Laser-induced bulk damage in various types of vitreous silica at 1064, 532, 355, and 266 nm: evidence of different damage mechanisms between 266-nm and longer wavelengths

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Laser-induced-damage thresholds (LIDT's) with various types of vitreous silica at 1064, 532, 355, and 266 nm are investigated. At 1064 nm no difference in the LIDT was observed in any sample. At 1064–355 nm the wavelength dependence of the LIDT of synthetic fused silica (SFS) can be described well by the relation $I_{th} = 1.45\lambda^{0.43}$, where I_{th} is the LIDT in J/cm^2 and λ is the wavelength in nanometers. At 266 nm, however, LIDT's were smaller than half of the calculated value from the relation above. This difference can be explained by the damage mechanism; at 266 nm two-photon absorption-induced defects lower the LIDT as in the case of KrF-excimer-laser-induced defects, whereas at longer wavelengths the two-photon process does not occur. LIDT's of fused quartz (FQ) at 532 and 355 nm and that of SFS containing ~1000 ppm of Cl and no OH at 355 nm were a little lower than those of the other SFS's. This lower LIDT may be related to the absorption of metallic impurities in FQ and dissolved Cl_2 molecules in SFS. At 266 nm, on the other hand, LIDT's of FQ's were higher than those of most SFS's. © 1999 Optical Society of America

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1. Introduction

Vitreous silica (v-SiO$_2$) has high optical transmission in a wide wavelength region from the vacuum UV to the near IR and a lower thermal expansion coefficient compared with multicomponent optical glasses.[1] Therefore this material is widely used as a fine optical component. The v-SiO$_2$ is a unique amorphous material that can be used for high-power UV lasers. Recently, this material has been used for industrial excimer laser applications such as semiconductor lithography steppers. However, the absorption and therefore the luminescence is sometimes induced by irradiation with excimer lasers. The mechanisms of the creation of the absorption are dependent on the types of v-SiO$_2$ and their production conditions.

The v-SiO$_2$ can be divided into two groups[1]: fused quartz (FQ) produced by melting natural quartz powder and synthetic fused silica (SFS). SFS contains extremely low metallic impurities compared with FQ. There are two types of FQ: type I and type II FQ produced by electrical melting and flame fusion, respectively. FQ has an optically observable granular structure.[1,2] In addition to the granular structure FQ has an inherent absorption band at ≲300 nm,[1] known as the B_2 band of which at least two kinds exist.[3]

There are several types of SFS. Most optical materials for the UV use type III fused silica produced directly by flame hydrolysis of SiCl$_4$ in an H$_2$/O$_2$ flame. This type of silica contains 500–1500 ppm of OH and is resistant to UV laser irradiation. In optical fiber for telecommunications, SFS produced by soot-remelting methods, such as vapor-phase axial

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deposition (VAD), are used. In the VAD method porous silica is synthesized by H_2/O_2 flame hydrolysis of $SiCl_4$ at a relatively low temperature, ≈1100 °C. Then the porous silica is vitrified at a temperature higher than 1500 °C. Since this method consists of at least two processes, soot forming and vitrification, the amount of $\equiv$SiOH and $\equiv$SiCl can be controlled more easily than the process of type III silica. Recently, a relatively large size of VAD silicas have become commercially available, and some are used in optical components such as lenses and prisms.[4] However, these types of $v\text{-SiO}_2$ cannot be used for excimer lasers, because the intensities of excimer-laser-induced absorption are considerably stronger than those in type III silica.[5] The nature of excimer-laser-induced absorption and emission bands is strongly dependent on the types and the production condition of $v\text{-SiO}_2$.[5–14]

Inasmuch as $v\text{-SiO}_2$ has excellent optical transmission and few defects such as bubbles and inclusions, it can be used as an optical material for high-power lasers. In these applications the laser-induced bulk damage of $v\text{-SiO}_2$ is important. Merkle *et al.*[15,16] studied the single- and the multiple-pulse bulk damage of $v\text{-SiO}_2$ and crystalline quartz at 1064–355 nm. Kitriotis *et al.*[17] studied the multiple-pulse damage of $v\text{-SiO}_2$, crystalline quartz, BK-7, and KCl. Their results showed the difference in the laser-induced-damage threshold (LIDT) between different types of $v\text{-SiO}_2$, as in the case of excimer-laser-induced absorption. It is also expected that the wavelength dependence of LIDT depends on the type of $v\text{-SiO}_2$. Recently we reported a LIDT for various types of $v\text{-SiO}_2$ at 1064, 532, 355, and 266 nm.[18,19] In the range of 1064–355 nm the LIDT of SFS was proportional to the power of a wavelength with a power index of ~0.5. The LIDT's at 532 and 355 nm of FQ and SFS containing 1000 ppm of Cl were slightly less than those of other SFS's. The LIDT at 266 nm is considerably smaller than the value extrapolated by the 0.5th power dependence of SFS at higher wavelengths. The abnormality at 266 nm suggests a different damage mechanism from that at higher wavelengths.

In this paper the origin of these differences in the damage mechanism of these samples is discussed with a comparison of the other properties such as inherent and excimer-laser-induced optical absorption and metallic impurities.

2. Experimental Procedure

A. Samples

The samples used in this study and their concentration of OH, Cl, and metallic impurities are shown in Table 1. They are commercially available $v\text{-SiO}_2$.[4] Samples ES, ED-A, ED-H, ED-B, and ED-C are of SFS; OX and HR are of FQ. ES is produced directly by flame hydrolysis of $SiCl_4$ in H_2/O_2 flame. Samples ED-A, ED-B, ED-C, and ED-H are produced by the VAD method[1] and contain various amounts of OH and Cl as shown in Table 1. OX and HR are produced by melting natural quartz powder in a H_2/O_2 flame and an electric furnace, respectively. These samples were cut into a size of 4 cm × 4 cm × 3 cm, and all the surfaces were optically polished.

Table 1. Impurities of Samples Used in the Present Study[a]

	Sample						
Impurities	ES	ED-A	ED-H	ED-B	ED-C	OX	HR
OH	1200	90	40	<1	<1	150	10
Cl	40	<1	<1	<1	1000	<1	<1
Al	<0.1	<0.01	<0.01	<0.01	<0.01	20	16
Na	<0.05	<0.01	<0.01	<0.01	<0.01	1.1	0.9
K	<0.05	<0.01	<0.01	<0.01	<0.01	1.0	0.7
Li	<0.05	<0.01	<0.01	<0.01	<0.01	1.0	0.5
Ca	<0.05	<0.01	<0.01	<0.01	<0.01	0.4	0.5
Fe	<0.05	<0.01	<0.01	<0.01	<0.01	0.3	0.2
Cu	<0.05	<0.01	<0.01	<0.01	<0.01	0.01	0.03

[a] In parts per million.

B. Measurement of the Laser-Damage Threshold

The damage threshold of the $v\text{-SiO}_2$ was measured at laser wavelengths of 1064 nm (pulse width, τ_p of 1.1 ns), 532 nm ($\tau_p = 0.95$ ns), 355 nm ($\tau_p = 0.85$ ns), and 266 nm ($\tau_p = 0.75$ ns). The output from a single-mode *Q*-switched Nd:YAG oscillator was amplified by two amplifiers with 10-mm apertures and frequency upconverted by KDP and CLBO crystals. The amplified laser pulse was focused with a lens of 10-mm focal length. The focused laser beam had a smooth Gaussian shape with a spot diameter with a $1/e^2$ peak intensity of 30–40 μm. The laser energy was adjusted with a combination of a half-wave plate and dielectric polarizer without changing the pumping energies of the amplifiers so as not to change the beam shape of the sample. The laser energy and the pulse shape were monitored with biplanar phototubes that had been calibrated with a standard calorimeter. The damage was defined to be any visible change in the laser-irradiated site by measurement of the scattering light under the dark field. The irradiated site on the sample was moved after each laser shot regardless of the presence or absence of damage.

3. Results

Figure 1 shows the wavelength dependence of the LIDT. The values of the LIDT for the silica glass samples are also shown in Table 2. At a 1064–355-nm wavelength no difference in LIDT can be seen for SFS except for ED-C at 355 nm. We found that the LIDT is proportional to the 0.43 rd power of the wavelength. The relation can be expressed as

$$I_{\mathrm{th}} = 1.45\lambda^{0.43}, \tag{1}$$

where I_{th} is the LIDT in J/cm^2 and λ is the wavelength in nanometers. The LIDT's of FQ at 532 and 355 nm are slightly lower than those of SFS that are calculated from Eq. (1). At 355 nm the LIDT of ED-C is lower than that of the other SFS's, and the LIDT's of the FQ's were lower than that of ED-C. At

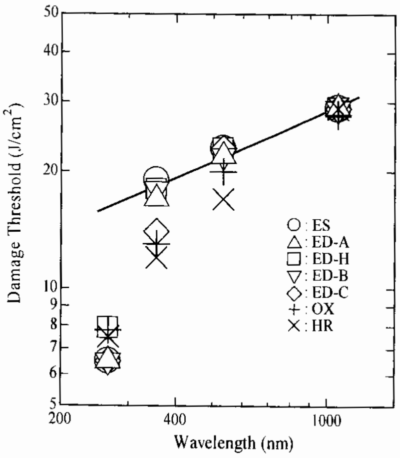


Fig. 1. Wavelength dependence of LIDT.

266 nm the LIDT's of all the samples are considerably lower than that of the calculated value from Eq. (1). The LIDT's of ED-H and FQ are approximately half of the calculated value, and those of the other SFS's are ≈2/5 of the calculated value. Figure 2 shows the transmission spectra of the samples in the UV region. ED-B has a strong absorption band at 247 nm (with photon energy of 5.02 eV). OX and HR also have absorption bands at almost the same wavelength (≈245 nm). At a wavelength shorter than this absorption band, the transmissions of the FQ's are considerably smaller than those of the SFS's. Increased absorption in the FQ may be caused by metallic impurities.

Table 2. LIDT of v-SiO$_2$

Samples	Wavelength 1064	 532	 355	 266
ES	29 ± 2	23 ± 2	19 ± 2	6.5 ± 0.5
ED-A	29 ± 2	22 ± 2	17 ± 2	6.5 ± 0.5
ED-H	29 ± 2	23 ± 2	18 ± 2	7.9 ± 0.6
ED-B	29 ± 2	22 ± 2	18 ± 2	6.5 ± 0.5
ED-C	29 ± 2	23 ± 2	14 ± 1	6.5 ± 0.5
OX	28 ± 2	20 ± 2	13 ± 1	7.8 ± 0.6
HR	29 ± 2	17 ± 2	12 ± 1	7.5 ± 0.6
Calculated[a]	29	22	18	16

[a] Calculated value from Eq. (1).

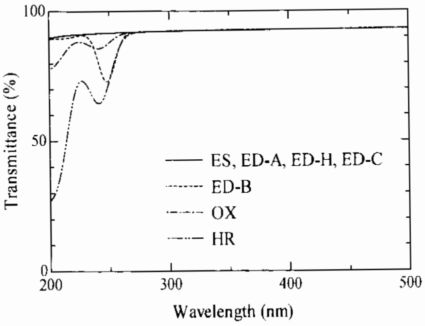


Fig. 2. Transmission spectra of samples used in the present study. The thicknesses of the samples are 1 cm.

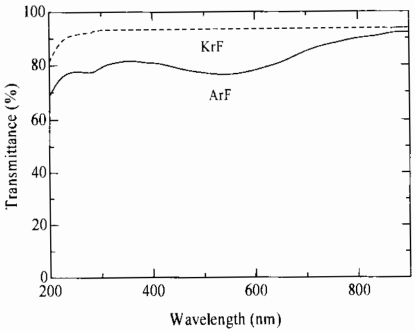


Fig. 3. Transmission spectra of annealed OX irradiated with KrF and ArF lasers (100-mJ/cm^2 pulse, 50 Hz, 10^4 shots). The sample was annealed at 1150 °C for 1 h.

4. Discussion

A. Laser-Induced-Damage Threshold at 1064–355 nm in Synthetic Fused Silica

At 1064 nm no difference was observed for any sample. This suggests that the LIDT is independent of impurities and defect structures in v-SiO$_2$.

At 1064–355 nm the LIDT's of SFS's can be expressed by Eq. (1) except for ED-C at 355 nm. This result is consistent with the result by Merkle *et al.*,[15] the LIDT's of two kinds of SFS and crystalline quartz were the same within experimental error at 1064–355 nm. The result suggests that the damage mechanisms of these SFS's are independent of the existence of the defect structures and impurities in the SFS's at these wavelengths.

B. Laser-Induced-Damage Threshold of Fused Quartz at 532, 355 nm

OX and HR have no internal absorption at 532 and 355 nm (see Fig. 2). However, when the samples are irradiated with an x ray[1,2] and an ArF laser, the color of the samples changes to smoky brown and internal absorption is induced in a wide wavelength region. Figure 3 shows the transmission spectra of OX irradiated with KrF and ArF excimer lasers after being annealed at 1150 °C for 1 h. By irradiation with an ArF laser, the internal absorption was induced in a wide wavelength region from the UV to the visible (200–800 nm). We also irradiated the KrF and the ArF excimer laser beams on OX before annealing, but such internal absorption was not induced. Similar absorption is induced by irradiation with an x ray.[1] Okada *et al.*[20] reported the creation of similar absorption in type I FQ (the same type as HR) irradiated with a KrF excimer laser. They also irradiated a KrF laser with type II FQ (the same type as OX). However, no such apparent absorption band was observed. Friebele and Higby[21] reported the creation of similar absorption spectra in FQ irradiated with an electron beam. These absorption bands were ascribed to being derived from metallic impurities.[21]

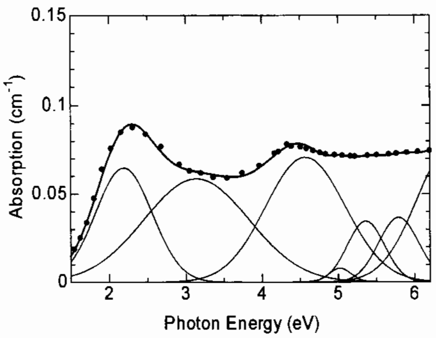


Fig. 4. Result of the Gaussian peak decomposition of the difference spectra of ArF- and KrF-excimer-laser-induced absorption spectra in the annealed sample of OX shown in Fig. 3.

They analyzed induced absorption spectra by the Gaussian peak decomposition.

We also tried to fit the absorption spectra by Gaussian absorption bands. The result is shown in Fig. 4. The spectrum shown in Fig. 4 is the difference spectrum between ArF- and KrF-laser-induced absorption. Peak positions and FWHM are shown in Table 3. The absorption components at 6.5, 5.8, 5.4, and 5.0 eV are absorption spectra originating from the defect structure of silica glass, which are observed in SFS's irradiated with excimer lasers,[5,11] x ray,[11] and γ ray.[22] Absorption bands at 4.6, 3.1, and 2.2 eV are not observed in the SFS's. Friebele and Hygby[21] showed similar peaks in a FQ irradiated with an electron beam. They reported that the induced absorption spectra were reproduced by seven absorption bands at 2.2, 2.9, 4.6, 5.4, 5.8, 6.5, and >6.5 eV. The peak at 3.1 eV with a FWHM of 1.6 eV in the present samples is similar to the A3 center in the crystalline quartz ascribed to the Al related center.[21] The 2.2-eV band is analogous to the Al center in crystalline quartz whose origin is unknown.[21]

As described above the creation of absorption in OX is promoted by annealing. Therefore the LIDT of OX becomes lower with the creation of absorption due to the heat effect by irradiation with 532- and 355-nm lasers similar to the experimental results mentioned above. The LIDT of OX at 532 nm is higher than that of HR, suggesting that the creation of internal absorption at 532 nm in HR is easier than that in OX. The difference of the LIDT between OX and HR is consistent with the experimental result of Okada *et al.*[20] mentioned above. At 355 nm, the LIDT of OX is almost the same as that of HR, suggesting that the intensity of 355-nm-induced absorption in both FQ's at 355 nm has approximately the same values.

Table 3. Peak Positions and FWHM of the Absorption Component in Fig. 4

Peak (eV)	FWHM (eV)	Intensity (cm^{-1})
2.19	0.87	0.065
3.14	1.62	0.059
4.57	1.23	0.071
5.02	0.34	0.008
5.36	0.57	0.035
5.79	0.62	0.037
6.5	1.1	0.078

C. Laser-Induced-Damage Threshold of ED-C at 355 nm

The LIDT of ED-C at 355 nm is lower than that of the other SFS's, perhaps because there are dissolved Cl_2 molecules in v-SiO_2, which have a peak value at 325 nm[23]; the absorption of Cl_2 lowers the LIDT of ED-C at 355 nm.

D. Laser-Induced-Damage Mechanism at 266 nm

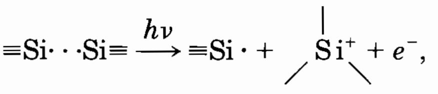
At 266 nm the LIDT of OX, HR, and ED-H is $\approx$8 J/cm^2 and that of the other SFS's is $\approx$6.5 J/cm^2, which is approximately a half and two-fifths of that calculated from Eq. (1), 16 J/cm^2. This discrepancy between the extrapolated value from Eq. (1) and the experimental results suggests that the laser-induced-damage mechanism at 266 nm is different from that of other wavelengths.

One might consider that the reason the LIDT at only 266 nm is considerably smaller than the calculated value from Eq. (1) is derived from inherent absorption at $\lesssim$300 nm. However, this mechanism must be ruled out, because the LIDT of FQ's is higher than those of SFS's in spite of FQ's having inherent absorption at $\lesssim$300 nm.

Arai *et al.*[24] studied the creation mechanism of a point defect called the E' center ($\equiv$Si $\cdot$) induced by excimer laser irradiation. Defects that are induced by KrF [248 nm (5.0 eV)] and ArF (193 nm [6.4 eV]) excimer lasers are produced through a two-photon process. However, such defects were not induced by irradiation with an XeCl excimer laser [308 nm (4.1 eV)],[24] because the photon energy of the XeCl laser is smaller than half of the energy bandgap of amorphous SiO_2 ($\approx$9 eV).[24] At wavelengths shorter than half of the bandgap energy, 4.5 eV (280 nm), the laser damage must have occurred in the two-photon process. Therefore the laser induced damage at 266 nm mainly occurs in the two-photon process.

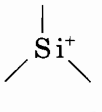
The LIDT of FQ is higher than that of the SFS's. A possible reason for this difference is the difference in Al content between FQ's and SFS's; the Al contents of FQ's are $\approx$20 ppm and those of SFS's are <0.1 ppm (see Table 1). Al is incorporated into the SiO_2 glass network as a form of Al_2O_3. This structure makes the silica glass network stronger. For example, the viscosity of v-SiO_2 is higher when it is incorporated into Al_2O_3.[25] Another interpretation of the difference is that the LIDT of FQ's and ED-H is almost an ideal material compared with the other SFS's, because the LIDT of the former is almost one-half of the calculated value from Eq. (1), but the LIDT of the latter is considerably lower than one-half of the calculated value. Since ED-H produced by vitrifying silica soot is more compact than the other ED-series samples, the structure of the glass network should be more stable than the other ED-series materials. The other SFS's could contain unstable structures. However, we cannot see such structures from chemical analysis and optical observations. Evidence that the structure of ED-H is more stable than the other SFS's is the irradiation time dependence of excimer-laser-induced and x-ray-induced absorption. The rate of the increment of the absorption in ED-H is considerably less than that of the other SFS's.[26]

As can be seen from Fig. 2, the transmittance of FQ's at ≲300 nm is smaller than those of SFS's. The apparent absorption bands in FQ are at 240–250 nm. Such a band is called a B_2 band.[3] There are at least two kinds of B_2 band, called $B_2\alpha$ and $B_2\beta$ bands.[3,27] The $B_2\alpha$ band is caused by an oxygen deficient center ($\equiv$Si· ·Si$\equiv$). The peak position and the FWHM of the $B_2\alpha$ band are 5.02 eV (247 nm) and 0.35 nm, respectively. The absorption band in ED-B is the $B_2\alpha$ band.[26] When the $B_2\alpha$ band is excited, a strong emission band at 4.4 eV (280 nm) and a relatively weak emission band at 2.7 eV (450 nm) are induced.[27] The 2.7-eV band is observed as cyanic blue. In fact cyanic-blue luminescence was observed when the 266-nm laser was irradiated on ED-B. The peak position and the FWHM of the $B_2\beta$ band, on the other hand, are 5.17 eV (240 nm) and 0.42 eV, respectively.[27] When this band is excited, emission bands at 4.3 eV (290 nm) and 3.2 eV (390 nm) are induced.[27] When the 266-nm laser was exposed on OX and HR, violet luminescence appeared. The peak position of the B_2 band in FQ is ≈240 nm as seen from Fig. 2. These facts suggest the existence of the $B_2\beta$ band in these samples. Kuzuu and Murahara[9] showed, by comparing the peak positions and FWHM's of the B_2 bands, that OX has mainly a $B_2\beta$ band and a type I FQ (the same type as HR) has mainly a $B_2\alpha$ band. They also showed that the B_2 band in OX can be easily annealed out but that in the type I silica it cannot be annealed out. In the present case, however, both OX and HR show violet luminescence when irradiated with the 266-nm laser. Therefore we speculate that OX has only a $B_2\beta$ band and that HR has both $B_2\alpha$ and $B_2\beta$ bands. Irradiating with a UV photon near 5 eV can easily change the $B_2\alpha$ band into the E' center ($\equiv$Si ·), causing an absorption band at 5.8 eV (215 nm) to be[3]



$$\equiv\text{Si}\cdot\cdot\text{Si}\equiv \xrightarrow{h\nu} \equiv\text{Si}\cdot + \text{Si}^{+} + e^{-}, \qquad (2)$$

where



Si^{+}

is a planar-three-oxygen-coordinated structure. In fact the absorption band at 247 nm decreased, and the strong absorption band at 215 nm increased from irradiation with a KrF laser as shown in Fig. 5. From irradiation with a 266-nm laser beam a B_2 band in OX, HR, and ED-B can disappear immediately with the aid of heat and/or a photon of a 266-nm laser beam of high power. This mechanism is supported by the fact that the LIDT of the ED-B is the same as that of ES, ED-A, and ED-C, which have no B_2 band.

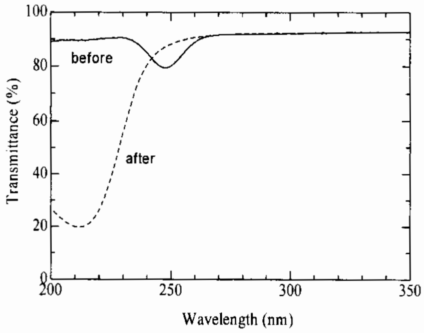


Fig. 5. Transmission spectra of ED-B before and after irradiation with a KrF laser (400-mJ/cm^2 pulse, 100 Hz, 10^6 shots).

5. Summary and Conclusion

We have studied the LIDT of various types of v-SiO$_2$ at 1064, 532, 355, and 266 nm. At 1064 nm we observed no difference between samples. At 1064–355 nm the LIDT's of SFS's were the same value at each wavelength and were proportional to the 0.43 rd power of the wavelength except for that of ED-C at 355 nm. At 266 nm the LIDT of all samples was lower than half of the calculated value from the 0.43 rd power relation. This lower threshold suggests different damage mechanisms from those at longer wavelengths; at 266 nm the damage occurs through two-photon absorption, whereas at longer wavelengths the two-photon process does not occur. The LIDT's of FQ's at 532 and 355 nm are slightly smaller than those of SFS's. This difference may be interpreted by the effect of metallic impurities contained in v-SiO$_2$. A sample containing ≈1000 ppm of Cl and no OH has a slightly lower LIDT than that of the other SFS at 355 nm. This lower LIDT may be related to the Cl$_2$ molecules dissolved in the silica glass network. At 266 nm the LIDT of FQ's is higher than most SFS's.

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