Photoluminescence from defect centers in high-purity silica glasses observed under 7.9-eV excitation

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Photoluminescence under 7.9-eV excimer-laser excitation was measured at room temperature on various types of high-purity silica glasses, which were classified based on the oxygen stoichiometry and OH concentrations. Several luminescence bands with different peak energies from 1.9 to 4.3 eV and decay constants were observed in different types of silicas. The 1.9-eV band was observed in low-OH oxygen-surplus and high-OH silicas. The 2.7-eV band was observed only in low-OH oxygen-deficient silicas. The 3.1- and the 4.2-eV bands were observed in a particular type of silica, which has a characteristic absorption band at 5.1 eV (referred to as the $B_2\beta$ band). The 4.3-eV band was observed in all types of silicas but the oxygen-surplus type. These results indicate that defects responsible for these luminescence bands are diamagnetic defects introduced during preparation or paramagnetic species induced during excitation.

I. INTRODUCTION

Since the first observation of defect creation in amorphous SiO_2 (a-SiO_2) induced by intense sub-band-gap photon irradiations,[1] much effort has been focused on defect-formation mechanisms.[2-5] Coupled with the increasing demand for the ultraviolet- (uv-) grade optics for excimer-laser applications, several studies to understand the nature of photodefect generation in a-SiO_2 have been reported.[6] During the course of these works, it has been argued that the defect process in SiO_2 involves both intrinsic and extrinsic processes. The intrinsic process involves the radiolytic displacements of oxygen from an otherwise perfect Si-O-Si network:

$$\equiv Si{-}O{-}Si\equiv \;\rightarrow\; \equiv Si\cdot\;{}^{+}Si\equiv +O+e^{-} . \tag{1}$$
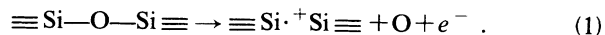
Here the dot and $\equiv$ denote an unpaired spin and the bonding with three separate oxygens, respectively. It has been proposed that reaction (1) is driven by the nonradiative recombination of self-trapped excitons.[7] On the other hand, the extrinsic process comprises transformation of precursor defects which were introduced during the preparations of a-SiO_2. For example, the generation of an E' center is considered as follows:

$$\equiv Si{-}Si\equiv \;\rightarrow\; \equiv Si\cdot\;{}^{+}Si\equiv +e^{-} \tag{2}$$

or

$$\equiv Si{-}H\rightarrow \equiv Si\cdot +\cdot H . \tag{3}$$

A considerable amount of work using electron-spin-resonance (ESR) spectroscopy has been reported, since it

can be the most powerful tool to identify defect structure in the *ground* state. Tsai, Griscom, and Friebele[3] reported the intrinsic creation of E' centers ($\equiv Si\cdot$) with a density of more than 10^{17} cm^{-3} from the normal Si—O—Si bond. Imai *et al.*[2] and the present authors[5] have reported that preexisting precursors can get involved in the defect process with paramagnetic concentrations of up to 10^{16} cm^{-3} in 6.4-eV excimer-laser-irradiated a-SiO_2. There still lacks, on the other hand, information on the *excited* states and the decay of the defect process. As for the intrinsic defect process, Itoh, Suzuki, and Itoh[8] recently observed a luminescence band at 2.2 eV and ascribed this to the radiative decay of self-trapped excitons in a-SiO_2 during pulsed-electron-beam excitation. Although Gee and Kastner[9] and Stathis and Kastner[10] observed various luminescence bands from defect centers in low- and high-OH silicas under vacuum uv (vuv) excitation, they did not correlate them with specific defect centers. Thus there remain questions on the extrinsic defect process: What roles do the defect precursors play in the defect formation and how do the excited states and the decay processes lead to defect creation?

In order to investigate the excited states and decay process of preexisting defects in a-SiO_2, photoluminescence excited by a F_2 excimer laser (7.9 eV) was observed on various types of silica glasses prepared under different conditions and methods. This paper describes the obtained results and discussion in view of their relations with preexisting precursors.

II. EXPERIMENTAL PROCEDURES

Samples used in the experiments are high-purity silica glasses with diverse contents of impurities such as

TABLE I. Sample list.

Sample name	Category	Manufacturing method	Impurity [Cl]	(ppm) [OH]
A	oxygen deficient	Ar plasma	3200	0.75
B	oxygen surplus	Ar $+O_2$ plasma	370	0.46
C	high-OH	direct	not detected	1000
D	high-OH $(B_2\beta)^a$	soot	0.3	270

^{a}See Ref. 12.

chlorine and hydroxyl, which were synthesized by soot, plasma, and direct methods. Characteristics of the samples are shown in Table I. They are first divided into low-OH (<1 ppm) and high-OH (>270 ppm) silica glasses. The low-OH silicas are further classified into oxygen-deficient and -surplus silicas based on oxygen stoichiometry, as previously reported.[11]

Excitations were done using a vuv laser pulse (7.9 eV, pulse duration ≈ 20 ns) from a F_2 excimer laser (Lambda Physik, LPX105i). The average power density of the vuv light was estimated to be $\approx 1-3$ mJ/cm^2 per pulse by using Scientech Calorimeter (38-2UV5). The luminescence observed at a right angle to the incident laser beam was dispersed by a monochromator and detected by a photomultiplier (PM, Hamamatsu, R212UH or R955). The PM current was dropped across an appropriate load resistor and recorded with a digital storage oscilloscope (Iwatsu DS-6121). Luminescence spectra are normalized to the spectral response of the detection system calibrated using a standard lamp. The time response of the system is $\approx 10^{-7}-10^{-6}$ s. All of the measurements were performed at room temperature. The optical path from the laser to the sample was under vacuum ($\approx 10^{-3}$ Torr). Before measurements samples were not irradiated with any radiations.

III. RESULTS

Figures 1–4 show (a) luminescence spectra obtained for various types of samples and (b) semilog plots of the decay measured at various photon energies.

The low-OH oxygen-deficient silica A exhibits luminescence bands at 2.7 and 4.3 eV, as shown in Fig. 1(a). The intensity of the 2.7-eV band is two to three orders of magnitude smaller than the 4.3-eV band. The 2.7-eV band decays exponentially with a time constant of 9.4 ms. The 4.3-eV luminescence decays faster than the time response of the system ($\tau_s \approx 10^{-7}$ s).

Luminescence can be seen at 1.9 eV in the spectra of the low-OH oxygen-surplus silica B, as shown in Fig. 2(a). The decay of the 1.9-eV band is exponential, and the lifetime is estimated to be about 14 μs, as seen in Fig. 2(b). It is also apparent from the spectra that there exists a broad luminescence band extending from 2 to 4 eV. Figure 2(b) shows that the decay of the 2–4-eV band measured at 2.5 eV is not exponential. The 2–4 eV luminescence band seems to exist in other spectra, but it was impossible to determine its lifetime from the data because of the overlap with other bands.

Figure 3(a) shows that the 1.9- and 4.3-eV bands exist in the high-OH silica C. Both of these bands decay with

the same constants as the corresponding bands observed in the oxygen-surplus silica B and the oxygen-deficient silica A, respectively.

As seen in Fig. 4(a), in sample D, a type of high-OH silica, luminescence was observed at 1.9 and 3.1 eV. Another broad luminescence band, centered at 4.3 eV, was also observed in this sample, which is categorized as $B_2\beta$ type.[12] The lifetime of the 3.1-eV band is estimated to be about 110 μs from Fig. 4(b). The broadband observed at 4.3 eV seems to be composed of the 4.3-eV band and a
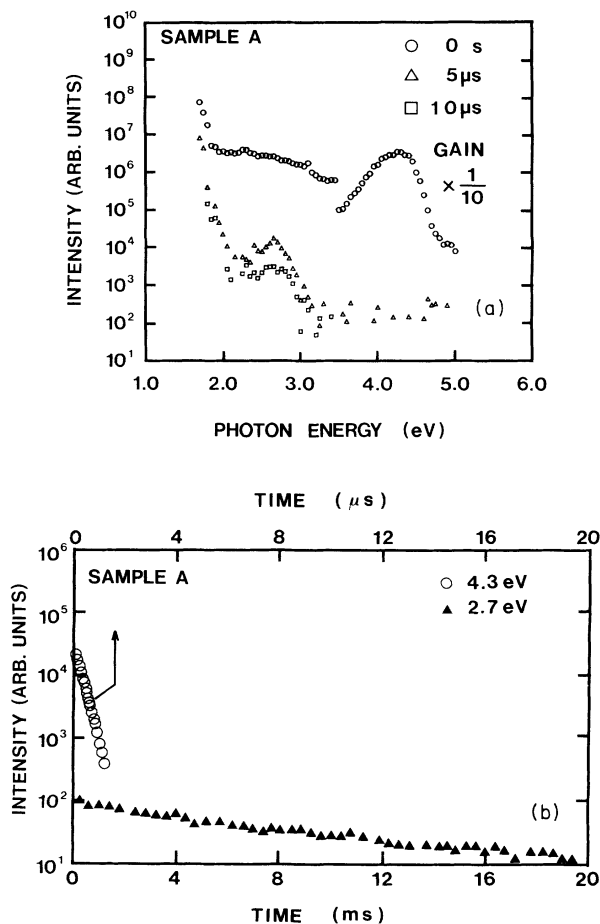

FIG. 1. (a) Photoluminescence spectra obtained at room temperature for the oxygen-deficient silica A excited by 7.9-eV photons and (b) semilog plot of the decay of the luminescence band. Note that since the decay of the 4.3-eV band is faster than or comparable to the time response of the system ($\tau_s \approx 10^{-7}$), the luminescence appears to decay exponentially with a time constant of τ_s.

new band at 4.2 eV. This can be confirmed from Fig. 4(b), which shows that the luminescence decay measured at 4.3 eV is exponential, while that measured at 4.2 eV is not.

Tables II and III summarize peak energies and lifetimes of the observed luminescence bands, respectively. The presence or not of luminescence bands depends on whether the samples are high- or low-OH type and whether they are oxygen-deficient or -surplus type. This suggests that preexisting precursors or their transformed species are responsible for the luminescence bands. This is similar to the case of permanent defect creation induced by 6.4- or 7.9-eV excimer irradiation,[5,13] as will be discussed later.

IV. DISCUSSION

A. Excitation process

First, we discuss the excitation process of the photoluminescence. Figure 5 shows the vuv optical-absorption spectra obtained for various types of silicas. Each type of silica exhibits a characteristic absorption band due to di-

amagnetic defect centers or impurities introduced during sample preparation.

The oxygen-deficient silica A has an absorption band at 7.6 eV, which was ascribed to the neutral oxygen vacancy ($\equiv$Si—Si$\equiv$).[12,14] The oxygen-surplus silica B exhibits an absorption tail at 7–8 eV, reported to be due to peroxy linkage ($\equiv$Si—O—O—Si$\equiv$).[2] The high-OH silica C has an absorption band at 8 eV, possibly due to hydroxyl group.[2] It is apparent from the vuv spectra that the 7.9-eV laser line is within these absorption bands. Thus the most possible excitation channel is a one-photon absorption process. Subsequent energy transfer from an absorption center to a luminescence center is also possible.

We cannot rule out another possibility, that a cross band-gap excitation goes through two-photon absorption and subsequent energy migration in the form of an electron-hole pair or exciton. In fact, we have found that 7.9-eV excimer-laser irradiation can create permanent defect centers through two-photon processes,[13] suggesting that two-photon excitation also occurs in the present experiment.
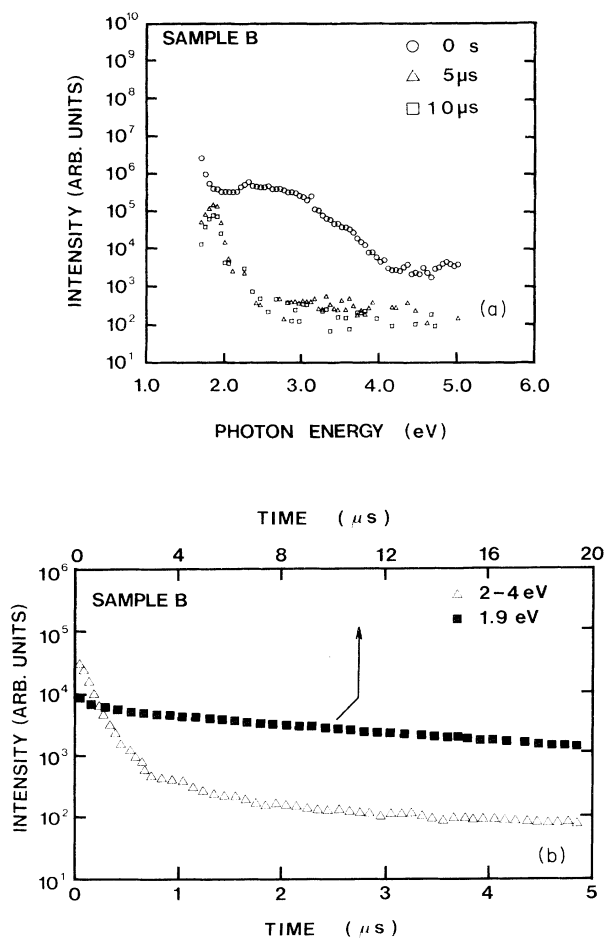

FIG. 2. (a) Photoluminescence spectra obtained at room temperature for the oxygen-surplus silica B excited by 7.9-eV photons and (b) semilog plot of the decay of luminescence bands.
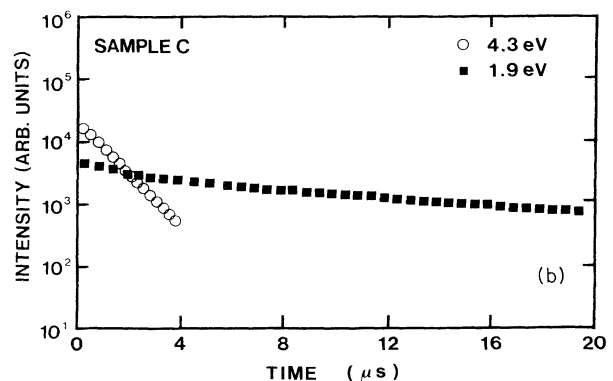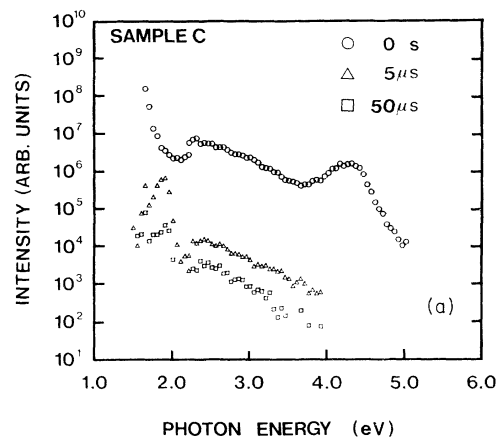

FIG. 3. (a) Photoluminescence spectra obtained at room temperature for the high-OH silica C excited by 7.9-eV photons and (b) semilog plot of the decay of luminescence bands. Note that the time response of the system is about 10^{-6} s only in this experiment, and that the decay of the 4.3-eV band is limited by this response.

TABLE II. Photoluminescence bands observed in various silicas (O and ● denote detected and not detected, respectively).

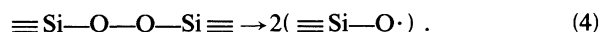| Sample	Energy (eV)					
	1.9	2.7	3.1	4.2	4.3	2–4
A	●	O	●	●	O	O[a]
B	O	●	●	●	●	O
C	O	●	●	●	O	O[a]
D	O	●	O	O	O	O[a]

[a]The luminescence band is recognized in the spectra, but the decay was not determined because of interference from other bands.
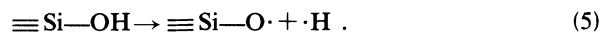
B. Luminescence bands

We found that the presence or not of luminescence bands varies from sample to sample, as shown in Table II. In the following we will discuss the cause of the luminescence band and decay process.

The 1.9-eV band is observed in the oxygen-surplus silica B and in the high-OH silicas C and D. The lifetime (14 μs) of the luminescence agrees well with the previous reports.[10,15] Several workers have assigned the 1.9-eV band
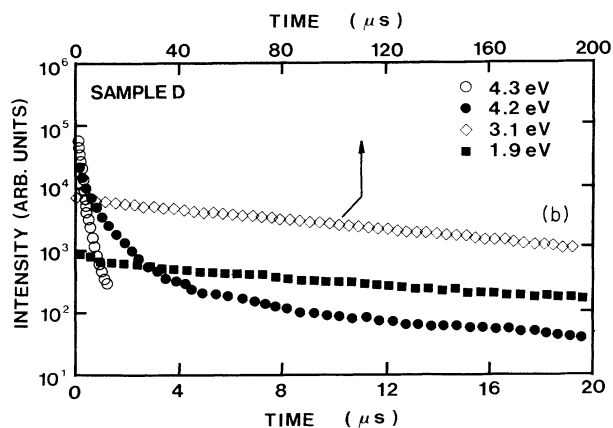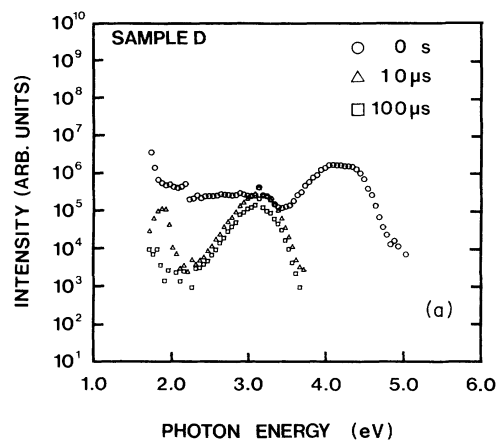

FIG. 4. (a) Photoluminescence spectra obtained at room temperature for the high-OH silica D ($B_2\beta$ type) excited by 7.9-eV photons and (b) semilog plot of the decay of luminescence bands.

to the nonbridging oxygen hole center (NBOHC, $\equiv$Si—O·).[15,16] Since the samples were not exposed to any radiations other than the excitation laser, the luminescence centers should be newly induced during the excitation by 7.9-eV photons. Actually, we observed the ESR of NBOHC's in both types of silicas irradiated with the 7.9-eV excimer laser.[13] Therefore, it is also possible for the NBOHC's to be induced in the present 7.9-eV photon excitation:

$$\equiv Si\text{—}O\text{—}O\text{—}Si \equiv \rightarrow 2(\equiv Si\text{—}O\cdot) \ . \tag{4}$$

In the case of the high-OH silica, hydroxyls are believed to be a form of precursors of the NBOHC's:

$$\equiv Si\text{—}OH \rightarrow \ \equiv Si\text{—}O\cdot + \cdot H \ . \tag{5}$$

It is thought that subsequent excitation of the NBOHC's results in the 1.9-eV luminescence. Various excitation channels are possible, as mentioned above, such as the one involving energy transfer from other luminescence centers[17] or the one involving recombination of an e-h pair.[18]

Recently, Awazu and Kawazoe[19] proposed a different model which assigned the 1.9-eV band to the photodissociation of O_3 in oxygen-surplus silicas. Although the O_3 model requires the presence of surplus oxygen in the form of O_2 molecules, the high-OH silica used in this study does not contain surplus oxygen, which can be checked by ESR measurements after 7.9-eV excimer-laser irradiation.[13] Thus we have to rule out the O_3 model for the observed 1.9-eV luminescence, at least in the case of the high-OH silica.

The 2.7-eV band is characteristic of the oxygen-deficient silica A, as shown in Table II. The lifetime of the luminescence is consistent with previous reports on the 2.7-eV band observed when excited at the 5.0-eV band ($B_2\alpha$ band) with a KrF excimer laser.[20] Assuming

TABLE III. Decay constant of the luminescence band.

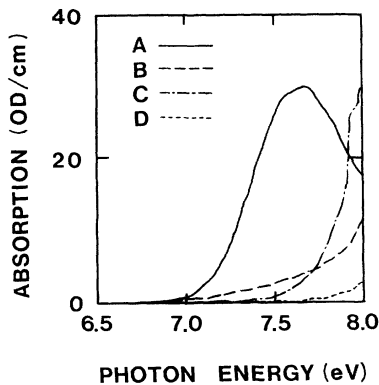| Energy (eV)	Decay constant (s)
1.9	1.4×10^{-5}
2.7	9.4×10^{-3}
3.1	1.1×10^{-4}
4.2	not exponential
4.3	$< 10^{-7}$
2–4	not exponential

FIG. 5. Vacuum ultraviolet absorption spectra of various types of samples between 6.5 and 8.0 eV. *A*, low-OH oxygen-deficient silica; *B*, low-OH oxygen-surplus silica; *C*, high-OH silica; and *D*, high-OH silica ($B_2\beta$ type).

the energy diagram obtained for the neutral oxygen vacancy ($\equiv$ Si—Si $\equiv$) based on *ab initio* molecular-orbital calculations, the 2.7-eV band can be described in the following way. The ground-to-singlet transition takes place by 7.9-eV photon absorption or, if any, by energy transfer from excited states such as excitons at the oxygen vacancy sites. Then intersystem crossing occurs between the excited singlet to the excited triplet states. The forbidden transition from the triplet state to the singlet state results in 2.7-eV emission. The lifetime of 9.4 ms is quite consistent with the present view. It should be noted that the present 2.7-eV luminescence is different in many ways (bandwidth, lifetime, etc.) from the luminescence observed in *crystalline* SiO$_2$, which was ascribed to the radiative recombination of the self-trapped excitons.[21]

The 3.1-eV band was observed only in a type of high-OH *pure* silica *D* ($B_2\beta$ type), while Skuja and Trukhin[22] proposed that the 3.1-eV band is due to Ge impurity in silica. Our present observation is consistent with our previous study[12] and the one by Kohketsu *et al.*,[23] reporting that the luminescence was observed in a *pure* silica. Kohketsu *et al.* used the samples produced under reducing atmosphere (H$_2$/He mixture), and they have concluded that the 3.1-eV band is due to twofold coordinated silicon lone-pair centers (O—S̈i—O). On the other hand, our investigation[5] on the paramagentic centers induced by 6.4-eV photons in the same type of silica has suggested the existence of weak O—O bonding as a preexisting defect, which may be responsible for the 3.1-eV band. The decay constant is relatively slow ($\tau \approx 10^{-4}$ s), when compared with other luminescence bands which have shorter lifetimes, such as the 4.3-eV band with a decay constant less than 10^{-7} s.

The 4.2-eV band is observed in $B_2\beta$-type silica along with the 3.1-eV band. This is consistent with the previous report on the same type of silica under cw excitation at the 5.1-eV band (referred to as the $B_2\beta$ band).[12] The decay of the luminescence cannot be expressed by a single exponential curve, suggesting that the decay constant is distributed. The assignment of the origin of the 4.2-eV luminescence has to wait for a future work.

The 4.3-eV band is observed in all types of silicas except the oxygen-surplus silica *B*, though the magnitude of the luminescence varies from sample to sample. The authors reported that the oxygen-deficient-type silica exhibits the 4.3-eV band along with the 2.7-eV band when excited at 5.0 eV ($B_2\alpha$ band).[12] Gee and Kastner[9] reported that the 4.3-eV luminescence has an excitation peak at 7.6 eV in a certain type of silica, in which the oxygen vacancies are probably created by neutron irradiation. We assume here our previous assignments of the 5.0- and 7.6-eV bands in oxygen-deficient silica, to the singlet-to-triplet and singlet-to-singlet transitions at a neutral oxygen vacancy site ($\equiv$ Si—Si $\equiv$), respectively. It is then considered that the 4.3-eV luminescence can arise from the oxygen vacancies, though the specific transition is not known for this luminescence.

The 4.3-eV band is also observed in the high-OH silicas *C* and *D*, though the intensity is one or two orders of magnitude smaller than that seen in the oxygen-deficient sample *A*. The fact that the 7.6-eV band is not seen in the vuv spectrum in Fig. 5 suggests that the concentration of oxygen vacancies is below the detection limit. If we assume that the 4.3-eV band as well as the 2.7-eV band is due to transitions at the oxygen vacancies, an explanation for the absence of the 2.7-eV band [see Fig. 3(a)] is that, in the case of the high-OH silicas *C* and *D*, the luminescence intensity is lower than the detection limit of the present experimental system. Thus the sample-to-sample variance in the 4.3-eV luminescence intensity may reflect the various concentrations of the neutral oxygen vacancy, which probably depend on the manufacturing methods and conditions.

A broadband spreading over 2–4 eV is apparent in Fig. 2(a). However, this band is not clear in the other spectra because of interference with other bands, and it was impossible to obtain the real decay. Thus it is difficult to determine whether this band is due to the same defect as those in the other samples or not. The decay of the 2–4-eV band, which is not exponential, is similar to the decay characteristics of the 2.2-eV band reported by Itoh, Suzuki, and Itoh.[8] They reported that the decay of the 2.2-eV luminescence band follows a power law and that the band is due to the radiative recombination of self-trapped excitons.[8] As discussed above, since the cross band-gap excitation through two-photon absorption of 7.9-eV photons is also possible under the present experiments, the formation of *e-h* pairs or excitons is expected. However, the present results indicate that the intensity of the 2.2-eV band is sample dependent (see Figs. 1–4). Thus surplus oxygen may play a role in the appearance of this band.

It is interesting to compare the present results with those reported by Stathis and Kastner[10] on commercially available silicas, Suprasil W ([OH] < 5 ppm) and Suprasil ([OH] $\approx$ 1200 ppm). As shown in Table I, samples used in our study are categorized by their hydroxyl contents and oxygen stoichiometry. Suprasil W and Suprasil are generally regarded as stoichiometric[3] or oxygen surplus.[24] As discussed above, the decay characteristics of the 1.9-, 2.7-, and 4.3-eV bands observed in our study agree well with those reported by Stathis and Kastner. The relative

intensity ratio of the 2.7- to the 4.3-eV band is less than 1 to 10^3, which is also consistent with their spectrum. Somewhat confusing is the fact that the 2.7- and 4.3-eV bands, both associated with oxygen vacancies, were observed in Suprasil W. Furthermore, the presence of these bands appears to contradict the observation of the 1.9-eV band, which is associated with surplus oxygen. A possible explanation is that there may be some place-to-place variation in the stoichiometry or distribution of oxygen vacancies in the Suprasil W. We observed the spatial distribution of defects and impurities in a variety of silica glass.[25] Assuming that this is the case, it is anticipated that Suprasil W exhibits the characteristics of both oxygen-deficient and -surplus silicas. The spectrum of Suprasil is similar to our high-OH silica C, though we could not detect the 2.2-eV band, which was assigned to an unknown impurity.

V. SUMMARY

Photoluminescence was observed for various types of silica glasses excited by a 7.9-eV excimer laser at room temperature. Six luminescence bands from 1.9 to 4.3 eV are identified whose presence has sample dependence, as was observed in our ESR investigation.[5] Such sample dependence strongly suggests that the cause of luminescence is related to preexisting defects or impurities introduced during the sample preparation. The 1.9-eV band is observed in both the low-OH oxygen-surplus silica and two types of high-OH silicas. The 1.9-eV band is due to

NBOHC's whose precursors are either peroxy linkages or hydroxyls. The 2.7-eV band can be seen only in the oxygen-deficient silica. The decay constant obtained for the 2.7-eV band is 9.4 ms, which is due to the triplet-to-singlet transition at the neutral oxygen vacancy, as the present authors' previous calculation had predicted. The 3.1- and 4.2-eV bands are observed in high-OH *pure* silica ($B_2\beta$ type), suggesting that both luminescence bands are due to intrinsic defect centers. The 3.1-eV band decays at a relatively slow rate of 110 μs, while the 4.2-eV band decays nonexponentially. The 4.3-eV band is observed in silicas other than oxygen-surplus silica. The 4.3-eV band observed in the oxygen-deficient silica is accompanied by the 2.7-eV band, while that in the high-OH silica is not. It is considered that 4.3-eV luminescence arises from neutral oxygen vacancies, as far as oxygen-deficient silica is concerned.

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