of the material. This method can be used to estimate the operation lifetime of optical elements in engineering.

Keywords: photothermal spectroscopy, fused silica, laser-induced damage

PACS: 79.10.Ca, 42.70.Ce, 79.20.Ds **DOI:** 10.1088/1674-1056/ab671d

1. Introduction

Fused silica optics is widely applied to large lasers[1–4] because of its excellent optical transmission as well as mechanical and thermal properties to ultraviolet spectrum. However, due to the impurity defects introduced in the process of processing and polishing, the irreversible damage to fused silica optical elements occurs when the laser fluence is far larger than the laser intrinsic damage threshold.[5–13] Therefore, the service life of fused silica optical elements and output peak power of high-power laser systems are greatly reduced.

Defects in fused silica optical elements play an important role in laser-induced fused silica damage.[14–21] Therefore, it is of great significance to characterize the internal defects of fused silica optical elements. In the experiment, surface damage morphology of optical element is observed by optical microscopy and scanning electron microscopy. The observed surface damage to the sample is visualized. However, optical microscopy and scanning electron microscopy cannot detect the changes of the sample before the visual damage occurs on the surface of the sample. Photothermal spectroscopy has great advantages in the measurement of weak absorption.[22,23] Photothermal spectroscopy is a non-contact and non-destructive method to measure the absorption defects on the surface and in bulk of optical element.[24,25] Compared with other absorption measurement methods, photothermal technology has the advantages of high sensitivity (< 10 ppb), high spatial resolution (< 1 μm). Photothermal absorption measurement based on photothermal effect is a relative measurement technology. In a certain range of test parameters, the photothermal absorption signal is directly proportional to the light energy absorbed by the sample, and the energy absorbed by the sample directly depends on the absorption rate of the sample. On the basis of satisfying the response linearity of the system, the absorption rate of the sample to be measured is obtained by calibration through using the sample with known absorption rate and its photothermal absorption signal. In this process, the sample irradiated by pump laser absorbs the laser energy and generates various physical responses due to local heating. These physical responses are detected by another weak probe laser. The intensity of photothermal absorption signal is closely related to the mechanism of damage to fused silica irradiated by laser. Previous investigations have focused on the morphology of laser-induced damage and the regularity of damage development with the change of parameters, but few papers have been reported on the initial stage of laser-induced damage. For the pre-damage stage and visible damage stage of fused silica, neither the change of photothermal absorption signal intensity with irradiation time nor the analysis of the process has been reported.

In this paper, the influence of laser irradiation time on photothermal absorption signal is discussed. In particular, two stages of pre-damage and visible damage of fused silica irradiated by high frequency ultra-violet (UV) laser are investigated by photothermal spectrum and optical microscope. The damage process of fused silica material by UV laser is analyzed by the change of optical spectrum absorption signal intensity. These results have not been reported, which can provide a reference for the damage repair of laser-induced fused silica op-

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tical elements in engineering.

2. Sample preparation and characterization

2.1. Sample preparation

Fused silica samples (20 mm ×20 mm ×10 mm; UV-grade Heraeus Suprasil S312) were polished by a conventional pitch-polishing process with CeO_2, with final surface roughness of 1 nm (RMS). Samples were irradiated by a 20-ns 355-nm pulsed laser with high repetition rate UV. The laser spot radius of near-Gaussian beam focused on the sample was 10 μm. The repetition frequency of the laser was 100 kHz. Laser power varied from 1 W to 10 W. Laser-induced damage threshold test was carried out in R-on-1 mode in ambient condition. In an R-on-1 mode, the damage threshold test was implemented by ramping the laser fluence incrementally in steps of 0.1 J/cm^2 until the damage occurred. In this study, the laser damage threshold of the sample was 8.9 J/cm^2.

2.2. Photothermal spectroscopy measurement

Each of all samples was divided into nine squares, each with 6 mm ×6 mm in size and 1 mm away from the edge as shown in Fig. 1. Firstly, for a selected power, region 1 was irradiated to a visible damage state in the center, the time T was recorded, and the photothermal absorption signal at this time was collected. Regions 2–8 were irradiated for $1/8T$, $2/8T$, ..., and $7/8T$, respectively. The irradiation time of region 9 was $1.5T$. Finally, the photothermal absorption signals measured in each region are compared with each other.

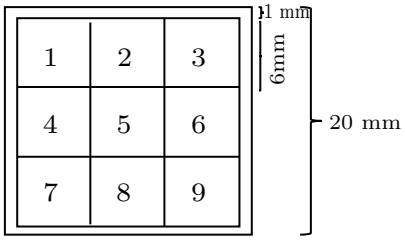


Fig. 1. Each of all samples is divided into nine squares, each with 6 mm in side length and 1 mm away from edge. Irradiation times of squares 1–9 are $1T$, $1/8T$, $2/8T$, ..., $7/8T$, and $1.5T$, respectively.

A commercialized photothermal measuring system (PTS-2000) was employed in this experiment. The detail of the apparatus is described in Ref. [24]. This method was used for measuring the on-line photothermal absorption signals of samples before and after laser irradiation damage. A Q-switched 355-nm laser with an average output power ranging from 1 W to 10 W and a repetition rate of 100 kHz was used as a pump source incident vertically on the sample surface. The photothermal absorption signal of the irradiated area could be collected on-line by using a 5-mW continuous wave (CW) He–Ne laser as an incident probe beam on the sample surface with an angle of 30°. The sensitivity of the device was 10 ppb and the spatial resolution was 10 μm in this experiment. The tested surface was rear surface.

2.3. Scanning electron microscope (SEM)

Cold field emission scanning electron microscope (JSM-6701F) was employed to observe the morphology of the damage pit in our experiment. Scanning electron microscopy (SEM) was used to observe the morphology of the damaged pit, an about 1-μm-thick gold layer on the fused silica sample surface was sprayed to increase the conductivity of the sample surface. The size of the damaged pits was in a range of several hundred microns.

3. Results and discussion

The experimental results show that when the laser power is in a level of 10 W, the sample is instantaneously destroyed by the laser. When the laser power is controlled at 5.5 W, the visual damage is found when the sample is irradiated by laser for 0.1 s. When the laser power is 5.3 W, the visual damage is found when the sample is irradiated by laser for 80 s. When the laser power is 5 W, the visual damage of the sample is found after 6-min laser irradiation. When the laser power is adjusted to 1 W and irradiated by the laser for 4 h, no visual damage is found in the sample.

3.1. Morphology and size of damage pits as well as damage threshold

In this paper, the laser power is defined as the laser-induced damage threshold of fused silica when the one-shot laser irradiates the fused silica, which causes visible damage to the exit surface of fused silica. When the laser power is 5.5 W, the visible damage to the back surface of fused silica sample is caused by one-shot laser irradiation, which can be found in the damage test of fused silica irradiated by UV laser. Therefore, in the experiments mentioned in this paper, the damage threshold of laser-induced fused silica is 5.5 W and the conversion flux is 8.9 J/cm^2.

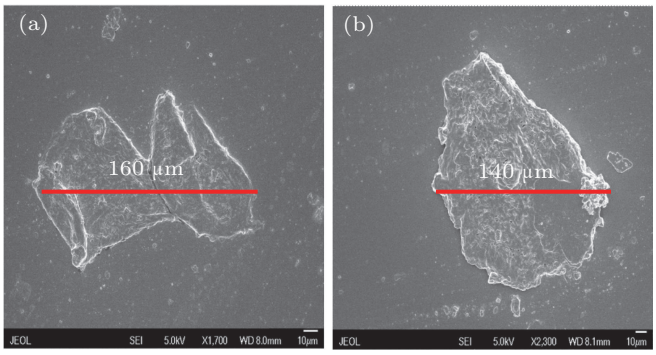


Fig. 2. SEM images of the exit surface of fused silica sample when the visible damage is observed at (a) 6 min after irradiation by 5-W high-frequency 355-nm laser, and (b) 80 s after irradiation by 5.3-W high-frequency 355-nm laser.

Figure 2 shows the scanning electron microscopy (SEM) images of the exit surface of fused silica sample when the visible damage is observed. Figure 2(a) shows the SEM image of

fused silica irradiation by 5-W high-frequency 355-nm laser for 6 min. The size of the damaged pit is about 160 μm as shown in Fig. 2(a). Figure 2(b) displays the SEM image of fused silica irradiation by 5.5-W high-frequency 355-nm laser for 80 s. The size of the damaged pit is about 140 μm as shown in Fig. 2(b).

3.2. Photothermal spectroscopy and optical microscopy of undamaged sample

Figure 3 shows the photothermal spectral characteristics and the optical micrograph of the undamaged region of the fused silica sample. Figure 3(a) describes the photothermal spectrum signal measured by the non-irradiated fused silica sample. Figure 3(b) describes an optical micrograph obtained at the same location as the optical micrograph of non-irradiated fused silica. The nanosecond UV laser damage of fused silica is probabilistic, and the distribution of surface defects has a great influence on the stability of the experimental results. In this paper, the results of photothermal spectrum measurement in the non-irradiated region show that the error range of the experimental data is within 0.2. The distribution of surface defects has little effect on the stability of the experimental results.

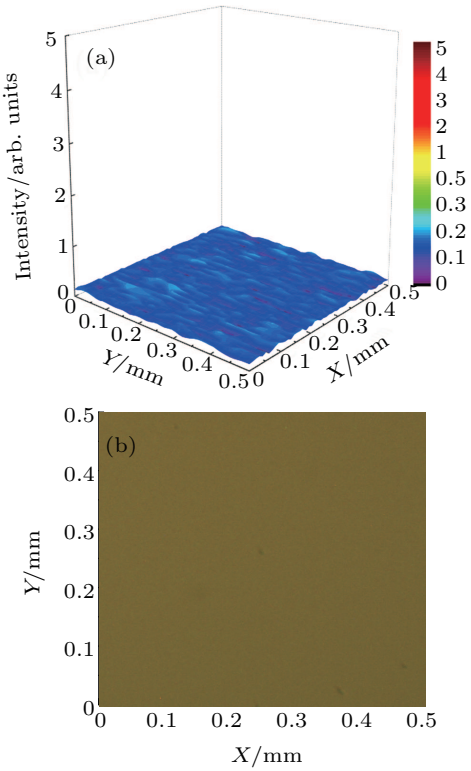


Fig. 3. (a) Photothermal characterization of undamaged area in fused silica and (b) corresponding optical microscopy of undamaged sample.

3.3. Changes of photothermal spectra before and after pre-damage

Figure 4 shows the photothermal characterization and the corresponding optical microscopy images of damage pits of fused silica irradiated by a 5-W high-frequency 355-nm laser at different times. When laser irradiation time on fused silica is 6 min, the visible damage appears on the surface of fused silica. Note the case of $T = 6$ min. When the time of laser irradiation is $5/8T$, the absorption signal intensity of photothermal spectrum on the exit surface of fused silica irradiated is still 0, just like that of non-irradiated sample as shown in Fig. 4(a) and the surface morphology of fused silica has no change as shown in Fig. 4(c). When the laser irradiation time of fused silica is $6/8T$, the absorption signal of photothermal spectrum on the irradiated surface of fused silica changes, unlike that on the unirradiated surface as shown in Fig. 4(b), but the surface of fused silica remains unchanged as shown in Fig. 4(d). At this time, the peak intensity of photothermal spectrum absorption signal on fused silica surface reaches 2. The photothermal spectrum absorption from scratch is due to the absorption of laser energy by defects in fused silica sample during laser irradiation. However, the visual damage cannot be detected by optical microscopy because the degree of visual damage to the surface of the sample is not reached yet.

3.4. Changes of photothermal spectra before and after visual damage

When the laser irradiation time of fused silica is $7/8T$, the absorption signal of photothermal spectrum on the irradiated surface of fused silica is increased as shown in Fig. 5(a) and the surface of fused silica remains unchanged as shown in Fig. 5(c). At this time, the peak intensity of photothermal spectrum absorption signal on fused silica surface reaches 4 as shown in Fig. 5(a). Visual damage is observed on the fused silica surface when the time of laser irradiation is T as shown in Fig. 5(d). At this time, the peak intensity of photothermal spectrum absorption signal on fused silica surface decreases to 2.3 as shown in Fig. 5(b). When the laser irradiation time of fused silica is $1.5T$, the size of the damaged pit increases slightly on the fused silica surface. However, the photothermal absorption signal of the fused silica irradiated surface is lower than that at the beginning of visual damage. At this moment, the peak intensity of photothermal spectrum absorption signal on fused silica surface decreases to less than 1. The reason for causing the intensity of the photothermal absorption signal to decrease is that the continuous irradiation after the visible damage to the sample will lead to the material to smash on the exit surface of the sample.

According to the experimental results shown in Figs. 4 and 5, it is observed that with the increase of laser irradiation time, the intensity of the photothermal absorption signal increases gradually for the absorption of laser energy, caused by defects. The visual damage to the surface of fused silica sample is observed to a certain extent. And then the intensity of the photothermal absorption signal decreases gradually after reaching the peak value due to the smashing of the material on the exit surface of the sample.

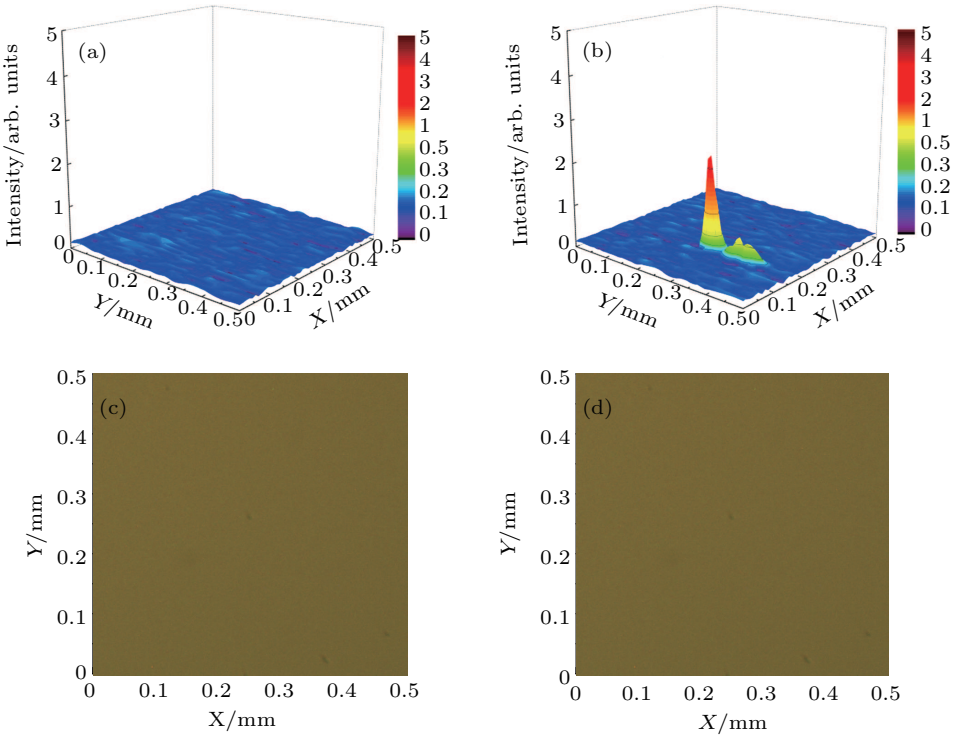


Fig. 4. Photothermal characterization of damage pits in fused silica irradiated by a 5-W high frequency 355-nm laser at time $t = 5/8T$ (a), $6/8T$ (b). (c) and (d) Optical microscopic photograph of irradiation points corresponding to panels (a) and (b).

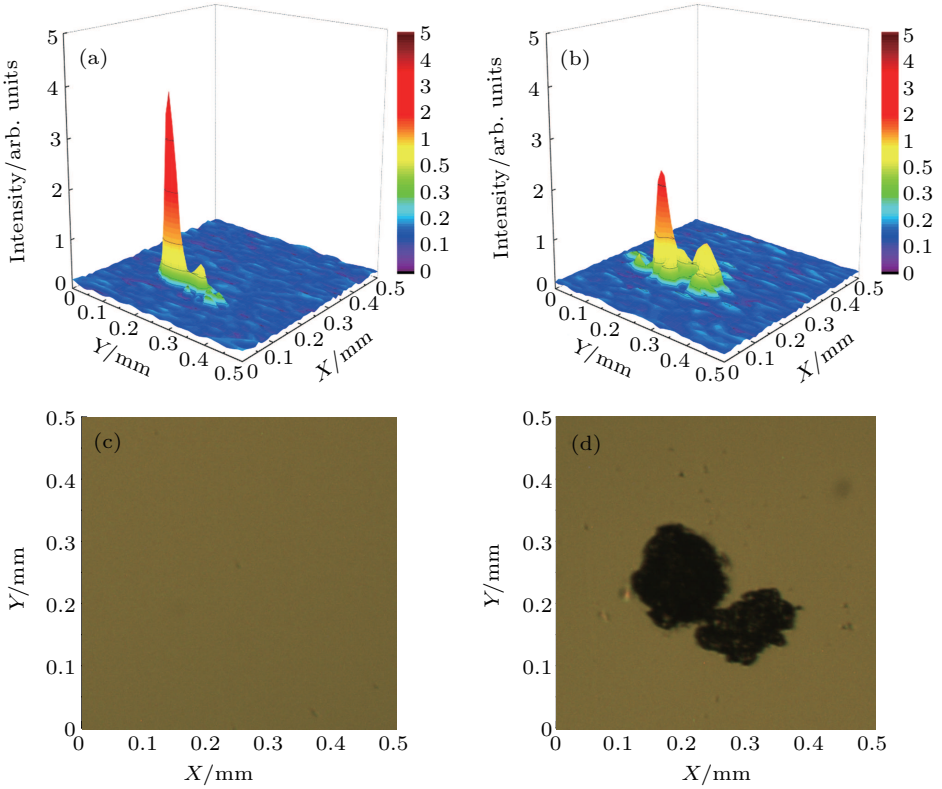


Fig. 5. Photothermal characterization of damage pits in fused silica irradiated by a 5-W high-frequency 355-nm laser at time $t = 7/8T$ (a), $1T$ (b). (c) and (d) Optical microscopic micrograph of the irradiation points corresponding to panels (a) and (b).

3.5. Time dependence of peak intensity of photothermal absorption signals

Figure 6(a) depicts the time dependence of peak intensity of photothermal absorption signal of fused silica irradiated by the 5-W high-frequency laser. The experimental results show that at the beginning of laser irradiation of fused silica, the fused silica sample has no damage and the photothermal absorption signal does not change. With the increase of laser

irradiation time, when the irradiation time is $3/4T$, there is no visible damage to the fused silica surface, but the intensity of photothermal absorption signal increases gradually. When the irradiation time is $7/8T$, the intensity of the photothermal absorption signal reaches a maximum value. As the laser continues to irradiate the surface of fused silica, the intensity of photothermal absorption signal decreases gradually. Visual damage to the surface of fused silica sample can be observed when the irradiation time is 6 min. Laser irradiation of fused silica continues for a period of time, and it is found that the intensity of photothermal absorption signal continues to decrease. From the point of view of the interaction between laser and fused silica with the increase of laser irradiation time, the absorption defects in fused silica sample absorb a lot of laser energy, resulting in the change of local structure. As a result, the local absorption rate of the material increases. Therefore, with the increase of laser irradiation time, the photothermal absorption signal of the material increases. But when the local structure is irreversibly damaged, the temperature of the material is very high because of the laser irradiation. When the melting point of the material is exceeded, the material will melt, relax and recrystallize. The absorption rate of the crystallized material is lower. Therefore, after the visible damage occurs, the photothermal absorption signal of the material will gradually decrease with the laser irradiation time increasing.

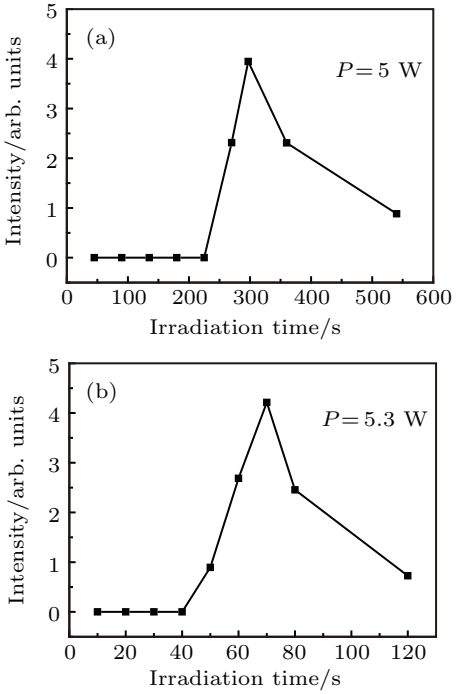


Fig. 6. Variation of peak intensity of photothermal absorption signal with irradiation time at $P = 5$ W (a) and 5.3 W (b).

Figure 6(b) shows the irradiation time dependent peak intensity of photothermal absorption signal of fused silica irradiated by 5.3-W high-frequency laser. The experimental results indicate that at the beginning of laser irradiation of fused silica, the fused silica sample has no damage and the photothermal absorption signal does not change. With the increase of laser irradiation time, when the irradiation time is $5/8T$, there is no visible damage to the fused silica surface, but the intensity of photothermal absorption signal increases gradually. When the irradiation time is $7/8T$, the intensity of the photothermal absorption signal reaches a maximum value. As the laser continues to irradiate the surface of fused silica, the intensity of photothermal absorption signal decreases gradually. Visual damage to the surface of fused silica sample is observed when the irradiation time is 80 s. The laser irradiation of fused silica continues for a period of time, and it is found that the intensity of photothermal absorption signal continues to decrease. With the increase of laser irradiation power, the time of visual damage caused by laser irradiation becomes shorter and shorter.

4. Conclusions and perspectives

In this paper, pre-damage and visual damage process of fused silica sample irradiated by a high-frequency 355-nm laser are investigated by photothermal spectroscopy and optical microscope. The laser-induced damage threshold of fused silica is defined as the laser power value which causes visual damage to fused silica sample when being irradiated once. When the laser power is slightly below the damage threshold, the irradiation time of the laser which produces visual damage decreases with the increase of laser power. If the laser power is too low, even if the irradiation time is too long, it will not cause visual damage to the fused silica surface. The experimental results indicate that when the laser power is slightly lower than the damage threshold, the intensity of the photothermal absorption signal on the fused silica surface increases gradually before the visible damage to the surface of fused silica occurs for the absorption of laser energy by defects, and then decreases gradually after the peak value was reached. Visual damage is observed. That is to say, in engineering, if the intensity of the on-line photothermal spectrum absorption signal on the exit surface of fused silica increases to a certain value and then decreases, the sample will be in visual damage. This has not been reported before. This can be used as a method to judge the service lifetime of optical elements in engineering.

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