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Fracture Induced Sub-Band Absorption as a Precursor to Optical Damage on Fused Silica Surfaces

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The optical damage threshold of indentation induced flaws on fused silica surfaces was explored. Mechanical flaws were characterized by laser damaged testing, SEM, optical, and photoluminescence microscopy. Localized polishing, chemical etching, and the control of indentation morphology were used to isolate the structural features which limit optical damage. A thin defect layer on fracture surfaces, including those smaller than the wavelength of visible light, was found to be the dominant source of laser damage initiation during illumination with 355nm, 3ns laser pulses. Little evidence was found that either displaced or densified material or fluence intensification plays a significant role in optical damage at fluences >35J/cm^2. Elimination of the defect layer was shown to increase the overall damage performance of fused silica optics.

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(140.3330) Lasers and laser optics: Laser damage

(160.6030) Materials: Silica

(350.1820) Other areas of optics: Damage

The maximum output energy or power available from high energy laser systems is typically limited by the obscuration resulting from high fluence laser-induced optical damage which results from the destructive absorption of sub band-gap light. The growth of even a few sites upon repeated exposure [1] can limit the lifetime of optical components [2,3]. Multi-photon absorption at high optical intensities (I>100GW/cm^2)

determines the ultimate intrinsic damage threshold of any bulk defect-free material. Even the highest quality fused silica optics, however, exhibit localized surface damage well below ($I<10GW/cm^2$) the intrinsic threshold [4]. Special finishing processes [5,6] and/or surface treatments [7] can effectively eliminate photoactive impurities such as Ce [8] from polishing compounds. Great progress has been made in reducing fabrication induced surface flaws [9-12] such as near surface scratches or indentations. It is exceedingly difficult, however, to entirely eliminate all surface quality imperfections although they are thought to dramatically reduce the optical damage initiation threshold. As described below, surface quality flaws contain structural defects which might act as precursors to optical damage. To date, no study has clearly isolated the most important (e.g., lowest threshold) precursors associated with such flaws. It is possible that knowledge of the particular damage initiating precursor could lead to methods to improve the damage performance of optical components without the need to completely remove such flaws.

In this work we have used static indentation to create localized flaws on fused silica surfaces. Such flaws are analogous to those invariably present on even high quality polished surfaces. Once created, flaws were characterized using optical microscopy, secondary electron microscopy (SEM), and confocal time resolved photoluminescence microscopy (CTP) [7,13,14] both before and after high fluence laser damage testing. By varying indentation geometry and load, and through the use of localized polishing and chemical leaching, it was possible to isolate specific structural and mechanical features and determine which features limit the optical damage resistance of high quality fused silica surfaces.

Even casual abrasion or indentations on optical components can result in the creation of localized regions of densified, fractured or plastically displaced material. Due to constructive interference, fractures can create optical field enhancements (FE) which some have suggested are the principal source of surface flaw induced damage [15,16]. Others, however, question if such enhancements are sufficient to reach the intrinsic threshold of fused silica [17]. In addition to fluence intensification, mechanically induced electronic defects [18] are known to absorb sub-band gap light [13,14]. In the present study, we investigated the role of structurally modified material, intensification in sub-surface fractures, and the absorbing properties of fracture surfaces themselves on laser damage initiation.

Arrays of indentations were created on a series of uncoated, 2" diameter, polished fused silica windows (CVI-Melles Groit). Prior to indentation, all samples were chemically leached [7] to remove surface impurities. A series of sharp [19,20] Vickers or Knoop indentations (Fig. 1) were applied to each substrate using loads of 0.1 to 2.0 N via a model HMV2 Shimadzu micro hardness tester. A loading rate of 0.2 mm/s and a dwell time of 60s were used in all cases. Variations in mechanical load, indenter geometry and/or materials properties (i.e. fracture toughness and hardness) result in the transition of a material's response to indentation from pure deformation to deformation plus fracture [19]. In the case of Vickers indentations, nascent fractures were consistently found on the periphery of the contact zone at loads well below (0.1N) that required for initiation of the typical radial and lateral crack systems [20] (see Fig. 1a). For Knoop indentations, peripheral fractures appear at loads between 0.1 and 0.25N (see Fig. 1b,c). Knoop indentations at these loads and for Vickers indentations below 1N, the nascent fractures are typically $<$100nm wide (see Fig. 1d). In contrast, a 2N Vickers indentation results in fractures $>1\mu$m in width.

In addition to fracture, all indentations result in permanent structural modifications due to densification and plastic flow. Material displaced by plastic flow accumulates adjacent to the contact zone [21] as shown in Fig. 1e. Previous studies using CTP suggest that regions exhibiting fast ($\tau<2$ns) PL, such as shown in Fig. 1f, are associated with electronically defective material that strongly absorbs sub-band gap light and is correlated to optical damage [13,14]. Such defects are spatially correlated with regions of both plastic flow and the narrow fractures identified in Fig. 1d. Finally, within the contact zone itself, permanent densification also occurs [21,22].

Such densification is most clearly seen in Fig. 2. Here, Magneto-rheological finishing (MRF) is used to locally polish away silica (Fig. 2). Following 1 μm of removal from a 0.5N Vickers indentation, all surface topography including the indentation, the fracture, and the plastically displaced material has been removed; only densified silica remains. Laser damage testing of prepared samples was performed at λ=355 nm, using a 3 ns wide pulsed beam having a $1/e^2$ diameter of 100 μm. Damage thresholds (ϕ_{TH}) were obtained by incrementally increasing the laser fluence until laser damage was initiated (R/1 damage testing). The estimated uncertainties in ϕ_{TH} are represented by the standard deviation (SDEV) of ~10 measurements on similarly prepared samples.

Table 1 summarizes the experiments performed here and the conclusions which can be drawn from each experiment. After leaching, and in the absence of indentation flaws, the damage threshold of reference surfaces was found to be ≈38J/cm^2. In contrast, a 2N Vickers indentation which contains fractured, displaced and densified material, dramatically reduces the ϕ_{TH} to ≈5 J/cm^2. Similarly the ϕ_{TH} associated with 0.5N Vickers indentations (≈7 J/cm^2) is experimentally indistinguishable from the 2N Vickers indentations. If one assumes fractures must have a width (w) of ≈1/4 the damage testing wavelength (λ) to contribute significant FE, then the similarity of ϕ_{TH} for the 0.5N (w ≈20 nm) and the 2N Vickers indentations (w ≈1000 nm) suggests that FE does not play a significant role in damage initiation. In contrast, the significant increase in ϕ_{TH} (w <100nm) for the 0.5N Knoop indentation suggests that the character of the fracture may play a role in the initiation of optical damage.

As shown in Table 1, the damage threshold dramatically increases when a 0.5N Vickers indentation is polished by MRF (Fig. 2). Such polishing removes all but the densified material. Since ϕ_{TH} again approaches that of the reference surface, densified silica cannot be the limiting structural defect. Futhermore, the 0.1N Knoop indentation contains displaced and densified material, but no fractures. Its threshold is indistinguishable from the reference surface, indicating that neither displaced nor densified material limits the damage threshold, although they may play a role at higher (>35J/cm^2) fluences.

Moreover, unlike the CTP image of the 0.5N Vickers indentation shown in Fig. 1e, the 0.1N Knoop indentation samples have no PL signal. That is, the FPL defects and the most important damage precursors are both correlated with the location of fractures. This is evident in Fig. 3 which shows the effect of a 5 min buffered oxide etch (20:1 BOE) on a 0.5N Vickers indentation. Such an etch removes sufficient material from each face of the fracture to increase the width of the fracture from 20nm to ≈500nm (Fig. 3b). As shown in Table 1 this increases ϕ_{TH} from 7 J/cm^2 to 21 J/cm^2 and reduces the FPL signal from 3400 to 530 counts (see Table 1, and Fig. 3). Also note that after etching, the width of the fracture is sufficiently wide (>100 nm) to provide the opportunity for FE. Since the ϕ_{TH} actually increased, we find no evidence that FE plays a role in damage susceptibility. Rather, the increase in ϕ_{TH} results from the removal of FPL-active defects from the fracture surfaces.

In fact, all of the indentations having a threshold less than the reference surface were found to be spatially correlated with fractures indentified by SEM or optical microscopy (e.g., Fig. 4). Hence, a fracture-induced defect layer <200nm thick represents the optical surface damage limiting structural feature of fused silica. The structural defects studied here mimic static and dynamic indentation induced imperfections typical of high quality fused silica components. Therefore, one expects that this fracture induced layer also limits the surface damage performance of fused silica optical components. The present experiments also suggest that the character of the fracture may play a role in the density of laser damage precursors.

We have identified a thin (<200 nm) fracture-induced defect layer as the optical damage limiting defect associated with silica surface flaws. This defect layer can be eliminated, leaving the structurally flawed material intact, using chemical etching processes. This has lead to the development of a method [23,24] which substantially improves the damage performance of fused silica optics that does not require removal of the structural flaws themselves and does not require explicit knowledge of the location of each of the damage precursors.

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Fig. 1. SEM of (a) 0.1N Vickers indentation, note fracture on periphery of contact zone, (b) 0.1N Knoop, fracture free indentation (c) 0.5N Knoop indentation, note fractures in the periphery of the contact zone (d) SEM and (e) surface profilometry diagonally through a 0.5N Vickers indentation; (f) CTP fast PL image of 0.5N Vickers indentation, the correlation between PL and peripheral fractures.

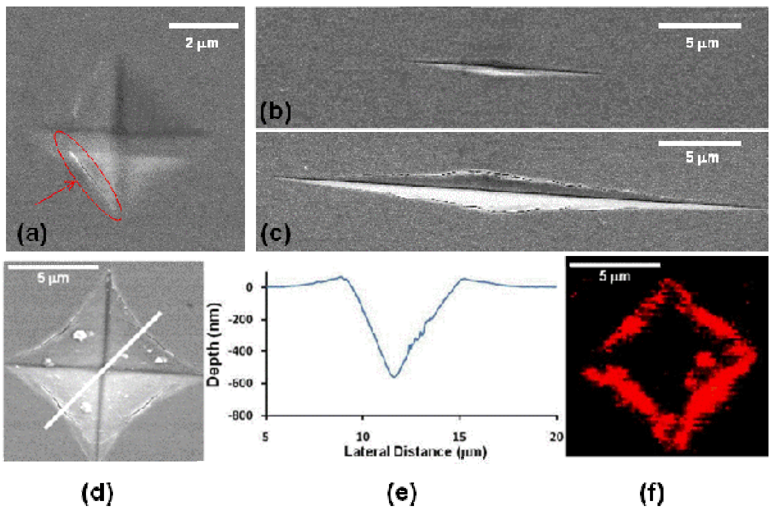


Fig. 2. (a) SEM of 0.5N Vickers indentation and optical micrographs following (b) 0.5μm and (c) 1μm removal by MRF polishing. Note the contrast in (c) indicating densified silica.

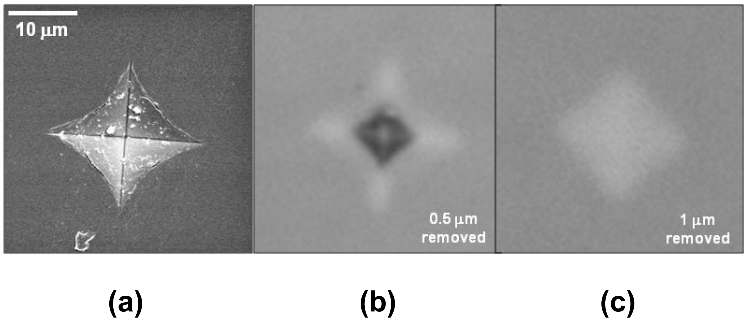
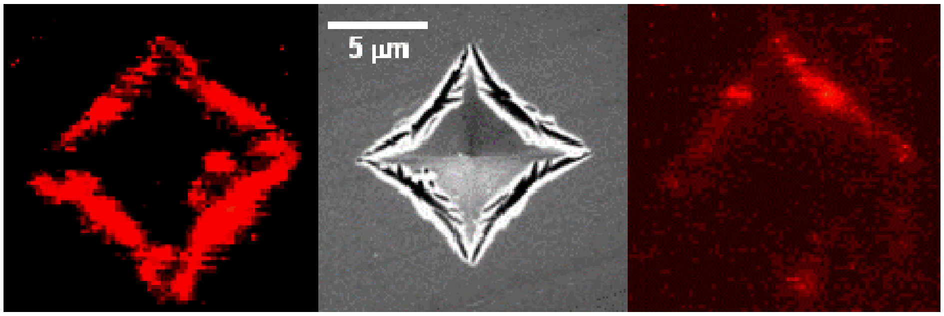


Fig. 3. (a) CTP image of a 0.5N Vickers indent; (b) SEM image after 5min BOE etch; (c) CTP image after 5 min etch. Note the reduction in FPL and increase in damage threshold (ϕ_{TH}).



(a) (b) (c)

Fig. 4. Optical micrographs before and after laser damage testing: (a) 0.5N Knoop indentation, ϕ_{TH} = 9.2 J/cm^2 ; (b) 0.5N Vickers indentation, ϕ_{TH} = 6.9 J/cm^2. The black dots indicate the location of fractures as determined by SEM; compare with Figs. 1, 2a and 3b.

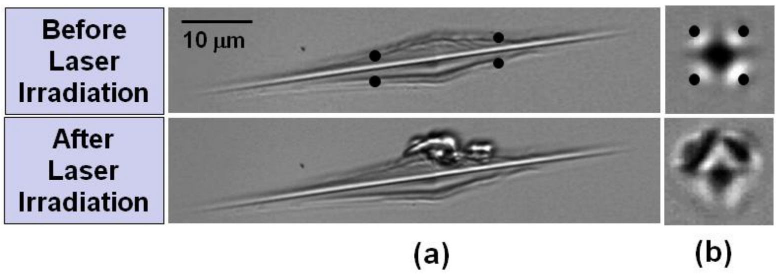


Table 1. Experiments to isolate the damage structural defect which limits optical damage threshold: Field Enhancement (FE) from fractures, plastically displaced (PLASTIC) and densified (DEN) material, fracture-induced defect layers (correlated to peak FPL Counts [13]). All laser damage thresholds (ϕ_{TH}) were measured using R/1 testing. Grey boxes indicate experiments which eliminate the indicated structural defects as the limiting optical damage precursor.

Indentation type and post-treatment	Candidate Precursors				FPL Signal (Counts)	ϕ_{TH} [J/cm2]		Conclusion
	Fracture width	FE	DEN	PLASTIC		ϕ_{TH}	SDEV	
Reference Surface	none	no	no	no	202	38	± 3.7	Baseline Polished Surface
2N Vickers	1000nm	yes	yes	yes	67500	5	± 0.7	Indentation ϕ_{TH} << surface ϕ_{TH}
0.5N Vickers	20nm	no	yes	yes	3400	7	± 2.0	FE is not the limiting precursor; fracture character important
0.5N Knoop	<100nm	yes	yes	yes	2000	14	± 5.1	Fracture character important
0.5N Vickers; followed by MRF	none	no	yes	no	170	29	± 2.2	Densified material does not limit ϕ_{TH}
0.1N Knoop	none	no	yes	yes	500	37	± 2.1	Displaced and densified material are not associated with FPL and do not limit ϕ_{TH}
0.5 N Vickers, then 5 min etch	>100nm	yes	no	no	530	21	± 5.2	Fracture Surface Layer (~100nm) is limits ϕ_{TH} and is associated with FPL