Experimental investigation of enhancing the subsurface damage threshold of Nd-doped phosphate glass

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Experimental investigation was performed with a 1064-nm, 10-ns Nd:YAG laser to determine the effects of the surface hydrogen acid etching on laser damage, compared with damage of conventionally polished surface. The investigation was helpful for us to understand the negative effects of Nd-doped phosphate glass surface and subsurface damage (SSD) on laser induced damage threshold (LIDT). A set of samples was polished, and then chemically etched in a cool buffered 10%HF + 20%H_2SO_4 solution at different times. Another set of samples was ground and etched in the hot-buffered solution, and then polished. All the samples were irradiated with Nd: YAG laser and characterized by optical microscopy. Results of LIDT were obtained according to International standard ISO/DIS 11254-1.2. Chemical treatment can remove the contaminants in the polished re-deposition layer and the SSD for improving the laser damage resistance of Nd-doped phosphate glass surfaces. The method of using hot solution was more effective than that of using cool solution.

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The output of high power laser systems has been limited for the last four decades because of the laser-induced damage of the interior (bulk) and the polished surface of optical glass. Specially, the laser systems for inertial confinement fusion (ICF) are capable of producing megajoules energy at peta-watt power levels. For example, the 193-beam national ignition facility (NIF) currently under construction at Lawrence Livermore National Laboratory (LLNL) is capable of generating energies up to 3.0 MJ at 1053 nm and, after frequency conversion, 1.8 MJ at the third harmonic 351 nm[1]. Bulk laser damage in glass generally originates from inclusions. With the development of new glass melting and forming processes, it is now possible to make both the fused silica and the Nd-doped phosphate glasses[2], which are the core materials of these systems, free of inclusions and high optical homogeneity. However, the resistance against surface damage is still a critical technological challenge. During the optical processes of cutting, grinding, and polishing, surface contamination particles may be imbedded in or near the surface, and defects such as fracture, cracks, and inclusions exist beneath the polished surface (subsurface damage). These residual contaminants, scratches, and defects are generally ubiquitous, which cause that researchers must employ a series of post-processing steps to reduce or eliminate the laser induced damage from these contaminants, scratches, and defects. For example, the wet etch processing can increase the laser induced damage threshold (LIDT) of the polished fused silica surfaces[3], and the magnetorhelolgical finishing processing (MRF) followed by acid-etching and laser conditioning[4,5] can reduce the damage density. In recent years, extensive studies have been conducted to correlate the surface structural properties with laser induced damage.Considerable attentions have been focused on improving the laser damage resistance of the polished optical surfaces at 355 and 1064 nm[6,7]. In particular, the surface of fused silica has been widely studied. However, few experimental results about the improving LIDT of Nd-doped phosphate glass have been reported.

This letter studies the effects on laser damage by chemical etching treatment for removing the surface contamination particles, surface scratches, and subsurface damage (SSD) of the Nd-doped phosphate glass. We describe a simple and low-cost processing method to improve the LIDT of Nd-doped phosphate glass. The apparatus used for laser-damage testing involve a Nd:YAG laser, operating at 1 Hz, with 1064-nm wavelength and 10-ns pulse duration. The beam was focused down to a spot approximate 1 mm which was $1/e^2$ diameter of the Gaussian beam on the sample plane. The energy of the incident beam was measured with a hydroelectric detector. The spatial profile of the focused beam is analyzed with an optical system linked to a CCD camera, and the temporal profile is measured with a fast photodiode. The effective area, which can increase the accuracy of the LIDT test, was measured about 0.36 mm^2 using an effective area CCD test system[8]. Figure 1 shows a typical damage metrology layout. Samples are observed through a microscope (magnification from 25× to 1000×). The test damage method uses one shot on each unexposed site on the sample surface (1-on-1)[9]. The sample is mounted in an automated scanning manipulator, which is used to move different test parts in beam, and irradiates at different energy densities. The microscopic examination of the testing before and after irradiation is used to detect damage. We obtain the laser damage probability on our samples by counting the number of damage sites at one pulse energy. This procedure is repeated for other pulse energies. We developed a plot of probability versus energy test for each sample. A linear extrapolation of damage probability data to zero damage probability yields the LIDT. Each curve is plotted with 120 data points that involve 12 different pulse energies and 10 tested sites at

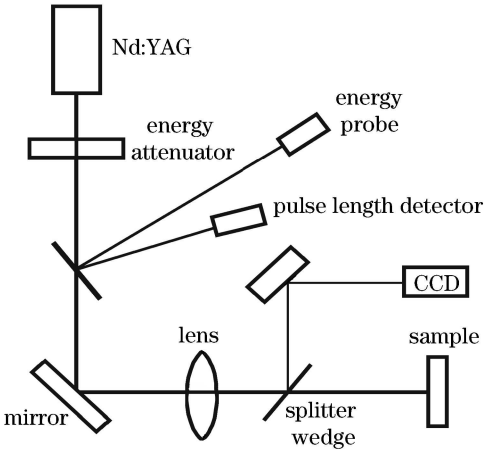

Fig. 1. A typical damage metrology layout.

each pulse energy.

Two sets of Nd-doped phosphate glass (SIOM N3105) of 40-mm-diameter and 10-mm-thick samples were tested. One set of samples, labeled A, was grounded using abrasives ($20-14$ μm then $14-10$ μm) and polished for optical surfaces with CeO_2. Then these samples were cleaned in distilled water and etched in a buffered $10\%HF + 20\%H_2SO_4$ solution with $0-110$ min at 27 °C to eliminate the contaminations and expose the cracks on the surface. Another set of samples, labeled B, was grounded conventionally using abrasives, and etched in a hot buffered solution at 90 °C with different times, and then polished with CeO_2. Etching was done through the whole sample immersion into the etch solution. The surface roughness for each treated surfaces was measured with a no contact surface profiler. The etching rates of the polished samples and the ground samples in the hydrogen fluoride solution were obtained through repeated experiments, as shown in Fig. 2. Etching rate of the polished sample is between 15 and 35 nm/min at 27 °C (the temperature error was ±3 °C), and that of the ground sample is between 60 and 70 nm/min at 90 °C (the temperature error was ±10 °).

The 1-on-1 test results will be discussed using zero probability laser induced damage. The data for analysis were collected from the both surfaces (front and rear). An example (the sample $A_{10\ min}$) of 1-on-1 test distribution is shown in Fig. 3. The laser induced damage

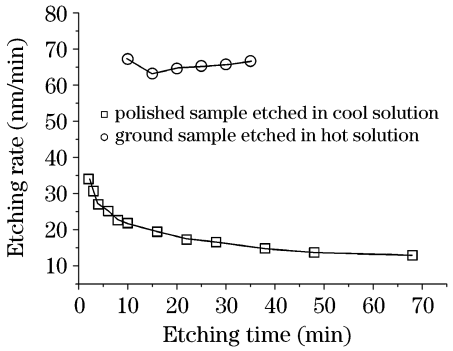

Fig. 2. Etching rate of polished sample and the ground sample.

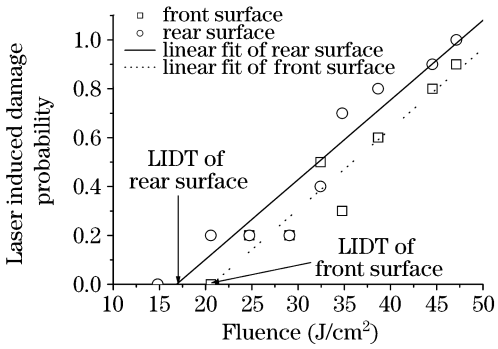

Fig. 3. Experimental results and the linear fit of the $A_{10\ min}$ sample in Table 1.

Table 1. LIDT of the Polished and Etched Nd-Doped Phosphate Glass Samples with Different Times

Sample and Etching Time	Etching Depth (nm)	Surface Roughness (nm)	Front Surface LIDT (J/cm^2)	Rear Surface LIDT (J/cm^2)
$A_{0\ min}$	0	1.06	7.7	4.9
$A_{1\ min}$	46	1.93	8.5	6.2
$A_{3\ min}$	92	1.24	13.6	10.9
$A_{6\ min}$	151	1.44	18.6	14.5
$A_{10\ min}$	218	1.54	21.4	13.7
$A_{20\ min}$	400	1.98	13.6	10.8
$A_{40\ min}$	590	2.31	14.4	9.75
$A_{70\ min}$	903	2.49	9.9	7.6
$A_{110\ min}$	1423	2.61	7.7	4.9

Table 2. LIDT of the Ground and Etched before Polished Nd-Doped Phosphate Glass Samples with Different Times

Sample and Etching Time	Surface Roughness after Polished (nm)	Front Surface LIDT (J/cm^2)	Rear Surface LIDT (J/cm^2)
$B_{0\ min}$	1.06	7.7	4.9
$B_{50\ min}$	0.99	24.5	18.6
$B_{110\ min}$	0.98	29.6	25.7

measurement apparatus were used to irradiate these treated samples. The incidence angle was 0°. LIDT data are presented in Tables 1 and 2.

The polished sample etched in buffered solution with 10 min has a LIDT about 21 J/cm^2, while the polished sample without being etched has a LIDT only about 6 J/cm^2. This indicates that the surface damage resistance of Nd-doped phosphate glass is enhanced approximately 2.8 times after being etched for removal of a 200-nm-thick glass layer. The effect of etching could be the removal of contaminants, such as grease, dunghill, on the surface and the chemical impurities coming from the slurry in the polished re-deposition layer[10−12]. Near the surface contaminates and impurities, the electric field was enhanced or the energy was absorbed to initiate damage[13]. Better resistance against damage can be achieved with etching, but the improvements could not increase the LIDT continuously. In fact, excessive etching decreases the LIDT. In the experiment, after etching 110 min, the LIDT, about 7 J/cm^2, decreases nearly close to that of the unetched surface. The relationship of LIDT and etching time is shown in Fig. 4. The LIDT could be affected by the less surface roughness and the lower SSD exposure through etching. The linear pattern scratches under the surface were observed (Fig. 5). Although the cracks were blunt, the surface roughness was decreased

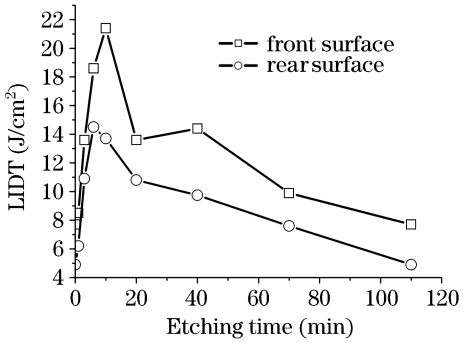

Fig. 4. LIDT of the front and the rear surfaces versus etching time in acid for Nd-doped phosphate glass.

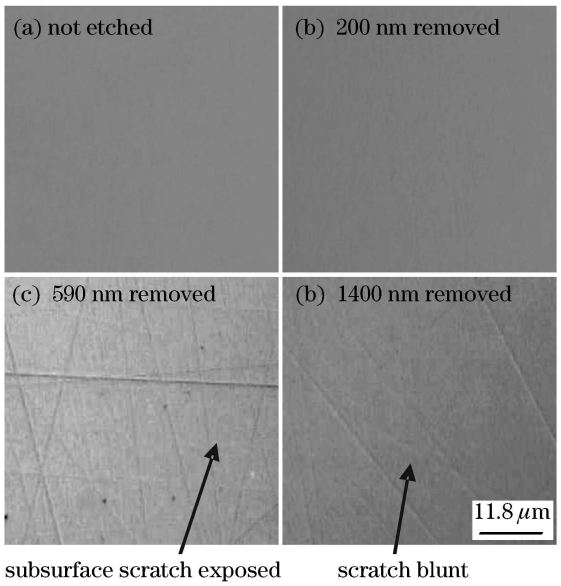

Fig. 5. Micrographs of samples. (a) Without being etched, and being etched with (b) 10, (c) 40, and (d) 110 min.

(Table 1). The images in Fig. 5 suggest that the etching process reveals the SSD hidden under the polished re-deposition layer. This method can effectively remove the contaminants in the polished re-deposition layer to increase the LIDT, while cannot remove the SSD.

Another set of ground samples, which was etched in hot solution with 50 and 110 min to remove about 3-μm-thick and 8-μm-thick materials, and then polished, respectively, had a LIDT about 24 and 30 J/cm^2 respectively. In Fig. 6, we can see that on the sample $A_{10\ min}$, some cracks appear around the central laser damage. We attribute these cracks to the SSD under the polishing re-deposition layer revealed by the laser irradiation. The cracks disappear on the sample $B_{50\ min}$ with the removal of the SSD using hot solution, which suggests that these cracks can produce during grinding. The etched sample after being polished demonstrates the highest threshold 20 J/cm^2. In comparison of the highest threshold 30 J/cm^2 of the polished sample after being etched, it is proved that the LIDT of the latter was more effective than that of the former. The negative effect of the polished process still exists. This indicates that the effect of SSD on laser damage is more negative than that of the contaminants on the polished re-deposition layer. As a whole, the two etching techniques were effective methods for enhancing surface damage resistance. Tables 1 and 2 summarize the experimental results for the polished and ground samples measured in experiments.

Furthermore, a significant difference of the damage morphology of entrance and exit surfaces under irradiation at 1064 nm should be noted. Figure 6 shows the damage morphologies of front and rear surfaces of the sample $A_{20\ min}$ and the sample $B_{50\ min}$. Exit surfaces are more prone to laser-induced damage than entrance surfaces, because exit surfaces endurd more light pressure than entrance surfaces. The relationship of the damage thresholds between the front and rear surfaces can be given by[14]:

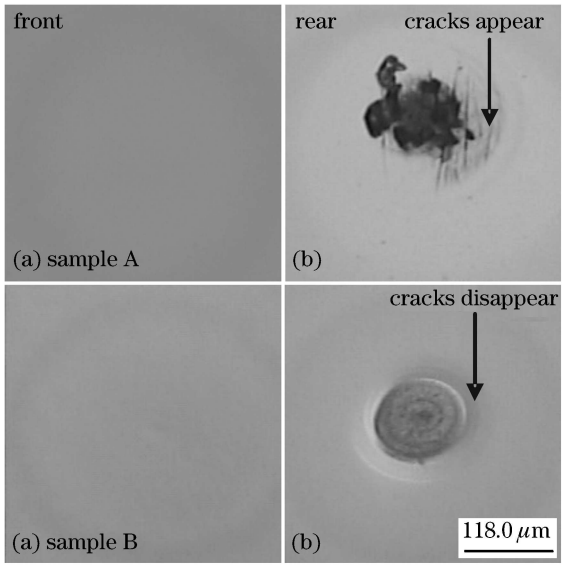

Fig. 6. Micrographs of the front and the rear surfaces damage of two samples with different processes. (a) Etched with 10 min after being polished; (b) etched with 50 min after being ground.

$$\frac{L_{\mathrm{F}}}{L_{\mathrm{R}}} = \frac{4n^2}{(n+1)^2}, \tag{1}$$

where n is refractive index of the material at laser damage wavelength, L_{F} and L_{R} are the LIDT of the front and the rear surfaces, respectively. For SOIM N3105 laser glass, $n = 1.53$ at 1.064 μm, and $L_{\mathrm{F}}/L_{\mathrm{R}} = 1.5$, which is approximately consistent with the statistical average result of 1.40 in the experiment.

The relationship of the surface LIDT and the surface chemical etching process for Nd-doped phosphate glass has been investigated. Two wet etching techniques were used to remove SSD for increasing of laser-induced damage threshold. Wet etch processing in a buffered hydrogen fluoride solution has been examined as an effective method for removing surface or subsurface defects. The LIDT of usually polished surface is enhanced by approximately a factor of 2.8 after cool etched processes. The LIDT of ground and etched surface is enhanced by approximately a factor of 3.8 after usually polishing. In comparison, the latter is more effective. Wet etch processing technique is shown to be a low-cost and practical method to improve the laser damage of the conventional polished Nd-doped phosphate glass surface. The methods would be useful to enhance the SSD threshold of Nd-doped phosphate glass for practical applications.

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