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Synchrotron micro-XRF study of metal inclusions distribution and variation in fused silica induced by ultraviolet laser pulses

C.H. Li [a,b], X. Ju [a,*], W.D. Wu [b], X.D. Jiang [b], J. Huang [b], W.G. Zheng [b], X.H. Yu [c]

[a] Department of Physics, University of Science and Technology Beijing, Beijing 100083, China
[b] Research Center of Laser Fusion, CAEP, P.O. Box 919-988, Mianyang 621900, China
[c] Shanghai Synchrotron Radiation Facility, Shanghai 201800, China

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ABSTRACT

Metal inclusions play critical roles in laser-induced damage for large fused silica optics. Here, the spatial distribution of sodium, aluminum, iron and copper in as-prepared samples is analyzed by synchrotron based X-ray fluorescence spectrometry microprobe system at the BL15U1 beam line at the Shanghai Synchrotron Radiation Facility. The as-prepared fused silica samples are induced by 355 nm laser pulses with no, or low, or high fluences. The spatial resolution of the obtained elemental maps is up to 50 μm. Analysis of the elemental maps indicates that the distribution of metals has a close association with the laser fluence and pulses. The normalized fluorescence signal attenuation for metal inclusions corresponds to the laser fluence. The decrement of metals depends chiefly upon the fluence other than pulses of the incidence laser, which is most pronounced for iron and least for copper. The decrement is evident for high fluence laser irradiation, while the amount is negligible for low fluence laser irradiation. Among the four metals, iron concentration is suggested as the most destructive factor for optics lifetime, especially under high fluence irradiation. The quasi-periodic feature of elemental distribution is partly ascribed to laser intensity modulation induced by Fresnel diffraction.

1. Introduction

The increasing requirement for higher power laser output in large laser system such as the Laser Megajoule [1] and National Ignition Facility [2] has led to considerable scientific interest in ultraviolet (UV) laser-induced damage in fused silica optics [3–6]. For nanosecond laser irradiation, most studies about mechanism of laser-material interaction were focused on thermal explosion. Laser-induced damage usually initiates at nano-absorbing centers [7,8] at or near the exit surface. The mechanisms of laser-induced damage initiating at micro-inclusions such as nanoscale platinum or gold particles have been intensively investigated [9,10]. Due to its high absorption coefficient and high thermal conductivity, local concentration of metal inclusions acts as precursor absorbing substances in the damage process [11]. Firstly, the metal inclusions absorb high fluence laser and experience heating, melting, boiling and evaporation. Secondly, once the precursor reaches a critical temperature (the temperature above which the intrinsic bulk silica becomes strongly absorbing), the surrounding silica begins to absorb laser energy. Rubenchik and Feit regarded the laser-induced damage process as a consecutive procedure of laser energy deposition at micro-particles, thermal explosion and plasma fireball growth, and then the formation and growth of the crater [12]. Neauport et al. investigated the correlation between metal inclusions concentration and damage density and concluded that some metals (cerium and lanthanum) played key roles in process of laser energy deposition [13]. Sources of metal inclusions can be both the fabrication process and the operating environment. For instance, aluminum is introduced during the polishing process as alumina is chosen as polishing powder. Critical to elucidate the role of metals in damage process is the knowledge of various elemental maps for samples created under various laser parameters.

To map metals with high resolution, the synchrotron X-ray fluorescence (SXRF) microprobe is employed. XRF is one of the most widely used spectroscopic techniques in elemental identification and quantification [14–18]. When using synchrotron sources to produce the exciting micro-beam, taking advantages of the high intensity, natural collimation, potential tunability, and high degree of linear polarization in the storage ring plane, these advantages offer the possibility to obtain high elemental and chemical sensitivity and high spatial resolution with trace level detection limits and on a microscopic scale [19].

This paper reports a SXRF microanalysis of metal inclusions in fused silica induced by high fluence UV 3ω (355 nm) laser pulses.

* Corresponding author. Tel./fax: +86 010 62333921.
E-mail address: jux@sas.ustb.edu.cn (X. Ju).

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Table 1
Parameters of laser damage test and details of micro-SXRF scanning test.

Sample	Fluence (J/cm^2)	Pulses	Damage results	Scanning area (*X* axis × *Y* axis)	Spot size (*X* axis × *Y* axis)
S1	0	0	Virgin	1 mm × 1 mm	50 μm × 50 μm
S2	20	100	Sub-damage	1 mm × 1 mm	50 μm × 50 μm
S3	25	4	Crater	1 mm × 1 mm	50 μm × 50 μm
S4	40	4	Crater	1 mm × 1 mm	50 μm × 50 μm
S5	65	200	Crater	4.2 mm × 3.7 mm	100 μm × 100 μm

We attempt to study the possible relationship between the variation of metal inclusions distribution and the incident laser fluence and pulses. These mapping results are expected to provide new and useful information concerning the role of metals in the damage process.

2. Experimental

2.1. Samples and laser damage generation

The fused silica samples were 36 mm in diameter and 0.9 mm in thickness, and they were specially polished on both surfaces for high power applications. Alumina was chosen as polishing powder. Prior to damage testing, all samples have been cleaned with de-ionized water to rinse off any particles induced by storage and handling on the optical surface, which was followed by an alcohol rinse with absolute ethanol to remove residual water.

A Nd-YAG laser with the mode lock technique was employed at ambient conditions in this experiment. The wavelength, pulse length (full width at half maximum, FWHM) and laser repetition rate were 355 nm, 6.8 ns and 1 Hz, respectively. The beam profile was near Gaussian with a $1/e^2$ diameter of 0.5 mm at the sample plane. Damage sites were produced exclusively on the laser exit surface with fluences ranging from 20 to 65 J/cm^2.

2.2. Equipment and measurements

SXRF microanalysis was carried out at the BL15U1 beam line of SSRF which is a third generation synchrotron radiation facility with 3.5 GeV energy of electron beam in the storage ring. The synchrotron white beam emerging from the bending magnet source was focused by a pair of Kirkpatrick-Baez-mirrors which provided a beam of 2–100 μm in diameter, and monochromatized with a double-crystal Si (1 1 1) monochromator. Samples were placed on a movable XYZ table whose *X* axis, *Y* axis and height can be adjusted with a reproducible positioning of 0.1 μm. The characteristic X-rays fluorescence signal and the signals needed for normalization, e.g. ionization chamber signals, detector dead time and ring current, were detected with a liquid nitrogen cooled energy dispersive Si drift detector. A 90° geometry between the incident synchrotron

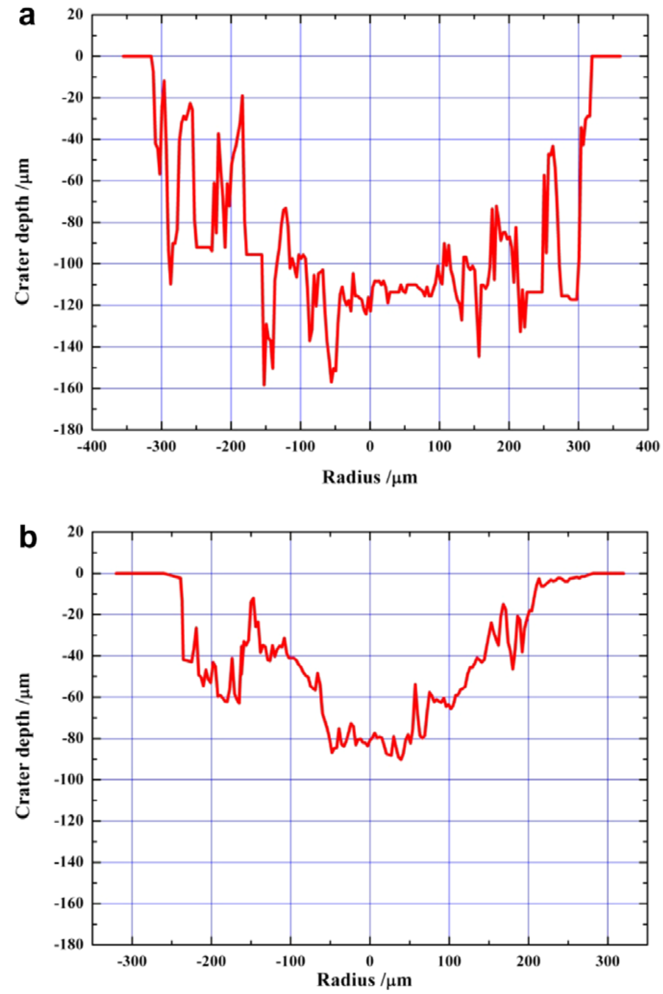

Fig. 2. Depth profiles of craters displayed in Fig. 1, (a) $F = 25$ J/cm^2 and (b) $F = 40$ J/cm^2.

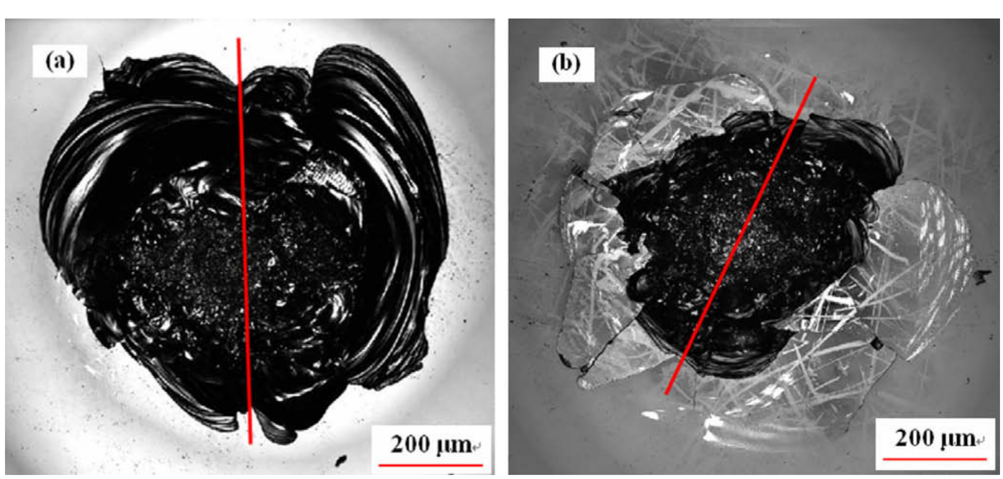

Fig. 1. Laser scanning confocal images of damage craters induced by 355 nm laser by four pulses at $F = 25$ J/cm^2 (a) and at $F = 40$ J/cm^2 (b).