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ArF-excimer-laser-induced emission and absorption bands in fused silica synthesized in reducing conditions

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ArF-excimer-laser-induced luminescence and absorption bands in type-III fused silica synthesized in a reducing condition were investigated. The fused silica shows 4.4-eV emission and 5.8-eV absorption bands ascribed to be the E' center ($\equiv$Si$\cdot$). By annealing in various atmospheres, the creation of these bands is suppressed: The order of the suppression effect is as $\mathrm{He} \cong O_2 > \mathrm{air} > \mathrm{N}_2$. The 5.8-eV band decays after cessation of the irradiation. To describe these phenomena we proposed a model that the precursor of these bands is the $\equiv$Si—H H—O—Si$\equiv$ structure: By irradiating with an ArF-excimer-laser beam, the E' centers are created as $\equiv$Si—H H—O—Si$\equiv$ $\rightarrow \equiv$Si$\cdot$ H—O—Si$\equiv$ +H, and the E' center decays after cessation of the irradiation by recombining with the hydrogen. By annealing, the structures are changed by the reaction $\equiv$Si—H H—O—Si$\equiv \rightarrow \equiv$S—O—Si$\equiv$ +H_2. This reaction is enhanced by removing the hydrogen molecules by annealing in helium and oxygen.

I. INTRODUCTION

Synthetic fused silica has high optical transmission in the uv region. Because of this optical property, it is widely used in uv lithography such as photomasks and lenses.

There are several processes used to produce vitreous silica:[1-3] fused quartz produced by melting natural quartz powder,[1] soot processes such as VAD,[4] MCVD,[5] and OVD,[6] plasma torch CVD,[7] the sol-gel method,[8] the β-Cristobalite method[9] and wet fused silica synthesized directly by flame hydrolysis of silicon tetracholoride in a hydrogen-oxygen flame (type III).[10] The type-III fused silica is widely used in uv lithography, especially in repeat-and-step projectors (stepper) lenses,[11] because we can get a material with good optical homogeneity over a large area.

The type-III fused silica is highly radiation resistive, but color centers may be created in some of the material by γ rays,[12,13] x rays,[14,15] rf plasma,[16] and excimer laser beams.[17-25] These centers are unfavorable because the color centers cause absorption and luminescence in the uv region.

The nature of the solarization is strongly affected by the history of the reaction conditions, such as temperature, and the ratio of chemical species in the flame.[26,27]

A well-known color center in type-III fused silica is related to an absorption band at 4.8 eV (260 nm)[14,15,17-20] which corresponds to a red luminescence band at 1.9 eV (650 nm).[14-17] The center is undesirable for KrF excimer laser optics since the photon energy of the absorption band is close to that of the KrF excimer laser [5.0 eV (248 nm)].

Creation of the 4.8-eV absorption band is a prominent effect in a fused silica synthesized in oxidizing conditions. In fused silica synthesized in O_2 plasma, a very strong 4.8-eV absorption band is induced by excimer laser irradiation.[25] A type-III fused silica synthesized in oxidizing conditions also shows the solarization.[25,26]

Creation of the 4.8-eV absorption band is prevented by annealing the material in hydrogen ambient.[26] But the solarization will appear again by annealing successively in air.[27] This fact suggests a possibility that the material improved by annealing in hydrogen would show the solarization again if irradiated by an excimer laser for a long period of time, i.e., several months or more.

Another way to produce an excimer laser resistive fused silica is to synthesize the fused silica under reducing conditions. The material seems to be stable since the 4.8-eV absorption band does not appear if irradiated by a KrF excimer laser. Therefore we can obtain a KrF excimer laser resistive material by synthesizing in reducing conditions.

However, in fused silica synthesized in reducing conditions, an absorption band at 5.8 eV (215 nm) and an emission band at 4.4 eV (280 nm) are induced by ArF excimer laser irradiation.[24] This solarization is unfavorable in ArF excimer laser optics since the tail of the absorption band will affect the transmission of the laser beam.

We studied in this paper the effect of annealing in various atmospheres on these bands to clarify their origin and to find a process to obtain an ArF-excimer-laser-resistive material.

II. EXPERIMENTAL

A. Sample

A fused silica was synthesized directly by flame hydrolysis in a hydrogen-oxygen flame in reducing conditions.[27] The flame was hydrogen rich, i.e., $[\mathrm{H}_2]/[\mathrm{O}_2]$

>2. The sample contains about 800 ppm of OH in weight which was calculated by ir absorption at 3650 cm^{-1} whose extinction coefficient is 77.5 l / mol cm.[28] The sample was cut into pieces of size $10\times10\times30$ mm^3, and the surfaces polished.

B. Luminescence and absorption measurement

The system of luminescence measurement is shown in Fig. 1(a). A Questek SQL 2240-type excimer laser was used, and an ArF laser was utilized in the present study.

The energy density was controlled by using a fused silica lens ($f=500$ mm). The diameter of the beam was adjusted to 3 mm by using an iris.

The luminescence was detected from the direction perpendicular to the excitation beam. Emission spectra were measured shot by shot monochromatically (using a Jasco CT-10) by diode array image sensor with an image intensifier (Hamamatsu Photonics C2808-03). Spectra bandwidth of each measurement was about 100 nm. Obtained data was processed by a microcomputer (NEC PC-9801mv).

Transmission of ArF excimer laser beam was measured by the system shown in Fig. 1(b). Laser power was monitored by two beam splitters before and after transmitting the sample. The transmission was obtained by the fraction of these two intensities. Excimer laser beam were exposed on a region without irradiated before.

Absorption spectra were measured by a spectrophotometer (Shimadzu UV-250).

C. Annealing

Samples not annealed nor irradiated were annealed at 900 °C for 2 h in a tube furnace with a fused quartz inner tube. Flowing gases was used during the whole annealing period. The annealing atmospheres were air, oxygen, nitrogen, and helium.

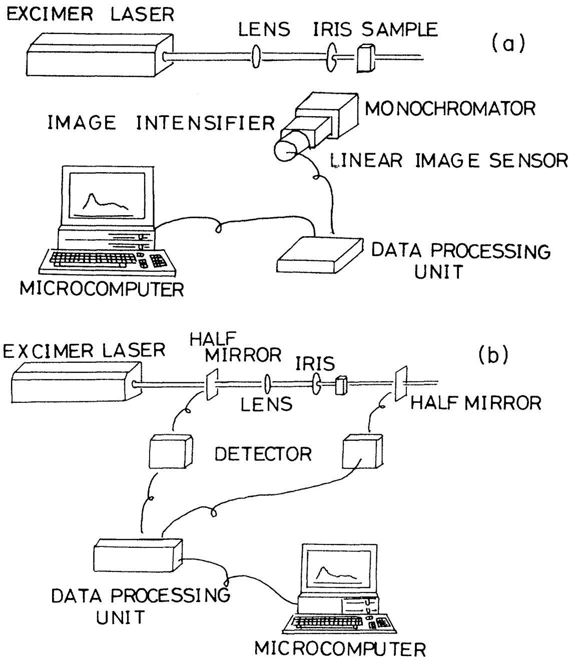

FIG. 1. Schematic drawing of the luminescence measurement system (a) and the excimer laser transmission system (b).

III. RESULTS AND DISCUSSION

A. Luminescence spectrum

An example of the luminescence spectrum of fused silica synthesized in a reducing condition irradiated with the ArF excimer laser is shown in Fig. 2. A strong peak is seen at 4.4 eV.

Shot-number dependence of the luminescence spectra near 4 eV before and after annealing are shown in Fig. 3. Before annealing, the intensity of the 4.4-eV band increases as the shot number of the ArF excimer laser increases. As shown in Fig. 4, the intensity of the luminescence increases almost linearly with time up to 1000 shots and then tends to saturate with larger shot numbers.

B. Annealing effect on the luminescence spectra

Figure 3 shows the effect of annealing in nitrogen, air, oxygen, and helium at 900 °C on the shot-number dependence of the 4.4-eV emission band. By annealing, the 4.4-eV band is diminished and a new emission band is created at 4.1 eV. Opposite to the 4.4-eV band, the 4.1-eV band decreases as the shot number of the ArF excimer laser increases. Therefore, these two bands are derived from quite different origins.

It might be suspected that the peak positions of the emission spectra shown in Fig. 3 shifted by the annealing and irradiation. However, any of these luminescence curves $h(\lambda)$ can be expressed by the linear combination of two spectra:

$$h(\lambda)=af(\lambda)+bg(\lambda)\ , \qquad (1)$$

in which λ is the wavelength, and $f(\lambda)$ and $g(\lambda)$ are any independent luminescence spectra of the present sample near by 4 eV. Let $f(\lambda)$ be the normalized spectra of be-

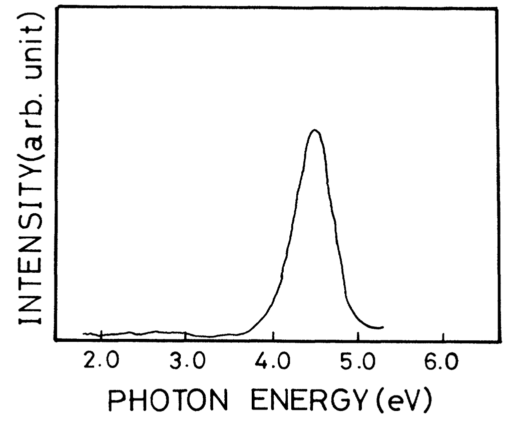

FIG. 2. Luminescence spectrum of type-III fused silica synthesized in reducing condition irradiated with ArF excimer laser beam (100 Hz, 200 mJ / cm^2, 6000 shots).

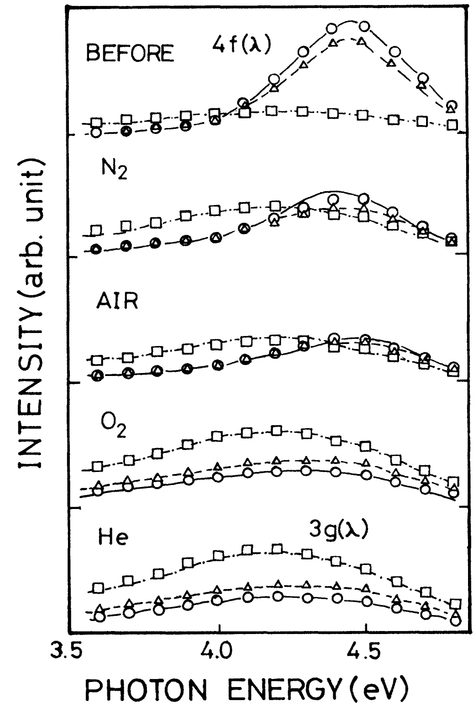

FIG. 3. Shot-number dependence of luminescence spectra induced by ArF laser irradiation (100 Hz, 200 mJ/cm^2) of the fused silica synthesized in the reducing condition before and after annealing: solid lines, 6000 shots; dashed line, 3000 shots; chain line, 100 shots. There are also plotted the calculated values from Eq. (3): □; 100 shots, △; 3000 shots, ○; 6000 shots.

fore annealing at 6000 shots and $g(\lambda)$ be the normalized spectra of after annealing at 100 shots, any spectra shown in Fig. 3 can be represented by Eq. (1). The calculated values are plotted in Fig. 3. They agree very well with the original curves. Calculated values of the parameters a and b are listed in Table I. The shot-number dependence of the 4.1- and 4.4-eV bands are shown in Fig. 4. In this paper we will only deal with the 4.4-eV emission band since the origin of the 4.1-eV band could not be clarified in the present study.

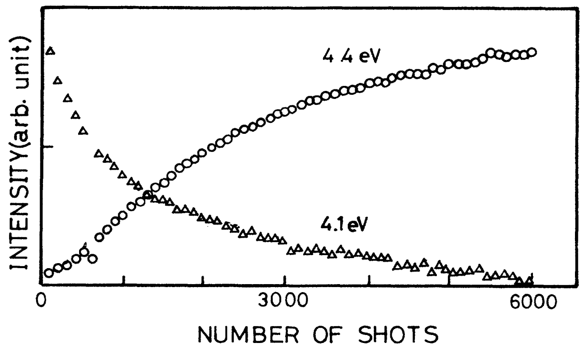

FIG. 4. Shot-number dependence of the intensities of 4.1- and 4.4-eV bands in a sample before annealing calculated by Eq. (1).

Before annealing, the intensity of the 4.4-eV band increases with increasing shot number. After annealing, the creation of the band is suppressed. Annealing in oxygen, the 4.4-eV band is almost extinguished. Annealing in air and nitrogen, the intensity of the 4.4-eV band is also suppressed. The suppression effect on the creation of the 4.4-eV band is on the order of $O_2 >$ air $> N_2$. This fact suggests that the suppression effect on the creation of the 4.4-eV band is mainly caused by a thermal effect, but the oxygen in the atmosphere promotes the suppression.

By annealing in helium, an inert gas, the suppression effect on the creation of the 4.4-eV band is similar to that of oxygen.

C. Induced absorption

The absorption spectrum of the fused silica irradiated with an ArF excimer laser is shown in Fig. 5. An absorp-

TABLE I. Calculated parameters a and b in Eq. (1).

Treatment	Shot number	Parameters (arb. unit) a	Parameters (arb. unit) b	Fraction (%) $a/(a+b)$	Fraction (%) $b/(a+b)$
Before	100	0.068	0.702	8.8	91.2
	3000	3.312	0.097	97.4	2.6
	6000	4.000	0.000	100.0	0.0
N_2	100	0.084	1.707	4.7	95.3
	3000	1.240	0.615	66.8	33.2
	6000	1.680	0.558	75.1	24.9
air	100	0.064	1.452	4.2	95.8
	3000	1.048	0.546	65.7	34.3
	6000	1.148	0.525	68.6	31.4
O_2	100	0.100	2.697	3.6	96.4
	3000	0.456	1.449	23.9	76.1
	6000	0.276	1.152	19.3	80.7
He	100	0.000	3.000	0.0	100.0
	3000	0.304	1.611	15.9	84.1
	6000	0.180	1.209	13.0	87.0

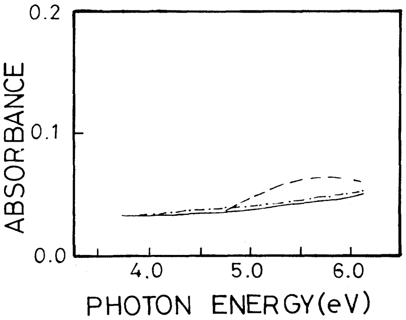

FIG. 5. Absorption spectra induced by ArF excimer laser irradiation (50 Hz, 500 mJ/cm^2, 6000 shots) before and after annealing in He: solid line, before irradiation; dashed line, before annealing; chain line, after annealing.

tion band appears at 5.8 eV (215 nm). This band is ascribed to be the E' center ($\equiv$Si$\cdot$).[29,30] Some authors suggest that the 4.4-eV emission band is related to the 5.8-eV absorption band. As seen from Fig. 5 the 5.8-eV band is extinguished by annealing in helium ambient.

The effect of the annealing on the solarization can be seen clearly by the excimer laser transmission as shown in Fig. 6. Before annealing, the transmission decreases with increasing shot number. But this decrease of the transmission can be annealed out with oxygen and helium.

We can obtain an ArF excimer laser resistive fused silica, i.e., without solarization and luminescence, by the following process.

In the first step, an ingot of fused silica is synthesized in a reducing condition. Then in the second step, the fused silica is annealed in helium or the other appropriate atmosphere to remove the precursor of the 4.4-eV and the 5.8-eV band.

The best annealing gas is helium. As shown in Sec. III B, the efficiency of the suppression of the creation of the 4.4-eV band in oxygen and helium gas is better than in the other gases. However, oxygen is considered to be the origin of the 4.8-eV solarization and red luminescence at 1.9 eV.[25] Therefore oxygen gas is inappropriate to obtain an excimer laser resistive fused silica. It is noted that the intensity of the red luminescence and the 4.8-eV absorption band are still smaller than the fused silica synthesized in oxidizing conditions even after annealing in oxygen[24] (see Fig. 7).

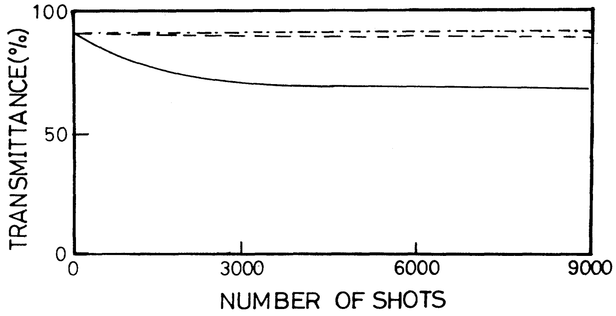

FIG. 6. Effect of annealing on the shot-number dependence of the ArF excimer laser (50 Hz, 100 mJ/cm^2, 6000 shots) transmittance before and after annealing in helium and oxygen: solid line, before annealing; dashed line, oxygen; chain line, helium.

D. Decay of the 5.8-eV band after cessation of the irradiation

It can be seen from Fig. 6, after irradiation with 6000 laser shots that the transmission decreases to about 60% of the initial value. The value corresponds to about 0.2 in absorbance. However, internal absorption, i.e., the difference between the absorbance of before and after irradiation, at 200 nm (6.2 eV) in Fig. 5 is only about 0.02. The internal absorbance at the photon energy of the ArF excimer laser (6.4 eV) is considered to be slightly smaller than absorbance at 6.2 eV, but the same order can be seen from Fig. 5, because the tail of the absorption band extends to the shorter wavelength at 200 nm. Therefore, the absorbance at 200 nm should be nearly equal to the value calculated from the transmittance of an ArF excimer laser beam.

This discrepancy suggests that the absorbance at the 5.8-eV band decayed after cessation of the irradiation. In fact, the spectrum in Fig. 5 was measured about 10 min after cessation of the irradiation. We then measured the time dependence of the 5.8-eV absorbance after cessation of the irradiation. The results are shown in Fig. 8. In the first stage, the intensity decreases steeply, and then decreases at a slower rate.

E. A model of the 4.4- and 5.8-eV bands

The 5.8-eV absorption band in the present sample is caused by the E' center. From the annealing effects shown above, the 4.4-eV emission band and the 5.8-eV

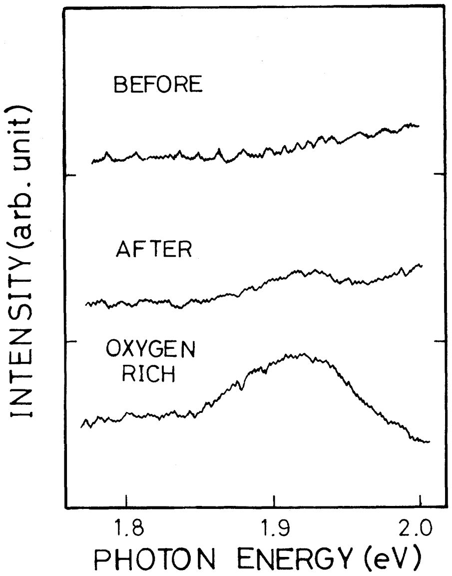

FIG. 7. Luminescence spectrum near by 1.9 eV induced with irradiating the ArF excimer laser (50 Hz, 200 mJ/cm^2, 6000 shots) before and after annealing in oxygen, and a fused silica synthesized in an oxidizing condition.

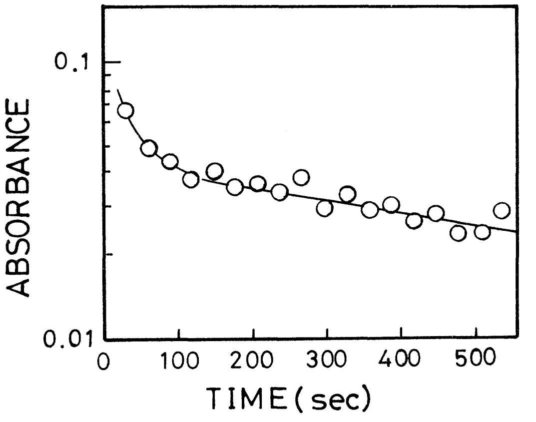

FIG. 8. Decay of the intensity of the 5.8-eV absorption after cessation of the irradiation (50 Hz, 500 mJ/cm^2, 9000 shots).

absorption band is related to hydrogen which is weakly bound to the glass network.[24] Based on these results we will discuss the characteristics of the absorption and luminescence bands, and attempt to construct a structural model.

We assume that the precursor of the center is

$$\equiv\mathrm{Si{-}H\ \ H{-}O{-}Si}\equiv\ . \tag{2}$$

This model can explain the annealing and solarization phenomena: when irradiating with the ArF excimer laser, the E' center is created as follows:

$$\equiv\mathrm{Si{-}H\ \ H{-}O{-}Si}\equiv\ \xrightarrow{h\nu}\ \equiv\mathrm{Si}\cdot\ \ \mathrm{H{-}O{-}Si}\equiv +\mathrm{H}\ , \tag{3}$$

and the annealing effect can be explained as

$$\equiv\mathrm{Si{-}H\ \ H{-}O{-}Si}\equiv\ \xrightarrow{\text{Annealing}}\ \equiv\mathrm{Si{-}O{-}Si}\equiv +\mathrm{H}_2\ . \tag{4}$$

Some of hydrogen on the right-hand side of Eq. (3) will be reacted with the E' center again to form the $\equiv\mathrm{Si{-}H}$ structure. Therefore, the concentration of the E' center will decay after cessation of the irradiation. In fact, as shown in Fig. 8, absorbance at 5.8 eV decays after cessation of the irradiation. Similar phenomena have been reported by Imai *et al.*[22] They have measured the ArF excimer laser induced E' center in several types of fused silica by electron spin resonance (ESR) at room temperature. They observed the decay of the E' center population after cessation of the ArF excimer laser irradiation in type-III fused silica. Although the history of the synthesized condition of the sample they used is not clear, the sample seems to have been synthesized in a reducing condition.

They had also shown that stable E' centers are created in oxygen deficient silicas which contain an unrelaxed oxygen deficient center. The creation of the stable center is considered to be[31]

$$\equiv\mathrm{Si}\cdots\mathrm{Si}\equiv\ \xrightarrow{h\nu}\ \equiv\mathrm{Si}\cdot+\ \overset{|}{\underset{/\ \backslash}{\mathrm{Si}}}{}^{+}+e^{-}\ , \tag{5}$$

in which $\equiv\mathrm{Si}\cdots\mathrm{Si}\equiv$ is an unrelaxed oxygen deficient hole center (ODC) showing 5.0 eV absorption and 4.3-eV emission bands. This E' center is stable even after cessation of the irradiation. Therefore the nature of the E' center is quite different from that of the type-III fused silica. By annealing the oxygen deficient silica in hydrogen ambient, unstable E' centers are induced in addition to the stable E' center. The decay time of the unstable component of the E' center is comparable to that of the type-III fused silica. The enhancement of the creation of the E' center by annealing in hydrogen ambient is also reported in type-III fused silica.[33] Shelby[34] reported a similar reaction in hydrogen impregnated fused silica by γ-ray irradiation.

Therefore, these unstable E' centers are considered to be derived from the same origin. The precursor of the E' center is the $\equiv\mathrm{Si{-}H\ \ H{-}O{-}Si}\equiv$ structure formed by the reaction of hydrogen molecules with a strained $\equiv\mathrm{Si{-}O{-}Si}\equiv$ bond in high temperature since there is a nonstoichiometric excess of hydrogen in the hydrogen-oxygen flame. It may be formed by reacting the H_2O molecule with the unrelaxed oxygen deficient center ($\equiv\mathrm{Si}\cdots\mathrm{Si}\equiv$) or Si—Si bond ($\equiv\mathrm{Si{-}Si}\equiv$):

$$\equiv\mathrm{Si}\cdots\mathrm{Si}\equiv +\mathrm{H_2O}\longrightarrow\ \equiv\mathrm{Si{-}H\ \ H{-}O{-}Si}\equiv\ . \tag{6}$$

Since the atmosphere in the reacting flame contains an extreme excess of H_2O in the present sample, these oxygen deficient centers are considered to be removed by this reaction.

The rapid decay in Fig. 8 in the first stage is considered to be the reaction between the hydrogen atom in Eq. (3) and the E' center. Some of the hydrogen atoms on the right-hand side in Eq. (3) will form hydrogen molecules during the irradiation. Therefore, the slow decay in the second stage is considered to be the reaction of hydrogen molecules and the E' center. The decay times of the second stage is estimated to be about 1000 s from Fig. 8 assuming a single exponential decay. The diffusion constant of the hydrogen molecules is about $2\times10^{-11}\mathrm{cm}^2/\mathrm{s}$ at 25 °C. The mean diffusion length is estimated to be about 1 μm. If we assume that the E' center concentration is $10^{15}-10^{17}\mathrm{cm}^{-3}$, the mean separation of the E' centers is estimated to be on the order of $1-10$ μm. If the hydrogen molecules are dispersed homogeneously in the glass directly after cessation of the irradiation, the correspondence of these lengths suggest that the slow decay reflects the diffusion of hydrogen molecules. As can be seen from Fig. 9, there are two square-root time dependent regions. This fact suggests that there are two separate diffusion controlled reactions.

Next, we will discuss the annealing effect on the luminescence characteristics. We consider that the 4.4-eV band is caused by the E' center.[14] The reaction of Eq. (4) is promoted by annealing in oxygen and helium ambient. At the present time we are not sure of the role of the oxygen and helium in promoting the reaction. A possible mechanism is as follows. If the sample is annealed in oxygen, the hydrogen molecules in the glass will react with the oxygen in the glass surface and promote the diffusing out of the hydrogen molecules from the glass and form a concentration gradient of the hydrogen. The concentration gradient further promotes the diffusing out of the hydrogen.

If the sample is annealed in helium, the helium atoms will diffuse into the glass, and will mix with the hydrogen molecules dissolved in the glass. By the mixing, the entropy of the system is increased and the reaction of Eq. (3) will be promoted. It is noted that it is not necessary to remove the hydrogen molecules dissolved in the glass to promote the reaction.

IV. SUMMARY AND CONCLUSION

Absorption and luminescence characteristics induced by the irradiation of ArF excimer laser in type-III fused silica synthesized in reducing conditions were investigated. When irradiating with an ArF excimer laser, an absorption band at 5.8 eV and an emmision band at 4.4 eV are induced. Creation of the 4.4-eV emission band is suppressed by annealing. The suppression effect in oxygen and helium is better than in the other atmospheres, i.e., nitrogen or air. These effects can be explained by assuming that the 4.4-eV band is related to weakly bound hydrogen: the oxygen pulls out the hydrogen from the glass network by reacting with hydrogen on the glass surface. Helium diffuses into the glass network to push out the hydrogen molecules. As in the case of the 4.4-eV emission band, the creation of the 5.8-eV band is also prevented by annealing in helium.

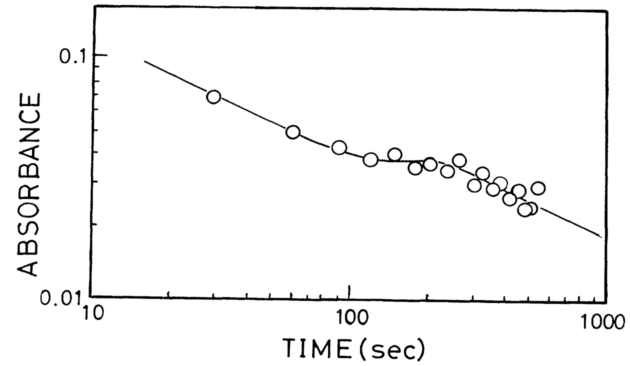

FIG. 9. Doubly logarithmic plot of the same data as in Fig. 8.

From these results we proposed a model that the precursor of these bands is the $\equiv$Si—H H—O—Si$\equiv$ structure. The E' center is created by the irradiation with the ArF excimer laser as given by Eq. (3). The hydrogen atom on the right-hand side of Eq. (3) will recombine after cessation of the irradiation. Therefore decay of the 5.8-eV band is observed.

By annealing, the precursor of the E' center is removed by the reaction previously given in Eq. (4). The reaction is promoted by removing the hydrogen molecules on the right-hand side of Eq. (4).

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