Applied Surface Science 357 (2015) 498–505

Contents lists available at ScienceDirect

Applied Surface Science

journal homepage: www.elsevier.com/locate/apsusc

Laser induced damage characteristics of fused silica optics treated by wet chemical processes

Hui Ye[a,b], Yaguo Li[b,*], Zhigang Yuan[b], Jian Wang[b], Wei Yang[a], Qiao Xu[c]

[a] School of Mechanical & Electrical Engineering, Xiamen University, Xiamen 361005, China
[b] Fine Optical Engineering Research Center, Chengdu 610041, China
[c] Research Center of Laser Fusion, China Academy of Engineering Physics, Mianyang 621900, China

ARTICLE INFO

Article history:
Received 8 May 2015
Received in revised form 27 August 2015
Accepted 6 September 2015
Available online 8 September 2015

Keywords:
Laser induced damage
HF etching
Fused silica
Metallic contamination
Etching rate

ABSTRACT

Laser damage to fused silica continues a main issue of high-power/energy laser systems. HF-based etching technique is known to mitigate laser damage initiation and growth under UV laser illumination. The responses of material surface properties, especially surface damage characteristics to various etching parameters are questioned in the article. Fused silica was submerged into HF-based etchants (HF, NH_4F:HF, HF:HNO_3 with diverse concentrations) in an attempt to improve its laser-induced damage threshold (LIDT). The results have evidenced that the LIDT relies on, to a greater degree, the etched thickness and the etchant composition. The secondary ion mass spectrometer (SIMS) testing was aimed at relating the LIDT to certain metallic contaminant; however, the LIDT exhibits weak direct correlation with Ce, La, Ca, Fe contaminants. The surfaces with the highest LIDT are, more often than not, such that the surface roughness is <10 nm RMS and few metallic impurities are present. In addition, we tried to link the LIDT to the hardness and Young's modulus of fused silica, but no testing data show that there exists direct dependence of the LIDT on hardness and Young's modulus, which are actually independent of the removed thickness.

1. Introduction

Fused silica is used as bulk material in high-power/energy laser systems such as National Ignition Facility (NIF, USA), Laser Mega-Joule (LMJ, France) and SG-III (China) [1–3]. Optical elements may suffer from laser-induced damage in these systems when subjected to 351/355 nm laser pulse irradiation at high fluence. As a result, improving the laser-induced damage threshold (LIDT) of fused silica optics contributes to the enhancement of the overall performance of laser systems [4–9].

Many research groups have been devoted to the understanding of laser damage initiation at 351/355 nm in the past few decades. It is now widely accepted that laser damage is associated with damage precursors such as metal impurities (Ce, Fe, Ca, etc.), subsurface mechanical defects (SSD) and re-deposited substances during etching and/or polishing [5–10]. Among them, metal impurities in the Beilby layer (an inherent layer left by most polishing processes) act as absorbers of UV-light and raise temperature in fused silica. Subsurface defects, including micro-cracks, are responsible for light intensification inside fused silica; moreover, impurities may also be embedded in SSD cracks. Re-deposited substances on bulk surface especially on the edge of scratches or cusps absorb laser light and induce laser damage. The above-mentioned precursors may absorb incident laser light, locally heat fused silica optics and ultimately trigger serious damage when operated at 351/355 nm UV laser. Various methods for improving optics damage resistance have been proposed. Suratwala et al. treated fused silica covered by scratches in HF-based etchants, increasing the LIDT from 7 J/cm^2 to 41 J/cm^2 with ~35 μm material removal [9]. Indentations with micro-cracks on silica surface were iso-thermally annealled at 750 °C to 1150 °C by Shen et al. to heal cracks and inhibit damage initiation [10]. Cormont et al. used CO_2 laser to remove fractures around damage sites and mitigate damage growth under subsequent laser exposure [11]. Of various methods, HF-based solution treatment takes the advantage of globally treating samples by removing absorbing impurities and blunting cracks simultaneously, emerging as a promising method for the LIDT enhancement [6,9]. The chemical etching with different solutions was performed on fused silica samples to find out the possible effects of etching on the LIDT in the article. The etching rate of fused silica in various HF-based

* Corresponding author.
E-mail address: yargolee@163.com (Y. Li).

http://dx.doi.org/10.1016/j.apsusc.2015.09.065
0169-4332/

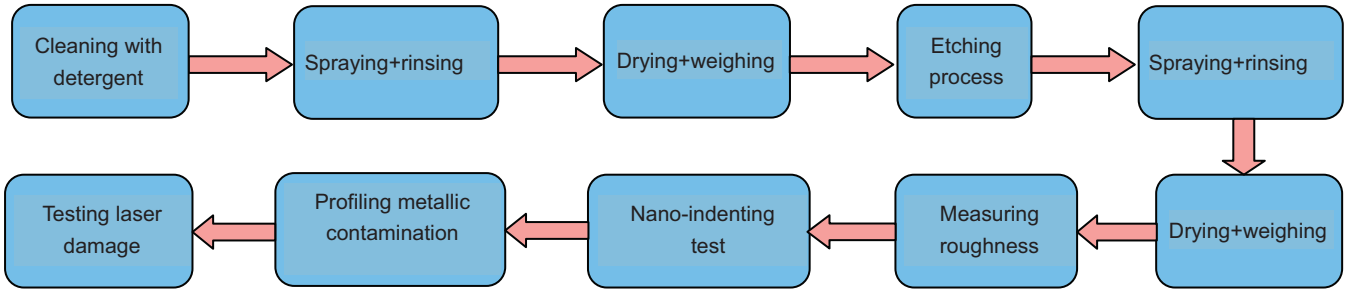

Fig. 1. Flowchart of experimental procedure. Each sample was immersed in a specific solution for a certain time.

solutions was first evaluated and it is found that the rate increases with HF concentration, but not linearly, and the addition of NH_4F will result in sharply elevated etching rate. However, the HNO_3 seems not to benefit the etching rate under experimented conditions. The surface roughness and surface hardness of etched fused silica were also examined and the results show that the etching, in particular deep etching (>5 μm), will roughen surface, but the surface hardness did not vary much with etching. The surface damage threshold which most concerns us can rise or drop depending on many factors, inclusive of the etching thickness, solution composition, surface roughness, metallic contamination, etc., which will be detailed in the following sections.

2. Experimental procedure

2.1. Sample preparation

The fused silica samples (50 mm in diameter, 5 mm thick) were polished on a polyurethane pad with 0.5–1 μm CeO_2 slurry. Samples were sufficiently polished to wipe off the subsurface cracks or fractures originated from grinding steps. Then the samples were ultrasonically cleaned with detergent in the tanks agitated by ultrasonics of 40–270 kHz. The pre-cleaning process comprises ultrapure water spraying and ultrasonic rinsing with 68 kHz and 132 kHz; after rinsing, samples were dried and weighed in a clean room, and then submerged into etchants for preset time. Post-cleaning was identical to pre-cleaning procedure (Fig. 1).

Fused silica optics was treated in nine groups of HF-based etchants for diverse time in our experiments. Group A–D were the pure HF acid with various weight concentrations (A: 1.7%wt. HF; B: 3.4%wt. HF; C: 5.6%wt. HF and D: 11.1%wt. HF); Group E–H were composed of the different mixing ratios of HF and NH_4F (named buffer oxide etchant, BOE) (E: 0.4%wt. HF and 12%wt. NH_4F; F: 1.7%wt. HF and 12%wt. NH_4F; G: 2.8%wt. HF and 12%wt. NH_4F and H: 5.6%wt. HF and 12%wt. NH_4F); Group I was the mixture of 1.7%wt. HF and 6.1%wt. HNO_3. Etching process was carried out in a sealed Teflon container at room temperature and humidity and samples were supported on a Teflon frame at the edges, as Fig. 2 shows.

2.2. Roughness measurement and nano-indentation test

A white light interferometer (NewView 7200, Zygo Corp., USA, ×20 magnification, sampling size ~350 μm × 260 μm, nominal Z-resolution 0.1 nm) was used to measure surface roughness of samples. Average of five measurement points on each sample was taken as the reported result. In addition, the mechanical properties of samples such as hardness (H) and Young's modulus (E) were tested using a nano-indenter (Hysitron TI-950, USA). The low-load Berkovich indenter was used with a constant loading and unloading rate of 1 mN/s.

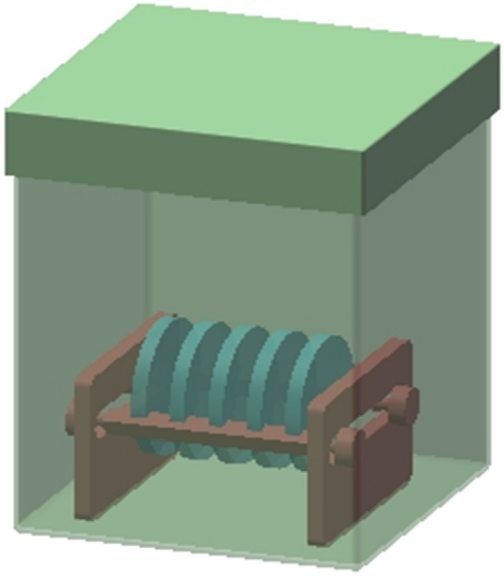

Fig. 2. Schematic of the etching apparatus.

2.3. Laser damage testing

The laser damage testing was performed on the exit surface of each sample using both R:1 and 1:1 protocols. R:1 testing revealed the damage threshold, by ramping the laser fluence incrementally (0.25–2.5 J/cm^2, depending on the surface to be tested) until the damage occurred. In 1:1 testing each test site on the sample was exposed to a single shot, and ~60 sites were tested with various fluences to obtain the 1-on-1 thresholds basically in accordance with ISO-11254 and damage probability after Weibull [12–15]. The laser beam was projected onto the sample with a focusing lens (f = 600 cm). The samples were perpendicularly illuminated by small beam with a diameter of 380 μm at $1/e^2$, 355 nm, 10 ns Gaussian spatial and temporal profile. The damage was monitored by a long-focus microscopy equipped with a CCD camera (resolution power ~10 μm) (Fig. 3).

3. Results and discussion

3.1. The etching rate of silica in HF-based solutions

The etched mass with etching time shows clearly that the etched mass exhibits excellent linear dependence with the etch time (Fig. 4), indicating that the etched mass in each HF solution is extremely steady and predictable in our experiments. The HF concentration was almost unaltered prior to and after the etching, guaranteeing the steady dissolution rate of