

Experimental investigation on laser-induced surface damage threshold of Nd-doped phosphate glass

Junjiang Hu (胡俊江)*, Lei Zhang (张磊), Wei Chen (陈伟), Changhe Zhou (周常河), and Lili Hu (胡丽丽)

Shanghai Institute of Optics and Fine Mechanics, Chinese Academy of Sciences, Shanghai 201800, China

*Corresponding author: hjj@siom.ac.cn

Received September 9, 2011; accepted October 25, 2011; posted online December 8, 2011

Nd-doped phosphate glass is the dominant amplifier material used in solid state high average power laser systems. Surface imperfection and subsurface damage (SSD) of the glass, resulting from the optical fabrication process, limit the increment of laser system energy output. Thus, it is important to enhance the surface damage threshold of Nd-doped phosphate glass surface. The influence of abrasive size, polishing powder, grinding mode, and chemical treatment on the laser-induced damage threshold (LIDT) of Nd-doped phosphate glass surface is investigated. Results show that the LIDT is affected little by different polishing powders and grinding modes. The LIDT correlates with the abrasive size, which produced different depths of SSD. A suitable acid etching treatment can remove the imperfection and the SSD for improving the LIDT of Nd-doped phosphate glass surface. The combination of several effective techniques and methods, which are low-cost and practical, should be useful to enhance the LIDT of Nd-doped phosphate glass surface.

OCIS codes: 140.3330, 140.3530, 240.6700.

doi: 10.3788/COL201210.041403.

In a high power laser system, when the irradiation level reaches a high enough level, laser-induced damage may occur at the surfaces of the optical components, at the interfaces between components, or in the bulk of these components. The study of the theory, mechanism, and improvement of the laser-induced damage threshold (LIDT) has been a major research topic in the laser community. As the dominant and core working material of the amplifier, Nd-doped phosphate laser glass is widely used in high-peak power solid state laser systems, such as SG-II, SG-III prototype, SG-III and NIF, all of which can generate mega-joules of energy at peta-watt power levels^[1]. Bulk laser damage in glass commonly results in inclusions such as Pt particles. With the development of new glass manufacturing processes, optical material free of inclusions and with high optical homogeneity, such as Nd-doped phosphate glass and fused silica, have been produced^[2]. However, improving the LIDT of optics surface still remains a challenge. During the optical fabrication process of cutting, grinding and polishing, surface contamination particles, surface scratches, and subsurface damage (SSD) (e.g., fracture, cracks, and inclusions) can be introduced. These imperfections are likely to absorb the laser energy, increase the electric field density or generate multi-photon ionization, all of which decrease LIDT^[3]. For the high-power laser systems, these imperfections may lead to catastrophic failure on the surface of the optics. This requires a series of post-processing steps that must be employed to eliminate imperfections, such as contaminants, scratches, and SSD. For example, the wet etch and laser treatments can increase the LIDT of the polished surface of the optical material^[4–6]. Recently, many studies have been conducted to correlate the surface structural properties with laser-induced damage. These studies have mostly been focused on improving LIDT of the polished optical material surfaces at 355 and 1064 nm^[7,8]. However, few experiments and discussions

exist on improving LIDT of Nd-doped phosphate glass surface. The SIOM N31 laser glass was chosen for our experimental investigation.

In this letter, we study the effects on surface damage by fabrication processes, such as different abrasive size grinding, grinding modes, acid treatment for removing the surface contamination particles, surface scratches, and SSD. We gain a better understanding of the negative effects of the LIDT of Nd-doped phosphate glass surface, leading to low-cost methods to improve this. The instrument used for laser-damage testing was a flash lamp pumped Nd:YAG laser system, repeating at 1 Hz, beam with 1064-nm wavelength, and 10-ns pulse duration^[9]. The parameters of laser system correlated with laser-induced damage^[10], and served as key factors in determining the reliability and uncertainty of the test results. Such parameters, including laser power, the spatial and temporal profile of the beam and the beam size, were detected during the test process. The effective area was measured to be about 0.36 mm² using an effective area CCD test system. The laser spatial beam profile was approximately Gaussian at about 1 mm on the plane of the sample. The sample was held on an *xyz* position stage. The test damage method was used to produce a single shot of laser pulse on each site on the sample surface before moving to another site at a different laser powers (1-on-1)^[11,12]. The LIDT can be obtained from the laser-induced damage data^[9,11]. The definition and diagnostic methods of the laser-induced damage are described elsewhere^[13–17].

The Nd-doped phosphate glass samples (SIOM N3105, Nd₂O₃ 0.5wt.-%) with diameter and thickness of 40 and 10 mm, respectively, were ground using different loose diamond abrasives with diameters ranging from 40 to 10 μ m. The material of the lapping plate, which was incised on concentric circular grooves, was cast iron. These samples were polished for optical surfaces using the well

known aqueous CeO₂ (TREO>99%, CeO₂>70%) and Fe₂O₃(red rouge) slurries. For the ceria polishing powders, the Mohs scale of hardness was about from 6 to 7, whereas that of the ferric oxide polishing powder was about from 5.5 to 6.5, which was lower than the ceria. The red rouge cut more slowly than the ceria. The slurry was placed on the pitch-bed circular polisher, which had “X” grooves on it. The D50 diameter of the polishing powder was about 2 μm. The influence of the powder particle size on the surface has already been investigated in the released literature^[18]. The samples were prepared in the same way to be as identical as possible. According to the traditional fabrication process of optical materials, the spindle speed was set to low speed, after which the proper load was chosen. The spindle speed and the grinding load have effects on the surface damage and SSD^[19]. In the grinding step, SSD extended into the bulk material with a magnitude nearly equal to the abrasive size^[20]. After grinding, the samples were extremely finished with different polishing powders, and then carefully cleaned to remove the defects (such as grease, dunghill, partial SSD). These were then measured and tested for damages. In order to reduce the hygroscopy of the finished surface, the samples were stored in dry box and tested immediately. The surface roughness (SR) for each finished surface was measured with a no contact surface profiler from Zygo Corporation (white light interferometer). The laser beam was used to irradiate all of these samples, using an incident angle of 0°. LIDT data of this series of tests are presented in Tables 1 and 2.

Table 1. Laser-induced Damage of Nd-doped Phosphate Glass Samples Ground with Different Loose Abrasives and Polished with CeO₂

Abrasive Size (μm)	Theoretical SSD Max Depth (μm)	Surface Roughness RMS (nm)	Surface Roughness Ra (nm)	LIDT (J/cm ²)
10	14.2	0.620	0.511	25.4
14	18.8	0.817	0.648	20.5
20	25.5	0.787	0.642	15.6
28	33.9	0.571	0.459	11.3
40	46.0	0.783	0.633	14.6

Table 2. Laser-induced Damage of Nd-doped Phosphate Glass Samples Ground with Different Loose Abrasives and Polished with Fe₂O₃

Abrasive Size (μm)	Theoretical SSD Max Depth (μm)	Surface Roughness RMS (nm)	Surface Roughness Ra (nm)	LIDT (J/cm ²)
10	14.2	0.721	0.614	26.2
14	18.8	0.617	0.678	22.2
20	25.5	0.887	0.649	20.4
28	33.9	0.672	0.550	17.4
40	46.0	0.795	0.645	17.1

Tables 1 and 2 show the correlation between the experimentally measured LIDT and the theoretically calculated SSD for samples that have been ground using loose abrasives with different diameters. Even though the textures of the sample surfaces are similar, the surface has different LIDTs. The experiment indicates that the smaller diamond abrasive diameter, which generates less SSD, has the higher LIDT. The measured values quoted that the residual SSD dominative occurs in the previous grinding process^[21]. Figure 1 depicts the relationship between LIDT and loose abrasive diameter. It should be noted that the effects of polishing powder on surface damage resistant are not obvious. The LIDT of polishing surface by CeO₂, after conventional grinding with 10-μm diameter loose abrasive, was about 25 J/cm². The LIDT of the polishing surface with Fe₂O₃ was about 26 J/cm². After grinding using the other size of the loose abrasive, the LIDT of the former was a little lower than that of the latter. This suggests that the effects of absorption of polishing powder at the work wavelength are not prominent, indicating that the effects of the absorption of polishing powder at the Nd-doped phosphate glass work wavelength cannot be taken into account.

Figure 2 shows the scanning electron microscope (SEM) of the finished surface and the rear surface damage. Figures 2(a) and (b) indicate similar surfaces polished by different powders. There are some obviously melted areas in Figs. 2(c) and (d). Figure 2(d) also shows the scratches that have become visible after the surface is irritated by the laser beam. The phenomenon indicates that the LITDs are associated with surface scratches.

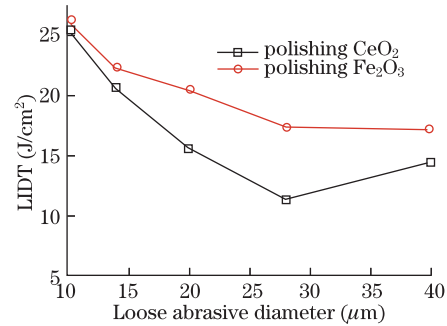


Fig. 1. LIDT versus loose abrasive diameter.

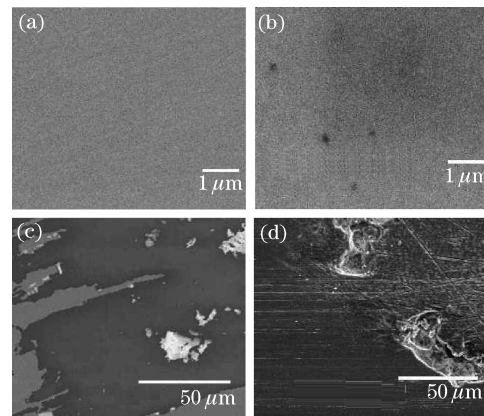


Fig. 2. SEM of the surface and the rear surface damage. (a),(c) Surface finished by Fe₂O₃; (b), (d) surface finished by CeO₂.

In order to investigate the effects of the grinding mode on the LIDT, two sets of Nd-doped phosphate glass similar to the ones mentioned above were tested. The first set of samples, labeled A, was ground using conventional, loose diamond abrasive (20–14 μm and then 14–10 μm) and polished for optical surfaces. The second set of samples, labeled B, was ground using bound abrasive and then polished. In the bound abrasive mode, the abrasive diameter ranged from 14 to 10 μm ; the bound materials used were Fe, Cu, and Si; and the spindle speed and grinding load were similar. The removal mechanisms were fracture and plastic scratching for the loose abrasive and bound abrasive grinding, respectively^[22,23]. The ground SR was analyzed through the surface roughness peak-to-valley (P-V) value, which was measured using a stylus profilometer (Taylor Hobson Company, UK). The relation of SSD and SR was represented by the proportionality constant k . The proportionality constant ($k=3.83$) was obtained Ref. [24]. The experimental results are shown in Table 3. The samples, which were ground by loose abrasive and bound abrasive, then polished with CeO_2 , had LIDT values at around 20 and 19 J/cm^2 , respectively. These suggest that the grind modes having similar SSD depths have little influence on the laser-induced damage of the Nd-doped phosphate glass surface.

Combining the advantages of the above mentioned techniques and methods, a set of similar-sized samples was prepared. These samples were labeled C1 and C2 in Table 4, conventionally ground by loose diamond abrasives (20–14 μm and then 14–10 μm), and etched with a buffered hydrogen fluoride solution^[9]. Then, the dominating SSDs were removed and the samples were polished using CeO_2 . The depth of removing material was equivalent to the depth of the SSD. All samples were irradiated and tested. Table 4 shows that the LIDT of the Nd-doped phosphate glass surface has been enhanced remarkably after combination treatment; in addition, the LIDT of the commonly fabricated sample is about 21 J/cm^2 . The results indicate that the LIDT of Nd-doped phosphate glass surface was increased by about a factor from 1.3 to 1.6 after removing the SSD. In order to evaluate the effect of the SSD on damage susceptibility in theory, we also calculated the light intensification, which can be induced by the SSD. The incident beam was set to be unit and the crack was on the rear surface. The deep crack was 12.9 μm , which was equal to the SSD depth of sample A. The electric field enhancement (EFE) factor was 1.70 in the TE and TM modes (see Fig. 3). The calculation indicated that the electric field is enhanced, which is possibly due to the decreasing LIDT^[25]. The experiment and calculation were also performed to indicate that the major SSD dominated the LIDT.

Table 3. Surface LIDT Ground Using Different Grinding Modes

Grinding Mode	SSD	Ground Surface	Surface	Front Surface
	Depth (μm)	Roughness P-V (nm)	Roughness RMS (nm)	LIDT (J/cm^2)
A	12.90	3.37	0.769	20.3
B	16.85	4.40	0.812	19.9

Table 4. LIDT of the Combination Treatment Surface

Sample No.	Surface Roughness RMS (nm)	LIDT (J/cm^2)
C1	0.99	27.9
C2	0.98	33.7

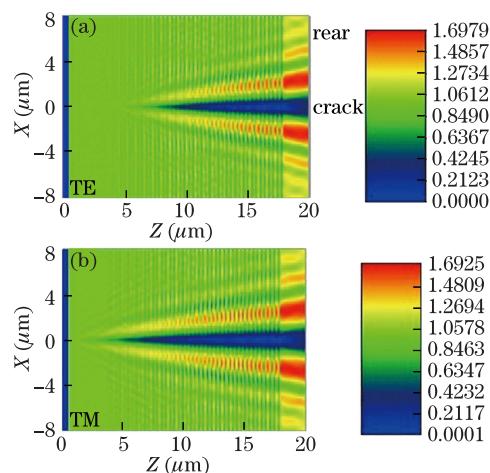


Fig. 3. Electric field intensity distribution in the vicinity of a planar crack (12.9- μm -deep) on the rear surfaces (right). The crack is 0.5- μm wide and perpendicular to the output surface. (a) TE; (b) TM.

In conclusion, the relationship between the surface LIDT and fabrication process for Nd-doped phosphate glass is investigated. The larger diameter of the loose abrasive leads to deeper SSD; in addition, the polishing material has little influence on the surface LIDT. After eliminating contaminations and SSD in the polishing and grinding processes, respectively, the surface LIDT can be improved remarkably. Cleaning and etch processing in acid solution is examined as an effective method for removing surface defects and part of the SSD. The SSD dominates the LIDT of the Nd-doped phosphate glass surface. By eliminating the SSD, the LIDT of polished surface can be enhanced after combination processes. All investigative results provide essential background for increasing the LIDT of the Neodymium-doped phosphate glass surface.

References

- G. H. Miller, Proc. SPIE **5341**, 1 (2004).
- Z. Jiang, Chinese J. Lasers (in Chinese) **33**, 1265 (2006).
- R. M. Wood, *Laser Induced Damage of Optical Material* (IOP, Bristol and Philadelphia, 2003).
- C. L. Battersby, L. M. Sheehan, and M. R. Kozlowski, Proc. SPIE **3578**, 446 (1999).
- H. Chen, D. He, L. Hu, S. Li, and J. Cheng, Chinese J. Lasers (in Chinese) **37**, 2035 (2010).
- S. Xu, X. Zu, and X. Yuan, Chin. Opt. Lett. **9**, 061405 (2011).
- P. A. Temple, W. H. Lowdermilk, and D. Milam, Appl. Opt. **21**, 3249 (1982).
- J. M. Yoshiyama, F. Y. Genin, A. Salleo, I. M. Thomas, M. R. Kozlowski, L. M. Sheehan, I. D. Hutcheson, and D. W. Camp, Proc. SPIE **3244**, 331 (1997).

9. J. Hu, J. Yang, W. Chen, and C. Zhou, *Chin. Opt. Lett.* **6**, 681 (2008).
10. A. V. Smith and B. T. Do, *Appl. Opt.* **47**, 4812 (2008).
11. ISO 11254-1.2, Lasers and laser-related equipment—Determination of laser-induced damage threshold of optical surfaces—Part 1: 1-on-1 test (2002).
12. A. Melnikaitis, D. Miksys, T. Balciunas, O. Balachninaite, T. Rakickas, R. Grigonis, and V. Sirutkaitis, *Proc. SPIE* **6101**, 61011J (2006).
13. X. Liu, D. Li, Y. Zhao, X. Li, X. Ling, and J. Shao, *Chin. Opt. Lett.* **8**, 407 (2010).
14. M. Josse, R. Courchinoux, L. Lamaignere, J. C. Poncetta, T. Donval, and H. Bercegol, *Proc. SPIE* **5647**, 365 (2005).
15. M. D. Feit, A. M. Rubenchik, M. R. Kozlowski, F. Y. Génin, S. Schwartz, and L. M. Sheehan, *Proc. SPIE* **3578**, 226 (1998).
16. R. Chow, M. Runkel, and J. R. Taylor, *Appl. Opt.* **44**, 3527 (2005).
17. J. H. Campbell, F. Rainer, M. Kozlowski, C. R. Wolfe, I. Thomas, and F. Milanovich, *Proc. SPIE* **1441**, 444 (1991).
18. B. Zhang, L. Bao, and J. Zhu, *Acta Opt. Sin.* (in Chinese) **29**, 1905 (2010).
19. W. Zhang and J. Zhu, *Optik* **120**, 752 (2009).
20. P. E. Miller, T. I. Suratwala, L. L. Wong, M. D. Feit, J. A. Menapace, P. J. Davis, and R. A. Steele, *Proc. SPIE* **5991**, 1 (2005).
21. D. Golini and S. D. Jacobs, *Appl. Opt.* **30**, 2761 (1991).
22. P. P. Hed, D. F. Edwards, and J. B. Davis, *LLNL UCRL* **99548**, 1 (1989).
23. P. P. Hed and D. F. Edwards, *Appl. Opt.* **21**, 26 (1987).
24. W. Zhang and J. Zhu, *Acta Opt. Sin.* (in Chinese) **28**, 268 (2008).
25. M. D. Feit and A. M. Rubenchik, *Proc. SPIE* **5273**, 264 (2004).