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Laser-induced damage characteristics in fused silica surface due to mechanical and chemical defects during manufacturing processes

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ABSTRACT

Mechanical and chemical defects incurred by grinding and polishing as well as post-processing have been recognized as the most influential culprits that hamper the elevation of laser power/energy in high peak power/energy laser systems. In order to find out the causes for limiting the operational power of laser systems, the effects of these defects on laser damage and removal and mitigation of the defects were investigated in detail in the article. Cracks and scratches were created, annealed, etched and damaged so as to reveal the likely effects of mechanical defects on damage and potential techniques to reduce their influence. The results show that HF-based etching can open and smooth cracks/scratches, improving laser-induced damage threshold (LIDT) at scratches by up to > 250%. Thermal annealing did heal, to some extent, cracks but the LIDT is little improved. Both HF-etching and leaching proves to be effective in removing metallic contamination during polishing process and handling of optics, which can "contribute" to damage/damage density in fused silica. However, HF-based etching may degrade surface roughness, from < 1 nm to > 20 nm under some conditions when > 20 μm material was etched away while the surface roughness was perceptibly altered by leaching (< 1 nm to 1–2 nm). Although the LIDT might not be directly correlated to each individual kind of metallic contaminants or surface roughness, it is found that the surfaces with the highest LIDT's have some distinguished characteristics: clean surface (almost no metallic contamination) plus very smooth surface (RMS surface roughness: < 5 nm). By removing metallic contamination and scratches, surface damage threshold of fused silica can exceed > 30 J/cm^2 (355 nm @3 ns, beam diameter ~400 μm @1/e^2), a significant progress.

1. Introduction

Laser induced damage in optical components has emerged as a momentous issue during the course of constructing high power/energy giant laser systems. The laser-induced damage in fused silica, which is a kind of prevailing material for lenses to transmit ultraviolet (UV) laser light in laser systems, is of paramount importance since energy and shape of laser beam will be affected seriously. The 3ω (351/355 nm) damage threshold of fused silica is theoretically estimated to be > 100 J/cm^2 and the onset fluence of observed bulk damage was experimentally shown to be > 100 J/cm^2 for 15 ns, 355 nm laser [1]. The surface damage threshold, however, is much lower than bulk damage threshold, as low as even < 5 J/cm^2 for a high quality surface of conventionally finished optics [2]. Many factors can affect damage threshold: materials, manufacturing procedures, testing protocols for laser-induced damage threshold (LIDT). Different properties of fused silica materials, such as refractive index, impurities, porosity, dielectric constant, thermal conductivity, etc., may be resulted in due to production techniques of different vendors, which may lead to different LIDT. Another factor that may make comparison unreliable between different damage testing laboratories is LIDT testing apparatus and protocols [3]. The LIDT may not be the same even for the same sample when evaluated with different testing apparatus in different laboratories. The above two factors are out of the scope of this article and we direct our attention to the third factor, manufacturing procedures, exclusive of coating or film deposition and we limit here our discussion to the LIDT of bare substrates of fused silica. On the other hand, the LIDT on rear surface is generally less than front surface when irradiated with 3ω lasers even if both surfaces are perfect and identical. Theoretical calculation shows that the ratio of the fluence F_{front} required to induce damage to the front surface (i.e., entrance surface) to the fluence F_{rear} to damage the rear surface (i.e., exit surface) takes the form of the following equation [4]:

$$\frac{F_{front}}{F_{rear}} = \frac{4n^2}{(n+1)^2}$$

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for dielectrics (n > 1, n is the refractive index of glass), F_{front} is always greater than F_{rear}, indicating higher LIDT of the front surface than the rear surface. Taking perfect fused silica as an example (n=1.47 @355/351 nm), the LIDT of front surface is 1.42 times rear surface, that is, the rear surface will be damaged first when increasing the fluence of incident laser. In the article we limit our discussion to rear surface damage unless otherwise specified.

As mentioned above, surface LIDT is much low as compared to bulk LIDT. The causes are ascribed to defects in the topmost layer of glass, where the structure as well as composition is different from bulk [5–7]. For instance, the layer may contain many tiny cracks invisible to naked eyes and extrinsic impurities [8,9]. These defects in the surface have proven experimentally and theoretically to lower the LIDT of fused silica [5,10–15]. We group the defects into physical and chemical defects. The physical defects refer to those that do not alter the elements of fused silica (e.g. mechanical scratches) while chemical defects introduce foreign elements into fused silica (e.g. chemical impurities). These defects will degrade the damage resistance of fused silica. Chemical impurities can absorb laser light and raise the temperature of local area to melting point or even evaporation point, resulting in permanent damage to fused silica [16–18]. Physical defects may modulate the light field of incident laser and they can also sever as a reservoir of chemical absorbers, aggravating the damage resistance of fused silica surface [16–18]. We thus investigated the most influential defects in the article, specifically speaking, cracks/scratches and metallic contaminants. We have utilized different chemical and physical techniques to process the defects in the hope of mitigating their influence on damage performance of fused silica optics. Cracks were thermally annealed and chemically etched. The LIDT of cracked surface was found not to be recovered through annealing whilst chemical etching can recover the LIDT to a great extent. Thermal annealing somewhat closes cracks while chemical etching opens cracks, which may account for the wide discrepancy between the results processing by annealing and etching. Based on the experimental results on cracks, we chose to process scratches pregnant with numerous micro-cracks with chemical etching instead of thermal annealing. The LIDT was improved by > 250% at the scratches by chemical etching. As to chemical defects, we examined metallic contaminations in detail because of widespread use of metallic oxides as polishing compounds in optical fabrication community, such as ceria, zirconia, rouge, etc. Chemical etching and leaching were employed to clean the polished surfaces of fused silica optics and is it found that both methods have demonstrated great effectiveness in removing metallic contamination, but on most occasions etching deteriorates surface roughness greatly. The details will be presented in the following sections. The experimental procedures are also described and the article ends with a summary to our results. Furthermore, optimal processing methods are suggested to remove or mitigate physical and mechanical defects.

2. Experiments

Fused silica samples of 50 mm in diameter and 5 mm thick were first ground with diamond wheels and pad-polished. The polished samples were then chemically etched slightly and re-polished to ensure that there were no grinding cracks and scratches on the sub-/surface of the samples. Then the samples were ultrasonically cleaned in a seven-tank ultrasonic cleaning machine (Guangwei, China) and dried with an infrared radiator in 10000-class cleanroom [19–21].

Some samples underwent chemical etching in HF-based solutions (HF solutions and HF+NH_4F solutions) of different concentrations for different time. Part of the samples were leached in the mixture of nitric acid and H_2O_2 at 45 °C for 1 h. After etching and leaching, the samples were spray-cleaned with deionized (DI) water before being ultrasonically rinsed in DI water. After that, the samples were dried in 10000-class clean room and ready for experiments. For the samples on which cracks and scratches would be created, the samples were indented or scratched and then cleaned once again in the seven-tank ultrasonic cleaner and dried in cleanroom. Then the samples were etched or thermally annealed.

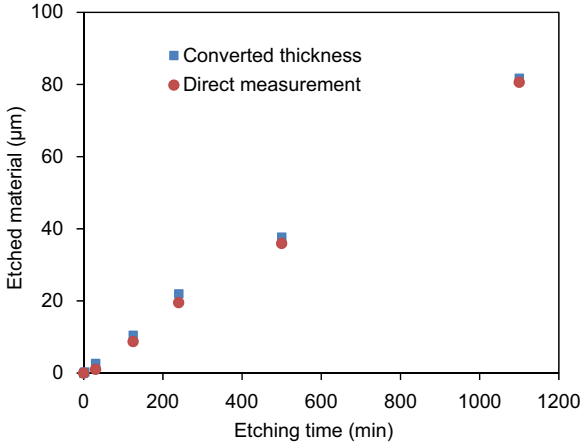

Fig. 1. The converted thickness calculated from weight loss and the direct measurements with a micrometer. The samples were etched in 5.6%HF+12%NH_4F solution.

The etched material was evaluated with an electronic balance (Sartorius CPA225D, Germany) and was then converted to thickness. We also compared the converted thickness from weight loss with the direct measurement with a micrometer and found the two thicknesses agree very well (Fig. 1). Thus we prefer to adopt the converted thickness because etched material by slight chemical etching cannot be directly measured with a micrometer due to limited resolution of the micrometer while it can be precisely evaluated with an electronic balance. Surface roughness of the samples was inspected with an optical surface profiler (Zygo NewView 7200, USA). The concentrations of HF-based solutions and HNO_3 solutions were determined with a potentiometric titration (Metrohom Titrando, Switzerland). Some samples with cracks and scratches were also examined with laser fluorescence microscopy (Nikon A1, Japan) in order to characterize cracks and scratches.

Damage testing for the LIDT was performed on rear surfaces of samples with a Nd:YAG pulsed laser (Beamtech SGR-Extra-10, China) of pulse duration 10 ns, 1 Hz repetition rate at 355 nm and ~400 μm beam diameter at $1/e^2$ (Fig. 2). The exit surface of fused silica optics was normally illuminated by a focused beam with Gaussian spatial and temporal profile. The irradiated area was inspected by a long-focus microscope (20×magnification) equipped with a CCD camera (resolution < 10 μm) to record damage initiation. Laser induced damage is considered to have occurred when physical irreversible change is observed with the microscope. The testing system was used for both R-on-1 and 1-on-1 testing protocols. In 1:1 testing each site on the sample was exposed to a single shot and 60–100 sites were tested with various fluences to obtain the 1-on-1 thresholds. R:1 testing deter-

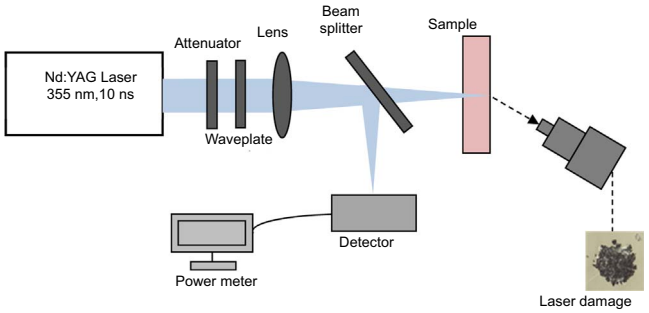

Fig. 2. A 3ω (355 nm, 10 ns) Nd:YAG pulsed laser at a repetition rate of 1 Hz was used to irradiate the rear surfaces of samples (exit surface), at near-zero incident angle. A CCD camera monitored damage initiation.

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